

UNIVERSITY OF THESSALY
SCHOOL OF ENGINEERING
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Dopamine detection electrochemical biosensors

by
Savvaidis Athanasios

Supervisor
Prof. Tsiakaras Panagiotis

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Επιβλέπων
Καθηγητής Τσιακάρας Παναγιώτης

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Εγκρίθηκε από τα μέλη της Τριμελούς Εξεταστικής Επιτροπής:

1st Examiner: Dr. Tsiakaras Panagiotis (Supervisor)

Professor

Department of Mechanical Engineering

School of Engineering

University of Thessaly

2nd Examiner: Dr. Charalampous Georgios

Assistant Professor

Department of Mechanical Engineering

School of Engineering

University of Thessaly

3rd Examiner: Dr. Brouzgou Angeliki

Assistant professor

Department of Energy systems

Faculty of Technology

University of Thessaly

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ABSTRACT

Electrochemical devices convert chemical energy into electrical energy or vice versa and include batteries, fuel cells, supercapacitors, and electrochemical sensors. Batteries store energy via redox reactions for use in electronics and vehicles. Fuel cells generate electricity by converting chemical energy from fuels like hydrogen, offering clean energy for transport and power. Supercapacitors provide quick energy bursts through electrostatic charge storage, which is ideal for rapid charge/discharge applications. Electrochemical sensors detect chemical changes by converting them into electrical signals, used in environmental monitoring, medical diagnostics, and industry. Each device type is optimized for specific energy and sensing applications. Applications for electrochemical devices are many and cover a variety of sectors. Supercapacitors and batteries are essential for energy storage since they power everything from renewable energy sources to electric cars and portable devices. For backup energy demands, stationary applications, and mobility, fuel cells provide a clean power production solution. Electrochemical sensors are essential for environmental monitoring because they can identify toxins and pollutants in soil, water, and the air. Electrochemical devices are also used in industrial settings for quality assurance, process control, and chemical reaction monitoring. These sensors are employed in the medical field for biomolecule detection and health diagnostics.

In this context, neuroscience and biomedical diagnostics have advanced significantly with the development of electrochemical dopamine detection instruments. A major neurotransmitter, dopamine, is involved in mood, cognitive processes, and motor control. A variety of neurological and psychiatric conditions have been related to dopamine dysregulation. Because of their ability to track the electrical changes brought about by electrochemical processes, electrochemical sensors provide a potent tool for the sensitive, selective, and real-time detection of dopamine. These instruments often use methods like impedance spectroscopy, voltammetry, and amperometry to accurately measure dopamine levels. The performance and stability of these sensors have been further improved by developments in electrode materials, such as the creation of new nanomaterials and biocompatible coatings. This study examines the fundamentals and practical uses of electrochemical devices and consequently their application in dopamine detection systems, emphasizing the advancements in technology that have increased sensitivity and selectivity. Better knowledge, monitoring, and treatment of dopamine-related illnesses may result from the further development of electrochemical sensors, which holds great promise for clinical diagnostics and research into these disorders.

CHAPTER 1 INTRODUCTION

1.1 Motivation

One of the most important neurotransmitters (NTs) is dopamine. Carlsson and colleagues at the Laboratory for Chemical Pharmacology at the National Heart Institute of Sweden made the initial discovery of its function in 1958. NTs are classified into two functional classes based on their ability to either inhibit or activate postsynaptic membrane receptors: the inhibitory class and the excitatory class. Dopamine is a neurotransmitter that belongs to both groups and is essential to the central nervous, cardiovascular, renal, and hormonal systems. Elevated dopamine levels are indicative of cardiotoxicity, which can cause increased heart rates, hypertension, heart failure, and addiction to drugs. On the other hand, low dopamine levels are associated with conditions such as depression, schizophrenia, Parkinson's disease, stress, and Alzheimer's disease (Liu & Liu 2021).

Dopamine detection yields many benefits for pharmacological research, particularly in drug development. Since many medications target dopamine pathways, accurately measuring dopamine levels is essential for developing and testing new drugs for various conditions, including mental health disorders, addiction, and neurodegenerative diseases. In patients receiving dopamine-related medications, monitoring dopamine levels is vital for adjusting dosages, ensuring effective treatment, thus optimizing therapeutic outcomes. It is evident that measuring dopamine is necessary to comprehend its biological activities and associated biological processes.

The great sensitivity, selectivity, and adaptability of electrochemical sensors make them especially suitable for the detection of dopamine in complex biological environments. These sensors provide continuous monitoring of dopamine levels in both clinical and research settings, providing quick, on-site readings. This capacity is particularly useful for studying brain functions, evaluating treatment efficacy, and expanding our understanding of dopamine-related diseases. The aim of this thesis is to review the current literature concerning the detection of dopamine by using biosensors.

1.2 Introduction to Literature Review

The scientific and industrial field of electrochemistry is founded on physicochemical phenomena that occur anytime chemical and electrical energy exchanges interfere with a reactive system. In this way, most of the time, electrochemistry studies electrical current-flowing systems, such as batteries or electrochemical generators, which are found in many commonplace devices like automobiles and cell phones (Lefrou et al., 2010).

For clinical applications, the development of a sensitive, dependable, accurate, rapid, and portable analytical instrument that can detect biomolecules is urgently needed (Akgonullu et al., 2020). For the quantitative measurement of biomolecules, several traditional methods are available, including spectroscopic and chromatographic methods. While these techniques are accurate, it is difficult to identify biomolecules in real time because they need specialized, sophisticated equipment, as well as skilled staff to operate the equipment and prepare samples (Soler et al., 2020). The development of an analytical enzymatic device for glucose detection by Clark and Lyons in the 1960s paved the way for biosensor research and the use of electrochemistry in biomolecule detection systems (Brouzgou et al, 2014).

Additionally, diagnostic tools like electrocardiography (ECG) and electroencephalography (EEG) are commonly used in clinical diagnostics. The heart's electrical activity is captured by the ECG, which helps in cardiac condition monitoring and diagnosis. The ECG gathers vital information about heart health by applying electrodes to the skin to detect the electrical impulses coming from the heart. Similarly, EEG uses electrodes on the scalp to detect electrical activity in the brain. This is crucial for diagnosing of neurological conditions including epilepsy and sleep disorders (Chen et al., 2014).

Pacemakers and defibrillators are examples of devices with therapeutic uses of electrochemistry that have transformed the treatment of cardiac problems. While defibrillators use high-energy shocks to restore normal heart rhythm for severe arrhythmias or cardiac arrest, pacemakers use electrical impulses to keep the heart muscles contracting to keep patients' hearts beating regularly (Untereker et al., 2011). Another significant application is Deep Brain Stimulation (DBS), which treats neurological diseases including essential tremor and Parkinson's disease. To alleviate symptoms, DBS entails implanting electrodes in certain brain regions to modify neural activity.

1.3 Thesis Organization

This thesis is organized into five main chapters, each addressing critical aspects of dopamine detection using biosensors. Chapter 2 examines existing knowledge about electrochemistry and its basic principles. Chapter 3, introduces the basic types of electrochemical sensors and their applications. Also, characteristics examples will be discussed on dopamine's role, related diseases, and traditional detection methods. Chapter 4 focuses on biosensors used for dopamine detection and gives details about the design, fabrication, and experimental setup, as well as the different types. In addition, Chapter 4, interprets the results in the context of existing literature, compares the biosensor performance with traditional methods, discusses the advantages and limitations, and explores future research implications.

CHAPTER 2 ELECTROCHEMISTRY BASICS AND DEFINITIONS

2.1 Basic definitions

One significant area of contemporary research that brought the study of chemistry and electricity together is the discipline of electrochemistry. Scientists have known for a long time that chemical energy can be converted into electricity, as Volta's discovery of the first battery in 1800 showed (Decker, 2005). A few years later, the Volta battery was utilized as an energy source to split water into hydrogen and oxygen. The revelation that the bonding forces between hydrogen and oxygen atoms are caused by positive and negative electrical charges was made possible by this significant chemical discovery. The theory put out by Swedish scientist Berzelius in 1812 states that all atoms are electrified; for instance, metals and hydrogen are positive while nonmetals are negative. Energy to break up the attraction forces and generate ions is provided when electricity is supplied to two electrodes submerged in a solution (Petrovic, 2021).

Similarly, electrical energy may be transformed into chemical energy to electrosynthesize a range of beneficial molecules. Nicolson was the first to demonstrate this in 1800, breaking down water into hydrogen and oxygen using Volta's battery. Later, Faraday showed that the quantity of electrolytic products in an electrochemical reaction is directly correlated with the amount of electricity spent in the reaction, opening the door for more thorough research on electrochemical reactions (O'Mullane, 2013).

Electrochemistry is the study of chemical processes related to the separation of charges, typically in media-like solutions. Charge transfer, which can happen heterogeneously on electrode surfaces or homogeneously in solution between distinct chemical species, is frequently linked to the separation of charge. Thus, it is instantly apparent that its applications are very broad and will be discussed later. At least two charge transfer half-reactions, oxidation (loss of electrons or rise in oxidation state) and reduction (gain of electrons or decrease in oxidation state), occur concurrently and in opposing directions to guarantee electroneutrality (Gerischer, 2019).

The oxidation and reduction half-reactions are spatially separated on electrode surfaces and often take place at distinct electrodes submerged in solution inside a single cell or in distinct cell compartments. To enable the transfer of charge and completion of the electrical circuit, conducting pathways are used to connect the electrodes both outside and within the solution. It is possible to capture the released electrical energy when the total of the Gibbs energy changes at both electrodes is negative. If the difference is positive, external electrical energy can be used to counteract the positive Gibbs energy and force electrode reactions to occur, converting chemicals (electrolysis).

Therefore, like in industrial electrolytic reactions, the products of the two electrode reactions can be separated if the cell arrangement allows it (Brett, 2014).

Devices called electrochemical cells are used to convert chemical energy into electrical energy or the other way around. Galvanic (also known as voltaic) cells and electrolytic cells are the two main kinds. Galvanic cells are essential to fuel cells and batteries because they produce electrical energy from spontaneous chemical reactions. On the other hand, electrolytic cells, which are frequently used in electroplating and the extraction of metals from ores, use electrical energy to propel non-spontaneous chemical processes. The anode of these cells is the electrode where oxidation happens, and the cathode is the electrode where reduction happens.

2.2 Laws and Equations

In electrochemistry, the magnitudes of electrolytic effects are expressed quantitatively by Faraday's rules of electrolysis. According to the first law, the total electric charge (Q) that passes through the cell is directly proportional to the mass (m) of the material that is changed at an electrode. The following formula may be used to explain this mathematically:

$$m = Q \cdot \frac{M}{n \cdot F} \quad m = \frac{Q \cdot M}{n \cdot F}$$

where M is the substance's molar mass, F is Faraday's constant (about 96485 C/mol), and n is the number of electrons involved in the reaction. This rule aids in the measurement of the material used in electrochemical reactions (Grimnes& Martinsen, 2014).

The second law states that the amounts of chemical changes produced by the same quantity of electricity in different substances are proportionate to their equivalent weights, whereas the first law states that the amount of chemical change being produced by a current at an electrode-electrolyte interface is proportional to the quantity of electricity used. The gram formula weight connected to a unit gain or loss of electrons is the equivalent weight of a material in electrolytic processes (Grimnes& Martinsen, 2014).

In electrochemical devices, several fundamental laws and equations provide a detailed understanding of their operation and performance. Ohm's Law is central to analyzing electrical circuits, relating voltage (V), current (I), and resistance (R) through the equation $V=I/R$. This relationship is crucial for designing and troubleshooting electrochemical cells and fuel cells ensuring that the voltage and current are appropriately managed (Tenny, 2023).

In electrochemical theory, the Butler-Volmer Equation connects the reaction kinetics and the overpotential (η) to describe the kinetics of electrochemical processes (Dickinson et al., 2020).

$$j = j_0 \left[\exp \left(\frac{\alpha_a n F \eta}{RT} \right) - \exp \left(- \frac{\alpha_c n F \eta}{RT} \right) \right]$$

j_0 is the current density at equilibrium, where the rates of the anodic and cathodic reactions are equal. It is a measure of the intrinsic activity of the electrode material.

α_a and α_c are the anodic and cathodic transfer coefficients, respectively. These coefficients (typically between 0 and 1) quantify the fraction of the overpotential that influences the rates of the anodic and cathodic reactions.

n is the number of electrons transferred in the electrochemical reaction.

F is Faraday's constant, and R is the gas constant (8.314 J/(mol·K))

Finally, an essential formula in electrochemistry for comprehending how the concentrations of reactants and products in an electrochemical reaction affect the electrode potential (E) of the cell is the Nernst Equation. The formula is (Amman, 2018):

$$E = E^0 - \frac{RT}{nF} \ln \frac{a_{\text{red}}}{a_{\text{ox}}}$$

E^0 represents the standard electrode potential measured under standard conditions (1 M concentration, 1 atm pressure, 298 K), providing a baseline reference

R is the gas constant (8.314 J/(mol·K)) and T is the absolute temperature in Kelvin,

n is the number of electrons transferred in the redox reaction,

F is Faraday's constant and

a_{red} and a_{ox} are the activities of the reduced and oxidized species

2.3 Electrochemical devices

Electrochemical devices are vital for the conversion of chemical energy to electrical energy and the other way around. They are used in many technologies, including fuel cells, batteries, electrolyzers, and supercapacitors. Energy conversion, material processing, sensing, and energy storage are all common uses for these gadgets. For example, electric cars and gadgets run on lithium-ion batteries, whereas fuel cells use hydrogen and oxygen to produce energy. Supercapacitors provide quick energy discharge, whereas electrolyzers use water splitting to make hydrogen. Furthermore, highlighting the significance of electrochemistry in contemporary technology are electrochemical sensors, actuators, and procedures like electroplating and corrosion prevention, which are essential in industries ranging from industrial manufacturing to healthcare. Below is a detailed description of the different types of electrochemical devices that are commonly used.

2.3.1. Batteries

Batteries may be divided into two categories: single-use batteries (such as non-rechargeable batteries) and multi-use batteries (such as rechargeable batteries). Most studies focus on batteries that are well-known for their capacity to repeatedly store and release energy, which makes them economical and environmentally friendly in a wide range of applications. Since the invention of the rechargeable battery in 1859, it has evolved from lead-acid to lithium-ion technology. Key developments in the 1970s–1980s revolutionized lithium-ion batteries. In the late 1970s, Dr. M. Stanley Whittingham at Exxon Mobil explored rechargeable batteries using titanium disulfide and lithium metal electrodes, though safety concerns arose (Boddula et al., 2020).

In the 1980s, Professor John B. Goodenough developed the lithium cobalt oxide cathode, and Professor Rachid Yazami demonstrated that graphite could intercalate with lithium (ref). Building on these advances, Yoshino created the first lithium-ion battery prototype in 1985. By 1991, Sony and Asahi Kasei commercialized the first true Li-ion battery. This technology has since been refined by major companies globally (Kim, 2022).

2.3.1.1. Basic principle and types

The battery, also known as a cell, is typically made up of an electrolyte, a separator, and two electrodes with differing potentials, or cell voltages. The main components are the electrodes, the anode, and the cathode, which are made of various materials with various purposes. These mostly impact the cell voltage and specific capacity. The anode serves as the host for the lithium source that is stored in the cathode. Ions-bearing charges are transferred by the electrolyte from the cathode to the anode and back. A barrier against short-circuiting is created by the separator, which keeps a specific distance between the two electrodes. The electrolyte-soaked separator must be both electrically insulating and aid in this ionic flow. They ought to watch the electrochemical stage as well. If they do take part, the cells will deteriorate because they will either dissolve quickly or cover one electrode in a solid electrolyte interphase (SEI), which can obstruct ionic transport. To create stable, ionically conductive SEIs, the electrolyte can be modified using different types of modifications (Zschornak et al., 2018). **Figure 1** explains the different parts of a battery.

A battery's capacity to hold a charge is determined by the potential difference between the active ion electrochemical processes occurring at the two electrodes, as well as the number of electrons participating in each reaction. To enhance the quantity of charge stored or the system's capacity, one can either utilize ions containing multiple electrons or raise the potential difference

between the two electrode processes. The process by which charge is transmitted affects both of these variables (Borah et al., 2020).

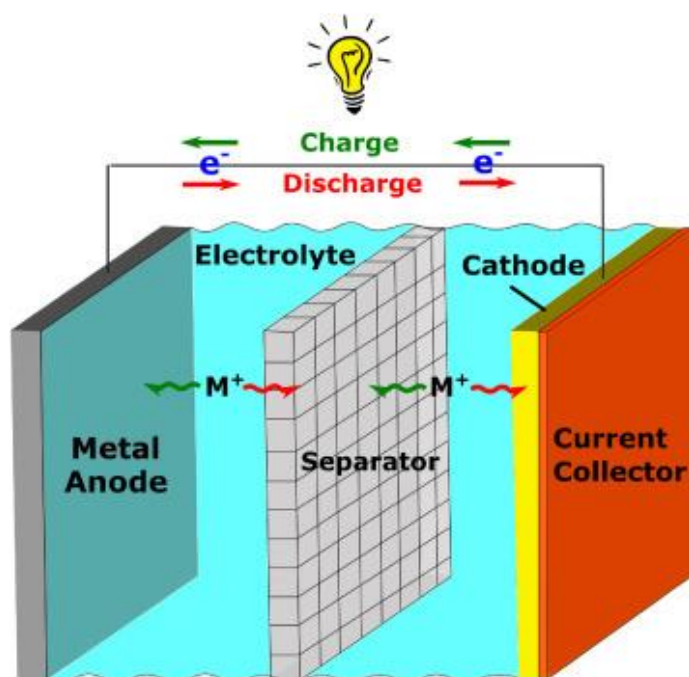


Figure 1. A typical battery format (Borah et al., 2020).

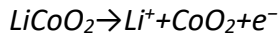
The anode's function is to maintain the active ions' high-energy state. The cell's final voltage will increase with increasing energy state. Because pure metal can retain the most metal atoms in the smallest amount of space and has the greatest energy state conceivable, it is theoretically the ideal material for an anode. However, the usage of pure metal is frequently hampered by dendritic development. As a result, other approaches—like adding ions to graphite's interlayer gaps—that can stop this dendritic growth are frequently employed in its place.

Metals, metal oxides, and electrolytes are the components most frequently found in batteries, and each is essential to the battery's operation. The anode of lithium-ion batteries, which are widely used in electric cars and portable gadgets, is usually constructed of graphite, a kind of carbon that permits lithium ions to intercalate, or insert, between its layers (Palacin, 2021). However, because of their high energy density and stability, lithium metal oxides like lithium cobalt oxide (LiCoO_2), lithium iron phosphate (LiFePO_4), or lithium nickel manganese cobalt oxide (LiNiMnCoO_2) are frequently found in the cathode. During charging and discharging, the electrolyte—typically a lithium salt mixed in an organic solvent—helps transfer lithium ions between the anode and cathode (Zhao et al., 2016).

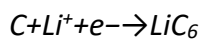
Li-ion battery

Lithium-ion batteries are commonly used in a variety of consumer gadgets that are portable. Lithium-ion batteries outperform other types of batteries in terms of energy-to-weight ratio, nearly never

have a memory effect, and have less self-discharge during idle periods. During charging, an external electrical power source applies a voltage across the battery, causing lithium ions (Li^+) to migrate from the cathode to the anode. At the cathode, lithium ions are extracted from the lithium metal oxide through an oxidation reaction, which releases electrons (Kim et al., 2020). This reaction can be represented as:



After that, the released lithium ions move to the anode via the electrolyte, and the electrons move to the anode via the external circuit, where they mix with the lithium ions. The lithium ions undergo a reduction process at the anode as they intercalate, or insert, between the graphite layers. This process stores energy in the battery, as lithium ions are accumulated in the anode. The reaction that takes place can be represented as: (Jiang & Peng, 2016)



Discharging involves the reverse procedure, and the aforementioned reactions are reversed. Through the electrolyte, the lithium ions return from the anode to the cathode. Along with the release of electrons, the lithium ions are deintercalated from the graphite at the anode and released into the electrolyte. The lithium ions return to the cathode when the electrons pass through the external circuit, supplying electricity to the attached device. These lithium ions undergo a reduction process at the cathode as they re-intercalate into the lithium metal oxide and then the battery releases the restored energy (Rouhi et al., 2022). A Li-ion battery's electrolyte, which is usually a lithium salt such as lithium hexafluorophosphate (LiPF_6) mixed in an organic solvent combination, is electrically insulating to prevent short circuits and essential for ion movement. Lithium ions are free to flow through a porous separator that physically divides the anode and cathode. This keeps the electrodes from coming into direct contact, which may result in a short circuit and perhaps cause battery failure (Ghijet al., 2020).

Figure 2 depicts the basic parts of a Li-ion battery.

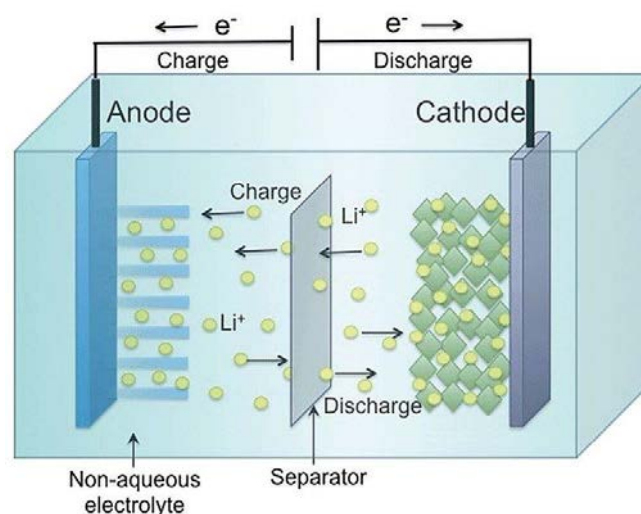


Figure 2. The Li-ion battery (Ghiji et al., 2020).

Alternative battery types employ distinct materials. Lead dioxide (PbO_2) is utilized as the cathode material while sponge lead (Pb) is used as the anode in lead-acid batteries, which are widely employed in automotive applications. The electrolyte in these batteries is sulfuric acid. Formerly used in portable electronics, nickel-cadmium (NiCd) and nickel-metal hydride (NiMH) batteries use potassium hydroxide (KOH) as the electrolyte and nickel oxide hydroxide (NiOOH) for the cathode and either cadmium (Cd) or a metal hydride alloy for the anode. In order to improve energy density, safety, and endurance, emerging battery research is investigating alternate materials such as silicon anodes, solid-state electrolytes, and sulfur-based cathodes. The selection of materials used in batteries is essential as it directly affects the battery's cycle life, energy capacity, efficiency, and safety.

2.3.1.2. Applications

One of the many benefits of batteries is their high energy density, which makes them perfect for electric cars and portable electronics as it allows them to store a lot of energy in relation to their weight and size. In addition, they offer a reduced maintenance requirement, consistent and dependable power production, and the capacity to be recharged, which prolongs their lifespan. Nevertheless, there are drawbacks to batteries as well. These include comparatively high prices, particularly for sophisticated chemistries like lithium-ion, and problems with weight and energy density in some varieties. Cycling can also cause a battery's performance to deteriorate over time, resulting in a decrease in capacity and efficiency. Significant obstacles also come from environmental and safety issues, such as the proper disposal of toxic chemicals and possible thermal runaway dangers (Ramasubramanian et al, 2024).

Concerning battery applications these may be divided into three main categories: stationary power storage, portable electronics, and transportation and automotive, which includes electric vehicles (EVs). Batteries are used in consumer electronics to power a variety of products, including wearable technology, computers, tablets, and smartphones. In particular, lithium-ion batteries have established themselves as the industry standard for these uses because of their high energy density, extended cycle life, and relatively lightweight. The need for effective, long-lasting batteries has increased as these gadgets have grown more indispensable to daily life, which has fueled ongoing developments in battery technology.

Batteries are essential to the functioning of hybrid electric vehicles (HEVs) and electric cars (EVs) in the automobile industry. Longer driving ranges and quicker charging periods are made possible by the development of high-capacity batteries, which are driven by the move to sustainable energy (Helmers & Marx, 2012). This market is dominated by lithium-ion batteries, although research on solid-state and

other next-generation batteries is still ongoing to get past constraints on things like price, weight, and charging speed. Furthermore, conventional automobiles still frequently utilize lead-acid batteries to start engines, run lights, and supply backup power (Chen et al., 2019).

Utilized for energy storage, batteries find extensive usage in the renewable energy industry. Because the generation of energy from solar and wind power is sporadic, reliable storage options are required to guarantee an uninterrupted flow of power. When demand is high or renewable output is low, large-scale battery systems, such as those used in grid storage, may store extra energy generated during peak production times and release it. This lessens reliance on fossil fuels and increases the dependability of renewable energy sources respectively (Nyamathulla&Dhanamjayulu,2024). Batteries are also necessary for the medical profession to power many vital equipment, such as implanted medical devices, hearing aids, pacemakers, and portable defibrillators. Because the equipment these batteries power is lifesaving, these batteries need to be extremely dependable and long-lasting without needing to be changed frequently. Because of their lifespan and stability, lithium batteries are frequently utilized in various applications (Nazaralizadeh et al., 2024).

2.3.2. Fuel cells

2.3.2.1. Basic principle and types

Fuel cells are cutting-edge devices that, without burning, use electrochemical processes to transform the chemical energy of fuels like hydrogen, methane, and alcohols into electrical energy. They work like batteries, but they do not run down or need recharging (Wu et al, 2019). Due to its circumvention of the Carnot cycle constraints and definition of the Gibbs free energy change of the reactions, this method is incredibly efficient. Fuel cells are considered ecologically beneficial as they release very little sulfur oxides (SOx) and no nitrogen oxides (NOx) or particles, depending on the situation (Carrette et al., 2001).

A fuel cell is composed of three active components: a fuel electrode (anode), an oxidant electrode (cathode), and an electrolyte sandwiched between them. The electrodes consist of a porous material that is covered with a layer of catalyst (often platinum in PEMFCs) (Sharaf& Orhan, 2014). Similar to batteries, fuel cells run on a constant fuel source. The oxidation of fuel occurs at the anode, whereas the reduction of an oxidant, such as oxygen, occurs at the cathode. Ions flow via an electrolyte between the electrodes, while electrons flow through an external circuit to produce electricity. Fuel cells don't require recharging as batteries do. To function effectively, fuel cells require a dense electrolyte to prevent fuel or oxygen crossover and ensure ion conduction.

Electrodes must be porous to enhance reaction efficiency and stability, providing a large active surface area while being chemically stable and electrocatalytic. The cost-effectiveness and durability of these components are crucial for the widespread adoption of fuel cell technology (O'hayre et al., 2016; Douvartzides et al., 2003). The next **figure 3** depicts the basic parts of a fuel cell.

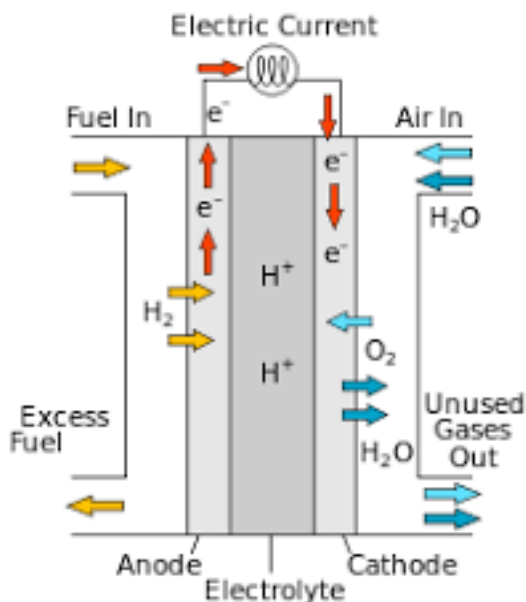


Figure 3. The working principle of a fuel cell (<https://journalstar.com/fuel-cell>).

One of the most popular forms of fuel cells is the proton exchange membrane (PEM) fuel cell, which uses a proton-conducting polymer membrane as the electrolyte. The most popular material for the membrane is Nafion, a perfluorosulfonic acid polymer, because of its superior proton conductivity and durability. Platinum-based catalysts are usually supported by porous carbon materials used to create the electrodes in PEM fuel cells. Because of its exceptional effectiveness in promoting both the oxygen reduction process at the cathode and the hydrogen oxidation reaction at the anode, platinum is the most often used catalyst (Alaswad et al., 2020).

Other catalysts have also been studied. The FeOx@graphitic carbon core-shell nanoparticles embedded in a nitrogen-doped ordered mesoporous carbon matrix (FeOx@GC-NOMC) developed by Wang et al., demonstrated superior electrocatalytic performance and stability compared to commercial Pt/C. This material showed enhanced activity in acidic media, followed a four-electron oxygen reduction reaction (ORR) process, and was resistant to methanol poisoning. The ordered mesoporous carbon matrix aided in rapid electron transfer, and active site exposure, and prevents nanoparticle agglomeration, while the core-shell structure and high “Fe-N” site content protected against harsh conditions and ensured durability (Wang et al., 2020; Tan et al., 2017).

In a study conducted by Andreadis et al., the researchers investigated the creation of parasitic current and how it affected the cathode's performance, which in turn affected the PEM fuel cell's overall functioning. A visual representation showed the direct association between the parasitic current creation and the ethanol crossover rate. Furthermore, the computation of the cathode activation overpotential when the parasitic current was enabled and disabled revealed that the mixed overpotential for a DE-PEMFC presents a significant issue impeding the fuel cell's functioning (Andreadis et al., 2008).

PEM fuel cells face challenges including the high cost and limited durability of platinum catalysts, sensitivity to operating conditions like humidity, and vulnerability to fuel impurities such as carbon monoxide. Additionally, developing cost-effective and durable materials for the membrane and bipolar plates is a significant hurdle (Beuscher et al., 2005).

The electrolyte used in solid oxide fuel cells (SOFCs) is often constructed of a ceramic substance that conducts oxygen ions at high temperatures, such as yttria-stabilized zirconia (YSZ) (Coutelieris et al., 2003). YSZ and nickel composites are often used for the anode, and mixed ionic-electronic conductors, such as lanthanum strontium manganite (LSM), are used for the cathode. These substances are selected because they can function well at the elevated temperatures—typically between 700 and 1,000°C—needed by SOFCs. (Lyagaeva et al., 2015). In this type of fuel cell at the porous air electrode, oxygen is reduced to generate oxide ions when an external load is supplied to the cell. When these ions reach the fuel electrode through the solid electrolyte, they combine with the fuel—H₂ or CO—to form H₂O or CO₂. An alternative is to employ a solid electrolyte that conducts protons, where H₂ is oxidized to create protons that then react with oxygen to make water. On the anode, CH₄ can be directly oxidized to produce CO₂ and H₂O (Yamamoto, 2000).

Several efforts have been undertaken to create low-cost, long-term stable commercially successful generators based on SOFCs. Reducing the operation temperature to the intermediate temperature (IT) range of 400–750 °C can help partially alleviate this issue since it allows for the use of less expensive electrode and interconnector materials and reduces stack deterioration. A study by Medvedev et al., used the solution combustion synthesis method of nitrate and different kinds of organic fuels (glycine, glycerin, citric acid, and a mixture of citric acid and ethylene glycol) to obtain nano-sized powders of 0.5Ce_{0.8}Sm_{0.2}O_{2-δ}–0.5BaCe_{0.8}Sm_{0.2}O_{3-δ} with good stability at intermediate temperatures (Medvedev et al., 2013).

One of the earliest contemporary fuel cells to be created was the alkaline fuel cell, which was initially created in 1960. The Apollo spacecraft's onboard electric power supply was the application at the time. The AFC's favorable characteristics include its versatility in using a variety of

electrocatalysts and superior performance in comparison to other candidate fuel cells because of its active O₂ electrode kinetics. The AFC is still in use today; UTC Fuel Cells' cells are used to power the Space Shuttle Orbiter when it is in orbit. These AFCs fulfill the specifications for space applications by operating at a reasonably high pressure and temperature (Shekhawat et al., 2011). Non-precious metals like silver or nickel are frequently employed as catalysts in alkaline fuel cells (AFCs), which use an alkaline electrolyte like potassium hydroxide (KOH). Typically, AFC electrodes are constructed of nickel or nickel alloys (Ferriday & Middleton, 2021). These fuel cells offer great power density and affordable, sustainable energy. Because alkaline medium exhibits better kinetics of complicated processes, alkaline fuel cells have an advantage over other fuel cells.

2.3.2.2. Applications

Fuel cells stand out due to their benefits. Their remarkable efficiency as compared to conventional combustion engines is one of their main advantages. Especially in combined heat and power (CHP) systems that employ waste heat, fuel cells can more efficiently transform chemical energy directly into electrical energy. Additionally, they are eco-friendly since they emit very little—usually just heat and water—which lowers air pollution and greenhouse gas emissions. Because of this, using fuel cells to produce electricity with a lower carbon footprint is a desirable alternative. Furthermore, fuel cells are scalable and adaptable, making them appropriate for a variety of uses, including large-scale power plants and backup systems as well as portable power supplies. They may be employed in a variety of contexts, including commercial, industrial, and residential ones, and operate quietly (Carrette et al., 2001)

A number of significant obstacles prevent fuel cells from being widely used. First off, fuel cell systems can be expensive overall due to the high cost of materials like platinum catalysts. Furthermore, the practical usage of fuels like hydrogen is limited by the absence of an all-encompassing infrastructure for their production, storage, and delivery. The long-term dependability and efficiency of fuel cells can be impacted by durability problems, such as catalyst deterioration and membrane failure. Further barriers include the energy-intensive and logistically difficult process of producing hydrogen, as well as efficiency losses under different circumstances. Technology advancements, cost-cutting, and better infrastructure are needed to overcome these obstacles (Wang et al., 2018; Gorbova et al, 2008).

Depending on the needed power output scale, fuel cells can be employed in various scenarios. PEMFCs stand out from other fuel cell types because of their unique characteristics, which include a quick start-up time, a wide operating temperature range (-40 to 90 °C), and high specific

energy. As a result, they are widely utilized in stationary and fuel cell vehicles. In transportation, fuel cells are used in vehicles such as cars, buses, trucks, trains, and ships, providing a clean alternative to traditional internal combustion engines by utilizing hydrogen as fuel while when they are employed in stationary power generation, offering reliable and efficient power for residential, commercial, and industrial applications, including backup power systems and off-grid locations(Chalk et al., 2000).

Fuel cells, which use electrochemical processes to transform alcohols like methanol or ethanol into hydrogen and energy, are a potential method for producing hydrogen from alcohol. Compared to direct hydrogen storage, this method uses alcohols as a fuel source to produce hydrogen in a more controllable and effective way (Liu et al., 2021). The first step in the process for these fuel cells is feeding the alcohol into the anode, where it is oxidized. Usually, a catalyst—such as platinum or an alloy based on platinum—is attached to the anode to help in the oxidation process. For example, methanol (CH_3OH) is oxidized in a methanol fuel cell to yield protons (H^+), electrons (e^-), and carbon dioxide (CO_2) (Vayenas et al., 1990). While the protons pass through the electrolyte membrane and reach the cathode, the liberated electrons flow through an external circuit to generate electricity. As a result of the interaction between oxygen from the air and protons and electrons at the cathode, water is produced along with electrical power, completing the circuit (Lima et al., 2009; Song et al., 2007).

Efficient ethanol electrooxidation was studied by Wang et al., who developed an ordered 2D hexagonal mesoporous carbon/tungsten carbide (OMC/WC) composite nanomaterial using a soft-template method, with ammonium metatungstate as the tungsten precursor and the tri-block copolymer (PEO–PPO–PEO, F127) as the template. The results showed that when platinum (Pt) is loaded onto this composite, the Pt@OMC/WC demonstrates significantly better performance for methanol electrooxidation compared to commercial PtRu/C. It achieves specific mass activities 2.4 times higher (per mg Pt) and 3.2 times higher (per mg metal) (Wang, et al., 2012; Molochas & Tsiakaras, 2021; Wang et al., 2014).

2.3.3. Photovoltaic cells

Over the last ten years, photovoltaics has emerged as significant players in the continuing energy revolution. Developments in materials science and production techniques have played a major part in that evolution. Still, a lot more work needs to be done before photovoltaics can produce cheaper, greener energy (Pastuszak & Węgierek, 2022).

2.3.3.1. Basic principles and types

Electrical devices called photovoltaic cells can convert light energy into electric current. A photovoltaic cell is essential to a solar cell. The term "photovoltaic cell," which is an acronym for this kind of cell, is frequently used to refer to it. A diode featuring a p-n junction is the most basic part of a solar cell. Solar cells are one sort of photoelectric cell, which is a device in which the presence of light modifies the electrical characteristics of the object (e.g., voltage, current, or resistance). A type of cell that falls within this particular category is the solar cell. Solar panel fabrication involves the use of modules, which are assembled from solar cells. After that, these modules are added to panels. Most single-junction silicon solar cells have an average open-circuit voltage of 0.5 to 0.6 volts when operating at maximum efficiency. When cells are operating at their best, this happens. While this amount may not seem like much, you should remember that these solar cells are really little. A huge solar panel may provide a good quantity of environmentally friendly electricity (Dixit et al., 2023).

The photovoltaic cell uses the photovoltaic phenomenon in which photons, or light energy, are absorbed by the semiconductor material, usually silicon, when sunlight strikes the cell. This energy causes the silicon's electrons to get excited, releasing their atomic bonds and forming electron-hole pairs. Two layers of semiconductor material, one negatively charged (n-type) and one positively charged (p-type), make up the structure of the solar cell. At their junction, these layers create an electric field. Electrons flow via an external circuit as a result of this field pushing holes toward the p-type layer and free electrons toward the n-type layer. Electric current is produced by this flow and may be stored or utilized to power devices (Yang, 2022).

A photovoltaic cell's fundamental structure is made up of several layers and parts that work together to convert sunlight into electrical energy. As shown in **Figure 4** the semiconductor material, which is often silicon, is in the center of the solar cell. It is this material that is essential for absorbing sunlight and starting the photovoltaic effect. The selection of silicon is based on its abundance and efficient electrical characteristics. To produce two different forms of silicon—n-type silicon, which has an excess of electrons (negative charge carriers), and p-type silicon, which has an excess of "holes" (positive charge carriers when electrons are absent)—the silicon is doped, which means that trace quantities of other elements are added to the silicon (Al-Ezzi & Ansari, 2022). The p-n junction is the crucial region where these two varieties of silicon converge. Because the p-type and n-type materials have different charges, this connection creates a natural electric field. As photons from the sun's rays enter the cell through the silicon, they provide electrons energy and enable them to break free from their atomic bonds, forming electron-hole pairs. A current of electricity is created

at the p-n junction by pushing the holes and released electrons in the direction of the p-type layer and the n-type layer, respectively (Ojha et al., 2022).

An antireflection coating is put on the cell's surface to increase efficiency. A large amount of sunlight would just bounce off the surface of the without this layer.

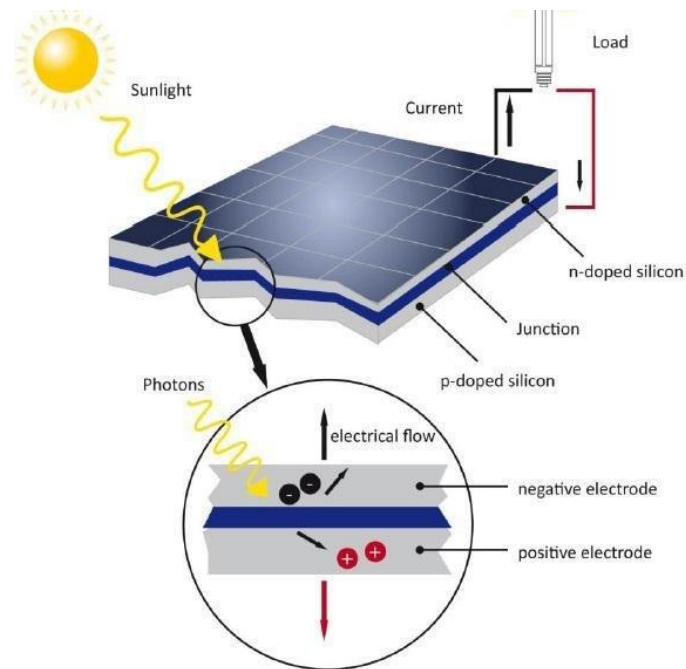


Figure 4. Schematic representation of a photovoltaic cell (<https://www.nrel.gov/pv>).

The evolution of photovoltaic cells is divided into four key generations, each marking significant advancements in solar technology. The First Generation includes photovoltaic cells based on monocrystalline and polycrystalline silicon, as well as gallium arsenide (GaAs). Despite their relatively high production costs and because of their comparatively high efficiency, these cells are the oldest and most widely utilized. The abundance of Si indicates both its long-term availability and its potential for lower acquisition costs. Utilized in a variety of applications, this stable and non-toxic element improves durability and aids in the prevention of contamination. Moreover, Si PVCs, are a flexible and sustainable option for the next technological advancements as they allow the integration of well-established technologies like integrated circuits and transistors with the Si-based microelectronics sector (Luceño-Sánchez et al., 2019).

The Second Generation builds upon this by developing thin-film technologies using materials like microcrystalline and amorphous silicon, copper indium gallium selenide (CIGS), and cadmium telluride/cadmium sulfide (CdTe/CdS). These cells are made to use less material and spend less money on production without sacrificing performance. These cells' increased flexibility and lightweight are made possible by thin-film technology, which opens a range of surface applications, such as flexible substrates and building-integrated photovoltaics (BIPV). Production costs can be

further reduced using large-scale manufacturing techniques like vapor deposition. In addition, these cells have greater operating temperatures and lower light levels than conventional silicon-based cells (Aamir Iqbal et al., 2022).

The Third Generation introduces more recent technologies, including nanocrystalline films, quantum dots, dye-sensitized solar cells, perovskites, and organic polymers. Key components comprise perovskites, renowned for their exceptional efficiency and economical manufacture; quantum dots, able to capture a wide range of solar radiation; and organic photovoltaics (OPVs), providing adaptability and economical production (Tsiakaras et al, 1998). Various materials are available, including multi-junction cells that stack layers such as gallium arsenide (GaAs) to collect more of the sunlight spectrum and dye-sensitized solar cells for a variety of applications (Brown & Wu, 2009).

Finally, the Fourth Generation combines the flexibility and low cost of thin-film polymers with the durability of innovative inorganic nanostructures, such as metal oxides, metal nanoparticles, and organic-based nanomaterials like graphene and carbon nanotubes (Rehman et al., 2023). These cells frequently mix organic and inorganic components through hybrid designs that combine the high efficiency and stability of inorganic semiconductors with the flexibility and cheap manufacturing cost of organic photovoltaics. Materials like carbon nanotubes and nanocrystals improve charge transfer and light absorption, therefore nanotechnology is vital. In order to maximize the use of the solar spectrum, fourth-generation cells may also include multi-functional layers, such as tandem or multi-junction structures. The goal of this generation is to produce solar cells that are efficient, economical, and ecologically friendly. These cells may be used in a variety of applications, ranging from wearable technology and creative building-integrated systems to conventional panels (Iqbal et al., 2022).

Each generation represents a step forward in enhancing the efficiency, cost-effectiveness, and versatility of solar energy technology (Luque & Hegedus, 2011).

2.3.3.2. Applications

Among the many benefits of solar cells is their ability to capture sunlight, a nearly infinite resource that may be used as a renewable energy source to minimize environmental impact and lessen dependency on fossil fuels. Once installed, they need little maintenance and don't require fuel, which results in long-term savings. Furthermore, solar energy produces clean energy without emitting any pollutants, which helps to mitigate climate change and improve air quality. By enabling customers to produce their own electricity, they improve energy independence, which is particularly

advantageous in isolated or off-grid locations. Small household systems to big utility-scale farms may benefit from solar technology's scalability and versatility, which also generates employment possibilities across a range of industries (Kant& Singh, 2022).

Solar cells can have several drawbacks, though. Their intermittent energy output is reliant on the availability of sunlight, which is influenced by the weather and time of day. This leads to uneven power generation. Solar panel installation and acquisition might have a significant upfront cost, but long-term savings and incentives frequently make up for this. To produce enough electricity, they need a large amount of room, which isn't always possible. Additionally, solar systems sometimes require pricey energy storage devices, like batteries, to guarantee a steady supply of electricity. Despite being largely offset by the long-term advantages, the energy and toxic chemicals used in the production of solar panels can nonetheless have an adverse effect on the environment. Finally, the efficiency of solar cells is limited, as they can only produce a small amount of power from sunlight (Almosni et al., 2018).

Solar cells are frequently used in rooftop solar panels for domestic usage, which lowers energy costs and lessens reliance on the grid by producing power for residences. Solar panels are mounted on the facades or rooftops of commercial buildings to supply clean energy for corporate activities, reducing operating costs and improving sustainability. Large arrays of solar cells are used in utility-scale solar power plants to produce significant amounts of electricity that support large-scale renewable energy objectives and the grid (Sharma et al.,2015).

Solar cells also power homes, schools, and medical facilities in rural and off-grid places where regular energy infrastructure is either nonexistent or not available. They are also utilized in solar-powered vehicles, such as buses, automobiles, and even boats, where they provide a clean energy source for mobility (Bagher et al., 2015). Furthermore, solar cells are essential to portable solar chargers for electronics, providing easy and environmentally responsible power for devices like laptops and phones. The use of solar energy in agriculture is growing, as solar-powered greenhouse heating and irrigation systems lower energy costs and improve farming productivity. These many uses highlight the adaptability and rising significance of solar cells in numerous facets of contemporary society and business (Hashemi et al.,).

2.3.4. Supercapacitors

A capacitor is a device with two conducting plates separated by an insulating dielectric substance. It is used to store and release electrical energy. The capacitor's charge storage capacity is increased by the dielectric, which can be ceramic, plastic, or paper, and which keeps the plates,

which are usually formed of metal, from coming into touch with one another electrically. In order to preserve its internal components, the capacitor is often housed in a protective casing and has exterior terminals for connecting to an electrical circuit. Capacitors play an essential role in electronic circuits, such as energy storage, filtering, and signal smoothing, thanks to their straightforward yet efficient construction (Shabbir et al., 2023).

In 1975, the Japanese business Nippon Electric filed for patents on supercapacitors (SCs) or ultracapacitors as a solution to the above-mentioned capacitor difficulties. In comparison to traditional capacitors, supercapacitors have much higher output power (between 50 and 100 KW), high charge density, a 12-year life expectancy, a 500,000-cycle life, and a high self-discharge rate. For applications requiring instantaneous high output power, such as hybrid car engine cranking applications and permanent magnet synchronous generators, SC are therefore appropriate (Nadeem et al., 2018).

2.3.4.1. Basic Principles and types

The basics of operation for SCs and Cs are the same. However, in SC, an electrolytic physical barrier made of activated carbon is employed as a dielectric to permit ionic conduction, helping the SC to have a high energy density and a large specific area. Although they normally have a lower energy density than batteries, supercapacitors are renowned for their high-power density, capacity to deliver brief bursts of energy, and long cycle life. Because of these qualities, they are perfect for uses that call for quick cycles of charging and discharging, including backup power supplies or regenerative braking systems (Zhang et al., 2023).

It is composed of two electrodes submerged in an electrolyte solution and separated by a porous separator. The electrodes are constructed of materials with high surface areas, including activated carbon. Positive and negative charges build up at the electrode surfaces when a voltage is applied, forming an electric double layer. Electrostatic energy storage is provided by this double layer. In order to improve energy storage, certain supercapacitors also employ pseudo capacitance, which is the result of quick, reversible redox processes on the electrode surface. Ions are driven from the electrolyte to the electrodes by the applied voltage during charging, which increases the energy in the electric field. When discharging, current flows across the circuit because the ions return via the electrolyte, releasing the stored energy (Zhang et al., 2022). **Figure 5** depicts the basic components of a supercapacitor.

Based on the materials they are made of and their underlying energy storage techniques, they are divided into three categories. The Electrochemical Double-Layer Capacitor (EDLC) is the most used type. Without requiring any chemical processes, EDLCs store energy by allowing charges

to separate electrostatically at the interface between the electrolyte and the electrode material. Graphene, carbon nanotubes, or activated carbon are examples of carbon-based materials that are commonly used as electrodes in EDLCs. Because of their extended cycle life and high-power density, EDLCs are highly appreciated in applications that call for rapid energy bursts, such as power backups or regenerative braking systems. However, their usage in applications needing long-term energy storage is limited due to their lower energy density in comparison to batteries (Jalal et al., 2021).

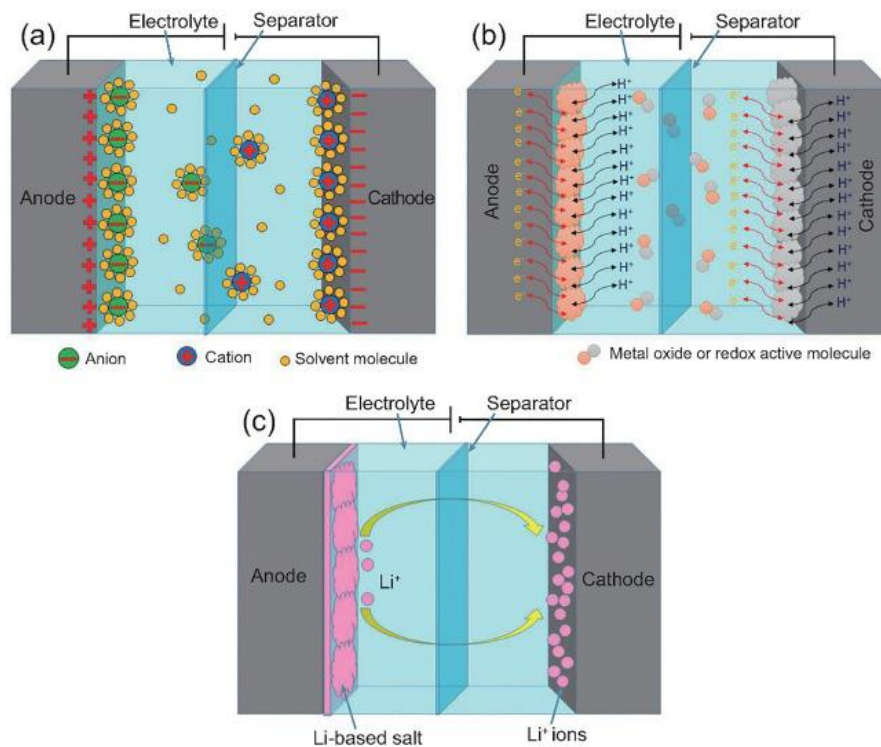


Figure 5. Schematic representation of the different types of supercapacitors (Zhang et al, 2023).

Pseudocapacitors, the second type, are identified by their capacity to store energy via faradaic processes, which are rapid and reversible redox reactions that take place at or close to the electrode surface. Compared to EDLCs, pseudocapacitors can attain a greater energy density because of this technique. Pseudocapacitors are commonly constructed from conducting polymers like polypyrrole and polyaniline, as well as transition metal oxides like manganese oxide (MnO_2) and ruthenium oxide (RuO_2). Although pseudocapacitors are more energy-efficient than EDLCs, their performance in high-power applications may be constrained by their lower power density and shorter cycle life (Bhojane, 2022).

To achieve a balance between energy density and power density, hybrid supercapacitors include the characteristics of both EDLCs and pseudocapacitors. Asymmetric supercapacitors and battery-supercapacitor hybrids are two examples of several varieties of hybrid supercapacitors (Chatterjee et al., 2021). In order to store more energy while still producing high power, asymmetric supercapacitors usually utilize a carbon-based material for one electrode (much like an EDLC) and a battery-like material for the second electrode. Lithium-ion capacitors are an example of a battery-supercapacitor hybrid that combines

the quick charge and discharge times of supercapacitors with the high energy density of batteries. For uses like grid energy storage and electric car energy storage that require both rapid energy release and large energy storage capacity, these hybrid devices provide a flexible option (Muzaffar et al., 2019).

2.3.4.2. Applications

Applications for supercapacitors are numerous, ranging from simple electronics like LEDs to defense and medical equipment. Because of their exceptional qualities, supercapacitors are widely employed in a variety of industries, including aerospace, medical, energy, automotive and transportation, and electronics. Supercapacitors are used in aerospace because of their capacity to provide short bursts of power, which is essential for systems on satellites and spacecraft. They are employed to control the flow of power during brief intervals of high demand, including when satellites are maneuvering or when communication systems are being powered. They are appropriate for demanding space applications where maintenance is difficult due to their great dependability and extended lifespan (Buerger et al., 2017).

Supercapacitors are essential components of renewable energy systems because they mitigate the intermittent nature of energy sources such as solar and wind. They stabilize the energy supply by storing surplus energy produced during periods of peak production and releasing it during periods of low output or high demand. Their quick charging and discharging times make them perfect for reducing energy output variations and guaranteeing a consistent, dependable power source. Supercapacitors are also utilized in combination with batteries to improve system performance. This is because supercapacitors' high-power density and batteries' energy density work together to maximize energy management and storage in renewable energy applications (Zhang et al., 2023).

In medicine, they offer dependable power for vital equipment like portable medical kits and defibrillators. For equipment that must continue to operate dependably in an emergency, their capacity to deliver quick energy bursts is crucial. Supercapacitors' extended cycle life and safety qualities are also beneficial in medical implants and monitoring systems (Nigam et al., 2020). Supercapacitors can improve patient comfort and device dependability by ensuring that these devices have a steady power source without the need for frequent battery changes. They are also used in portable diagnostic devices and wearable health monitors, where their fast energy delivery enables continuous operation and speedy data processing. They are an important part of the advancement of medical technology and the enhancement of patient care because of their extended cycle life and stability (Chodankar et al., 2024).

2.3.5. Electrolysis devices or electrolyzers

Electrolyzers are electrochemical devices that use electricity primarily to create oxygen as a byproduct of water and hydrogen as the main commodity. The electrolyzer is the only commercially accessible technology that can manufacture high-purity hydrogen without the need for hydrocarbon feedstock or carbon emissions, in contrast to all other techniques of producing hydrogen. Nevertheless, electrolyzers have been available for over a century, this technology has not yet been applied with success (Yan et al., 2020). Many factors contribute to this, such as the lower cost of other hydrogen production technologies and the general lack of knowledge in the public and industrial sectors regarding the benefits of electrolyzers, particularly in terms of ecologically beneficial hydrogen generation (Dincer& AlZahrani, 2018).

Scientists have been increasingly interested in the breakdown of water since the middle of the eighteenth century. Paets van Troostwijk and Deimann explained to de la Metherie in a letter dated 1789 that water was converted into vitalized air and combustible air by an electric spark. However, Alessandro Volta's invention of the voltaic pile—which was first made of silver disks, zinc, and platinum, and connected by gold and copper—was what led to the discovery of the notion of water electrolysis in 1800 (Smolinka et al., 2022; Long, et al., 2017; Long et al., 2019).

One of the most important steps in producing hydrogen, a pure and adaptable energy source, is water electrolysis. Using electrical energy, water is divided into hydrogen and oxygen in this process. Along with other physical and chemical processes like adsorption, electron transfer, the creation of molecular hydrogen from atomic hydrogen, and so on, a number of electrochemical reactions, including reduction and oxidation, may take place during the electrolysis process. The specific type of electrolyzer used will determine the types of electrochemical reactions that are involved in the process (Naterer, et al., 2013; Goula et al., 2004).

An electrolyte separates the two electrodes in an electrolyzer, which is made up of a positively charged anode and a negatively charged cathode. Electrochemical processes happen when an electric current is run through the water. At the anode, water is oxidized to produce oxygen gas and positively charged hydrogen ions; at the cathode, hydrogen ions pick up electrons to make hydrogen gas (Chi et al., 2018). The electrochemical decomposition process of water occurs according to the following anode and cathode evolution reactions. The anode and cathode reactions and their respective reversible potential in a water electrolytic cell can be expressed as follows:

Anode: $\text{H}_2\text{O} \rightarrow 2\text{H}^+ + \frac{1}{2} \text{O}_2 + 2\text{e}^-$ ($U_{\text{rev}}=1.229\text{V}$)

Cathode: $2\text{H}^+ + 2\text{e}^- \rightarrow \text{H}_2$ ($U_{\text{rev}}=0.00\text{V}$)

Overall reaction: $\text{H}_2\text{O} \rightarrow \text{H}_2 + \frac{1}{2} \text{O}_2$ ($U_{\text{rev}}=1.229\text{V}$)

The utilization of renewable energy sources, such as solar, wind, or hydropower, to provide the required electrical energy makes water electrolysis a very effective process for manufacturing green hydrogen. This ensures that the process is sustainable. (Lu et al., 2019). Furthermore, being adaptable, this approach may be used on a wide range of scales, from small-scale laboratory experiments to massive industrial systems. Notwithstanding its potential benefits, hydrogen generation is not without its hurdles. These include high energy needs and related expenses. The kind of electrolyzer, the water's quality, and the energy source all affect how effective water electrolysis is. It will not be possible to produce hydrogen on a big scale by water electrolysis and contribute to a sustainable energy future without advancements in electrolyzer technology, such as bettering catalyst materials and lowering energy usage (Awad et al., 2024).

2.3.5.1. Types of electrolyzers

Conventionally, electrolyzers are classified according to a number of attributes, such as the type and content of the electrolyte, operation temperature, and operating pressure. Both the operating temperature and the kind of electrolyte are the basis for the most accepted categorization. By kind of electrolyte, electrolyzers fall into three primary categories: solid oxide electrolyzers (SOEs), PEM electrolyzers, and alkaline electrolyzers. Regarding their operating temperature, electrolyzers are separated into two categories: (1) low-temperature electrolyzers, which normally run at temperatures lower than 80°C , and (2) high-temperature electrolyzers, which run at temperatures higher than 500°C (Dincer & AlZahrani, 2018).

SOEs are advanced devices used for electrochemical water splitting to produce hydrogen, leveraging high-temperature electrolysis (Yu et al., 2017). They operate at elevated temperatures, typically between 700°C and 1000°C , which enhances the ionic conductivity of the solid oxide electrolyte. The high operating temperature reduces the electrical energy required for the electrolysis process by providing thermal energy that drives the endothermic reaction. In an SOE, water is split into hydrogen and oxygen using an applied electric current (Xu et al., 2023; Yu et al., 2019).

SOEs are highly efficient due to their ability to operate at high temperatures and their capability to use low-cost, non-precious metal catalysts. The high operating temperatures, reduce ohmic losses and improve reaction kinetics and thermodynamics compared to alkaline and PEM electrolyzers. The high temperature enables the use of both heat and electrical energy for the endothermic steam

reduction process, decreasing the electrical energy requirement. This process is well-suited for integration with existing chemical plants, where waste heat from exothermic processes, such as synthetic fuel production or steel manufacturing, can be utilized (Hauch et al., 2006). Additionally, the internal cell resistances in SOEs generate Joule heat, further contributing to the required enthalpy. Also, the production of the precise power needed for electrolysis without requiring extensive external thermal management, results in high energy efficiency and cost-effectiveness due to the use of inexpensive catalysts. They are particularly promising for large-scale hydrogen production, especially when integrated with renewable energy sources or surplus electricity from other industrial processes, thereby contributing to a sustainable energy future (Wolf et al., 2023).

Proton exchange membranes, which act as solid polymer electrolytes that promote the transport of protons (hydrogen ions) from the anode to the cathode while being impermeable to gases, are the fundamental part of PEM electrolyzers. Water is supplied into the electrolyzer's anode side during operation, where it is oxidized to yield protons, electrons, and oxygen gas. While the protons pass through the proton exchange membrane and reach the cathode side, the oxygen gas is expelled from the system. The energy required to power the reduction process at the cathode is supplied by the electrons, which move via an external electrical circuit. Protons and electrons mix at the cathode to create hydrogen gas, which is then collected and used (Ahmed et al., 2022).

Finally, alkaline electrolyzers produce hydrogen in alkaline conditions, achieved with the use of an alkaline electrolyte like potassium hydroxide. In order to transmit hydroxide ions between the electrodes and improve the cell's overall conductivity, the alkaline electrolyte is essential. Comparing alkaline electrolyzers to other electrolyzer technologies, they are renowned for being inexpensive, simple, and durable. They are efficient in the large-scale generation of hydrogen and can function at moderate pressures and temperatures. Alkaline electrolyzers have several drawbacks, though, such as reduced efficiency and current density when compared to more recent technologies like PEM electrolyzers. Despite this, they are a well-liked option for energy storage and industrial hydrogen generation because of their resilience and financial feasibility (Brynolf et al., 2018).

A study conducted by Hu et al., compared the efficiency of the different types of electrolyzers. The outcomes demonstrated that since high temperatures activate the electrodes and lower electric demand, the SOEC's current density achieves its maximum. PEM is ranked second due to the solid polymer electrolyte's superior performance. Next, using various definitions of efficiency, the three types of electrolyzer's respective levels of efficiency were compared and the findings indicated that when it comes to energy conversion efficiency, SOE is not as superior to PEM cells as it may be if improper heat management is practiced. However, if inexpensive or even free heat

sources are included, we may still hypothesize that SOEC is more cost-effective than the others. This makes the combination of SOE with calorific devices and the creation of reversible solid oxide electrolyzers promising (Hu et al., 2022).

Several devices use both electricity and alternative energy sources to carry out water electrolysis in a hybrid mode. The photo-electrochemical cell is the type of system that is most frequently seen. Presented here are a few pertinent hybrid electrolysis systems. An apparatus known as a photo-electrochemical cell drives water electrolysis and produces hydrogen from water by heterogeneous photocatalysis. Both an electrical power supply (direct current) and a photon source are needed for the system to function (Roh et al., 2024). Sunlight radiation is one possible source of photons, but there are also other potential sources, such as Cherenkov radiation, or blue light, found in nuclear reactor spent fuel pools. An alternative hybrid method for carrying out water electrolysis involves combining a homogeneous catalytic process that occurs in a solution volume with a heterogeneous electro-catalysis process that occurs at the electrode surface. Applications utilizing a liquid electrolyte can employ this method. While homogeneous catalysts operate in tandem with the heterogeneous process, a net increase in hydrogen generation is achieved (Naterer et al., 2013).

Another type of electrochemical devices exists, the electrochemical sensors, but further details about them will be provided in the next chapter.

CHAPTER 3 Electrochemical sensors

The IUPAC defines a chemical sensor as "a device that converts chemical data into an analytically usable signal, ranging from the concentration of a single sample component to complete composition analysis." A chemical sensor is primarily composed of two fundamental functional components: a physicochemical transducer and a receptor. The market for electrochemical sensors is predicted to develop at a compound annual growth rate (CAGR) of 8.5% from 2024 to 2031, from an estimated valuation of USD 11.87 billion in 2024 to USD 21.03 billion by 2031 (<https://www.coherentmarketinsights.com>).

Since they lead the market, electrochemical sensors are the most widely used kind of sensor because of their benefits, which include fast sensing, low detection limits (as low as picomoles), and inexpensive sensing equipment. Electrochemical sensors are available in several form factors, from wearable devices with complete integration to top-bench models. A chemical sensor's use is to provide precise, up-to-date information on the chemical makeup of its environment. Such a gadget would ideally be able to react continuously and reversibly without affecting the sample (Meti et al., 2021).

Electrochemical gas sensors provide a fast, small, sensitive, portable, and quick way to find analytes for a variety of uses, such as air quality, hydrogen safety, clean coal power plants, automobile emissions, and breathing analysis to find chronic illnesses. The mixed-potential-based scheme has some distinctive features among the different detection methodologies in electrochemical (EC) gas sensors, including (a) a simple device architecture; (b) compactness, to fit into critical areas; (c) fast response; and (d) direct voltage read-out, which eliminates the need for additional conditioning circuitry (Murray & Cui2016).

3.1. Basic principles and types

Developing electrochemical sensors presents several significant challenges. Achieving high selectivity is crucial to distinguish the target analyte from interfering substances, requiring the use of highly selective materials or membranes to avoid cross-sensitivity. Stability and longevity are also major concerns, as sensors must resist degradation, fouling, and signal drift over time to ensure reliable performance. Additionally, enhancing sensitivity and lowering detection limits is essential for accurate measurement of trace concentrations, necessitating optimization of electrode materials and sensor design. Addressing these hurdles demands

advances in material science, innovative design, and rigorous calibration to ensure effective and reliable sensor operation (Devnani& Sharma, 2022).

The electrochemical sensor is a stand-alone, integrated instrument that converts data from electrochemical processes into signals suitable for study. Using a chemical recognition element, also called a receptor, positioned in direct spatial contact with the electrode, it is beneficial to offer quantitative or semi-quantitative analytical data. The two-electrode (2E) and three-electrode (3E) systems are the most often used electrode configurations in electrochemical sensors. While the 2E system only has reference and working electrodes, the 3E system's cell has counter, reference, and working electrodes. The electrolyte solution's state, the amount of current flowing through the electrode-solution contact, and the electrochemical method being used all influence the utilization of 2E and 3E systems (Saputra, 2023).

The working principle of electrochemical sensors is based on current that passes via the working electrode and another electrode during a measurement. The reference electrode or another third electrode is referred to by this other electrode. The reference electrode's potential will alter and eventually disappear when it is utilized as the electrode delivering current. Therefore, it is best to avoid employing a reference electrode as a current-carrying electrode. It is true that a third electrode, known as the counter or auxiliary electrode, is needed to complete the circuit and allow current to flow (Saputra, 2023). **Figure 6** depicts the three-electrode system consisting of working, reference, and auxiliary electrodes.

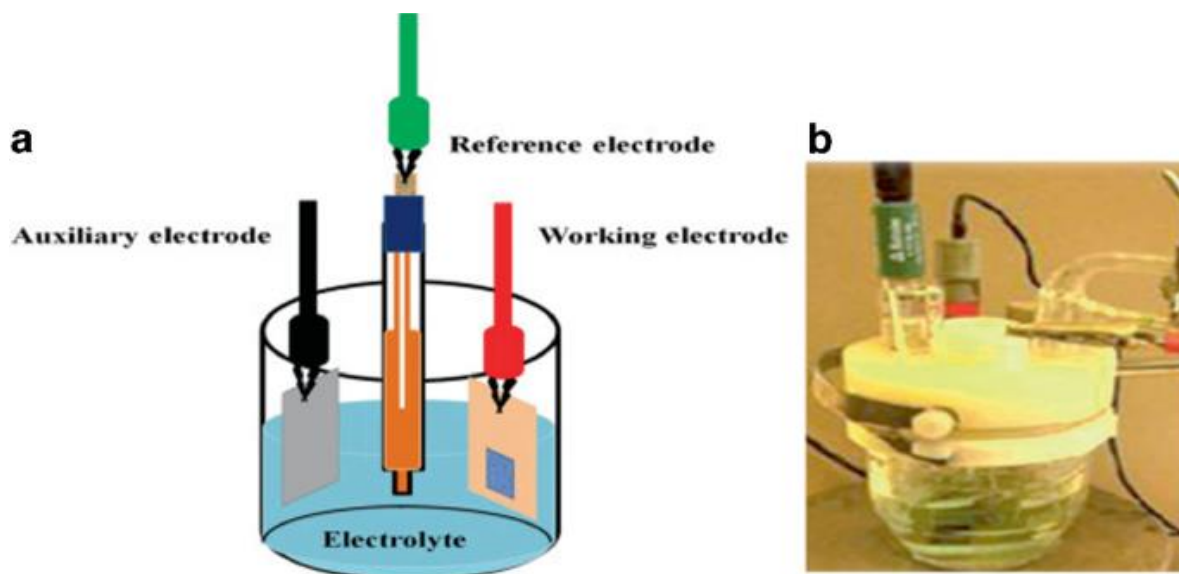


Figure 6. Three-electrode system consisting of working, reference, and auxiliary electrodes (Erkmen et al., 2022).

Electrochemical sensors can be categorized based on their measurement methods into several types: potentiometric, conductometric, voltammetric/amperometric, coulometric, and impedimetric.

3.1.1. Potentiometric sensors

Potentiometric sensors measure the potential difference between an indicator and a reference electrode when no current is flowing, operating under equilibrium conditions. These sensors detect changes in charge accumulation due to selective binding. They can be adapted into biosensors by incorporating biological materials, such as enzymes, which catalyze reactions to produce ions. These ions are then detected when they interact with an ion exchange membrane (Erkmen, et al., 2022).

For many years, pH electrodes have been the most used potentiometric device overall and for a number of decades prior to that. The most popular kind of electrodes are glass electrodes, which come in a range of shapes and sizes and are made from a thin, ion-sensitive glass membrane. However, in addition to those mentioned above, other kinds of potentiometric sensors may be used to measure pH. These sensors use redox-active chemicals (such as quinones and ferrocenes) or organic polymers (like polymethylene blue) (Baranwal et al., 2022).

There are three basic types of potentiometric devices: ion-selective electrodes (ISE), coated wire electrodes (CWES), and field effect transistors (FETS). An ISE is designed to measure the activity of a specific ionic species selectively. ISEs may be classified into three categories based on the type of membrane: glass electrodes, liquid electrodes, and solid electrodes. Typically, these electrodes are membrane-based devices made from ion-conducting materials that are selective for the target ion. The electrode system generally includes two key components: a working electrode, which responds to the ion concentration in the sample, and a reference electrode, which maintains a stable potential through a solution with a fixed ion activity. The potential difference between these two electrodes, known as the cell potential, reflects the concentration of the ion of interest. For an in-depth explanation of the processes occurring at the membrane interface that generate this potential, refer to detailed theoretical resources (De Marco et al., 2007).

CWEs are employed to identify certain ions present in a solution. The core element for electrical conduction in these electrodes is a thin, conductive wire, frequently composed of platinum, silver, or graphite. An ion-selective substance, such as glass or a polymer, is applied to the wire and interacts with the target ion in the solution in a preferred manner. The ion-

selective coating of the CWE produces a potential difference depending on the concentration of the particular ion when it is inserted into the sample. The ion concentration is then ascertained by measuring this potential. The simple design, simplicity of preparation, and versatility of CWEs make them popular for use in a wide range of applications, such as industrial operations, clinical diagnostics, and environmental monitoring (Cunningham et al. 1986).

FET potentiometric sensors utilize the principles of FETs to measure the concentration of specific ions in a solution. These sensors incorporate a field effect transistor with a sensitive gate electrode that interacts with the target ions. To be noted, FETs are a kind of semiconductor device that uses an electric field to regulate the flow of electrical current. They serve as switches or amplifiers and are essential parts of contemporary electronics. Three terminals make up the fundamental design of a FET: the gate, source, and drain. Through the creation of an electric field that modifies the movement of charge carriers in the semiconductor material, the gate terminal controls the conductivity between the source and drain (Kaisti, 2017).

3.1.2. Conductometric sensors

Conductometric sensors measure a solution's ability to conduct electricity by detecting changes in electrical conductivity caused by the presence of an analyte. These sensors work by monitoring how the conductivity of a film or mass is affected by the ionic species in the solution. In conductometric detection, variations in ionic concentration led to changes in conductivity. There are two main types of conductivity detection: contact-mode and contactless. Contact-mode sensors, which have electrodes in direct contact with the solution, offer high sensitivity and quick response times but risk electrode degradation and sample contamination. Contactless sensors, where electrodes are insulated from the sample by a thin layer, avoid these issues but require the insulation layer to be as thin as possible to maintain effective capacitive coupling (Yunus et al., 2013).

These sensors are appreciated for their simplicity, robustness, and ability to provide continuous, real-time measurements. They are effective in a range of applications due to their sensitivity to changes in ionic concentration and ease of calibration.

3.1.3. Voltammetric

Voltammetric sensors measure the current concerning the applied potential, generating a voltammogram by varying the potential either incrementally or continuously. Various voltammetry techniques used in these sensors include cyclic, differential pulse, square wave, linear sweep, stripping, and hydrodynamic voltammetry. These methods are widely used for

analyzing a broad spectrum of samples, including clinical specimens, pharmaceutical products, and environmental samples (Devnani& Sharma,2022).

The voltammetric sensors come in different types depending on the system they rely on. In the electrochemical method known as linear sweep voltage measurement (LSV), the electrode potential is changed linearly over time while the resultant current is monitored. Redox reactions between electroactive substances in the solution and the electrode surface occur when the voltage is ramped up or down at a steady pace in an LSV experiment (Garreau, et al., 1972). Peaks corresponding to the analytes' redox processes are displayed in a voltammogram that is created by recording the current response as a function of the applied voltage. With the use of this technique, electroactive species may be identified and measured by examining their distinctive redox activity. LSV is a vital technique in electrochemical analysis since it is especially helpful for researching reaction kinetics, figuring out a substance's electrochemical characteristics, and comprehending the mechanics behind electrochemical reactions (Yamada et al., 2022).

In the electrochemical method known as cyclic voltammetry, a triangle waveform is created by alternating the electrode potential between two predetermined values. The potential is swept back and forth between these limits throughout a CV experiment to create a voltammogram, which shows the current response in relation to the applied potential. This procedure makes it possible to see the analyte's redox behavior, including its oxidation and reduction peaks. Analyzing reaction mechanisms, comprehending electrode operations, and assessing the stability and reversibility of the analyte are among the applications where CV is most helpful. Researchers can obtain an in-depth understanding of the electrochemical characteristics, kinetics, and dynamics of the species under study by examining the forms and locations of the current peaks in the voltammogram (Elgrishi et al., 2018).

With Differential Pulse Voltammetry (DPV), a sequence of small amplitude potential pulses is superimposed atop a linear sweep to increase measurement sensitivity and precision. Periodic pulses are administered as the electrode potential is scanned linearly during DPV, and the current is monitored at predetermined intervals before and following each pulse. A voltammogram with an enhanced signal-to-noise ratio is produced by plotting the differential current created from the difference between these current measurements versus the applied potential. High accuracy and low detection limits are critical for applications in clinical diagnostics, analytical chemistry, and environmental monitoring, where this technology is

particularly successful in identifying trace quantities of analytes and resolving closely related molecules (Molina & González, 2016).

An improved electrochemical method called square wave voltage measurement, or SWV, delivers square wave pulses to the electrode in which a linear sweep is overlaid with alternating forward and reverse potentials. The current response is monitored during these pulses, with an emphasis on the changes in current that take place during the pulse intervals as opposed to the steady-state sweep. By reducing background noise and enhancing the detection of low-concentration analytes, this method improves sensitivity. Since SWV has a high temporal resolution, it is perfect for accurately and quickly analyzing electroactive species. It is especially helpful for researching dynamic reactions and quick electrochemical processes. Applications ranging from biochemical analysis and industrial quality control to environmental monitoring benefit greatly from its accuracy and efficiency (Mirceski et al, 2007).

3.1.4. Amperometric

Amperometric sensors measure the current generated at a constant applied potential to detect the concentration of an analyte. By fixing the voltage at the working electrode, these sensors monitor the flow of current resulting from electrochemical reactions with the analyte. The current, which is proportional to the analyte's concentration, is recorded over time. This approach provides sensitive and selective measurements, making amperometric sensors effective for detecting low concentrations of substances in various samples, such as glucose in blood or pollutants in environmental samples (Helm et al., 2010' Kalyakin et al., 2020).

3.1.5. Other types of sensors

Coulometric sensors measure the total amount of electricity used for electro-reduction or oxidation, rather than relying on analyte concentration. Coulometry is considered a highly accurate technique because the entire amount of analyte is consumed quantitatively in the electrochemical reaction. This absolute technique does not require chemical standards or calibration, as the amount of analyte is directly proportional to the electricity consumed (D'Orazio & Meyerhoff, 2014).

Finally, impedimetric sensors measure resistance, capacitance, and inductance using a small amplitude alternating current potential across a wide frequency range. The impedance spectrum is obtained by analyzing variations in frequency, with resistive and capacitive components derived from in-phase and out-of-phase AC current measurements. This method is particularly useful for developing affinity biosensors, as it enables the monitoring of impedance

changes related to the charge and mass transfer of analytes at the sensor surface (Sharma et al., 2019).

3.2. Applications

Among the many benefits of electrochemical sensors are their high sensitivity, which allows for low LODs and LOQs; quick analytical response, which makes them perfect for flow analysis and alert systems; simplicity, which permits an almost infinite range of geometries, electrode materials, and configurations and ease of use (low-cost, basic equipment, adaptability as a detection module in a range of analytical systems). The diverse benefits allow also the wide applications of this type of sensors (Baranwal et al., 2022; Yuan et al., 2013).

Applications for electrochemical sensors are many and span many different industries. These sensors are vital to environmental monitoring because they detect gases like ozone and carbon monoxide and evaluate water quality for contaminants like heavy metals and nitrates. Electrochemical sensing devices have a major impact on the monitoring of priority pollutants allowing the instrument to be taken to the sample (rather than the traditional way of bringing the sample to the laboratory). They are employed in industrial processes to detect corrosion to stop equipment damage, monitor pH and oxidation-reduction potential, and regulate chemical processes (Hanrahan et al., 2004).

As a result of continuing scientific study, several hydrogen detection systems have been developed. Among the well-established technologies having a significant influence on the market for sensors is electrochemical hydrogen sensing. These sensors detect hydrogen at high temperatures using solid electrolytes that transport protons and ions. They are often used in a wide range of industrial industries, including steel, automobiles, etc. Solid-state electrolytes are widely used because of their exceptional qualities, which include stability, strong ionic conductivity at high operating temperatures, and resistance to challenging operating environments (Gorbova et al., 2022).

3.2.1. Gas sensing

Particularly useful in situations requiring temperature-sensitive measurements, such as biological assays, food quality control, and environmental monitoring in colder climates, low-temperature electrochemical biosensors are specifically engineered to operate effectively at temperatures below ambient, usually ranging from just above freezing to approximately 20°C. At these lowered temperatures, the kinetics of electrochemical reactions and the behavior of the biological recognition elements are affected, necessitating careful design and optimization.

For low-temperature sensors, the selection of electrode materials is essential. Electrodes made of carbon, conducting polymers, and metal electrodes like gold or platinum are often utilized materials. These materials must continue to function steadily even at lower temperatures. To guarantee that biorecognition components—like enzymes or antibodies—continue to function in cold environments, careful selection and potential modification are also necessary. It may be necessary to employ stabilization methods or specific materials to stop certain components from degrading (Ghaderahmadi et al., 2021).

Low-temperature electrochemical biosensors have a wide range of applications. They are employed in biological assays to guarantee precise readings while maintaining the integrity of biological responses that are temperature sensitive. By examining biochemical changes that occur during cold storage, these sensors assist the food business in tracking freshness and identifying deterioration. They are also used in environmental monitoring to find toxins and pollutants in the air, water, and soil—especially in colder climates. Metal oxide nanohybrids-based low-temperature combines different metal oxides at the nanoscale, due to the enhanced sensing performance, improved surface area, catalytic activity, and electronic properties (Mishra et al., 2019)

The capacity of these sensors to deliver precise measurements in temperature-sensitive applications is one of their key benefits; this reduces the deterioration of delicate biological components and ensures long-term stability. They do, however, also must contend with issues including decreased reaction rates at lower temperatures, which can have an impact on response time and sensitivity. Furthermore, cautious material selection and potentially sophisticated treatment techniques are needed to guarantee that electrode materials and biorecognition components function dependably in cold environments.

High-temperature electrochemical sensors are essential devices designed to function reliably in extreme environments, often found in industrial, environmental, and energy applications. Appropriate detection systems for tracking combustion processes have been developed using a range of methodologies. Using solid electrolytes in electrochemical sensing devices is a strategy that works very well in hot conditions (Fergus, 2007).

These sensors are constructed from robust materials like ceramics and platinum, enabling them to withstand elevated temperatures while maintaining high sensitivity and accuracy. They are particularly valued for their ability to provide real-time monitoring of gases such as oxygen and carbon monoxide, which is crucial in dynamic processes like combustion in engines or industrial furnaces (Liu et al., 2015). The selective detection capabilities of these

sensors, achieved through specific electrode materials and electrolytes, make them ideal for optimizing processes, such as controlling air-fuel ratios in automotive systems or monitoring gas composition in solid oxide fuel cells (SOFCs).

While they offer significant benefits, including durability and precision, challenges such as maintaining calibration, managing power consumption, and higher costs due to specialized materials must be carefully managed. Despite these challenges, high-temperature electrochemical sensors are indispensable for ensuring efficient operation and compliance with environmental standards in demanding settings.

3.2.2. Industries

Electrochemical sensors are used by the food and beverage sector to measure fermentation processes in breweries and wineries, as well as to monitor pH, oxygen levels, and pollutants to assure product quality and safety (Sharifi & Pirsá, 2020). Maintaining food safety and quality is one of the food industry's core concerns. Food safety and quality have traditionally been evaluated using spectroscopic and chemical techniques. The old-fashioned ways are expensive, time-consuming, and arduous. When it comes to monitoring food, the biosensors are a great substitute for the conventional techniques. Sensors are economical, rapid in reaction, selective, and efficient. It has been effectively demonstrated that a biosensor based on cobalt phthalocyanine can track the aging of beer in storage. A bacteria called *E. coli* might be a sign of contaminated food as it is present in vegetables. Its presence in the veggies may be determined by the electrochemical biosensor (potentiometric), which works by simply measuring the pH fluctuation (Singh et al., 2021).

Electrochemical sensors are essential for preserving process control and guaranteeing product quality in the industrial industry. Monitoring pH Level: Precise pH management is critical to chemical manufacturing to maximize reactions and guarantee uniformity in the final product. Real-time measurements of the acidity or alkalinity of solutions are made possible by electrochemical pH sensors, which provide operators the flexibility to quickly change a solution's pH to maintain the proper chemical conditions and avoid process errors.

Electrochemical sensors are also used in wastewater treatment plants to monitor a range of parameters, including dissolved oxygen (DO), chemical oxygen demand (COD), and particular ions or contaminants. By verifying that the water satisfies regulatory requirements prior to discharge or reuse, these sensors aid in evaluating the efficacy of treatment procedures. They

improve the efficacy and efficiency of wastewater management by enabling timely modifications to treatment procedures through the provision of real-time data (Karthik, et al., 2022).

Another sector in which electrochemical sensors gain attention is consumer electronics which are gradually integrating these sensors into wearable devices to monitor physiological data such as sweat glucose levels. These many uses demonstrate how essential electrochemical sensors are to improving efficiency, safety, and health in a variety of fields (Privett et al., 2008).

3.2.3. Medicine

Sensitive and precise methods are crucial for early disease diagnosis, as they significantly impact patient survival and prognosis. Electrochemical biosensor technologies offer an effective solution for point-of-care health assessments, enabling early detection of diseases and monitoring their progression. They are vital to medical diagnostics because they provide vital health information such as measuring cholesterol and blood glucose levels in diabetic patients.

These biosensors are user-friendly, cost-effective, and capable of non-invasive monitoring of treatment outcomes. Their small size and compatibility with electronic modules make them ideal for integration into various biosensing platforms on a single chip. Recently, the use of electrochemical biosensors in healthcare has grown, encompassing applications such as detecting foodborne pathogens, bacterial infections, clinical diagnoses, and respiratory diseases (Yunus et al., 2023). Also, by examining medication formulations and identifying contaminants, they support quality control and drug development in the pharmaceutical sector.

Potentiometric, amperometric, combination (amperometric-potentiometric), and impedance-based gas sensors are the different types of EGSs depending on how they work. Potentiometric and amperometric electrochemical sensors have been discussed earlier. Concerning the combined type of high-temperature electrochemical sensors, they include two electrochemical cells, one of which functions as an amperometric sensor and the other as a potentiometric one. A glass sealant with a high melting point is used to seal the seams that exist between the cells or between the cells and the capillary barrier. In contrast to a single amperometric or potentiometric sensor, the theory of functioning of these devices is more complicated. The first cell operates by pumping the target gas into or out of the sensor chamber while measuring the limiting current. In addition to measuring the EMF, the other cell reports on the target gas's concentration and the amperometric sensor's correct operation (Volkov et al., 2017; Kalyakin et al., 2016).

An alternative method is used by the impedance metric gas sensors to identify the target gas. Impedance-based sensors and mixed-potential sensors have a similar architecture. They are operated by applying a sinusoidal voltage and measuring the current response. The impedance is computed by knowing the applied voltage and the current response. Solid oxide and semiconductor-based sensors are both subjected to impedance-based approaches. There are reports of impedancemetric sensors based on YSZ that can identify steam (Gorbova, et al., 2021).

3.3. Biosensors

3.3.1. Definition and characteristics

A biosensor is an apparatus that uses signals proportionate to the concentration of an analyte in a reaction to quantify biological or chemical responses. Applications for biosensors include illness monitoring, medication development, and the identification of contaminants, pathogen-causing microorganisms, and disease-indicating markers in physiological fluids (blood, urine, saliva, sweat) (Bhalla et al., 2016). These are self-contained integrated devices that employ a biological recognition element connected to a transduction element to deliver qualitative and semi-quantitative analytical data. These analytical tools are designed only to quickly and reliably give real-time, correct information about an analyte of interest (Mehrotra, 2016)

The first reported biosensor is attributed to Leland C. Clark Jr., who developed the Clark electrode in 1962. This pioneering device was designed to measure oxygen levels in the blood, utilizing an amperometric detection method where a platinum cathode and silver anode, separated by an electrolyte and covered with an oxygen-permeable membrane, generated a current proportional to the oxygen concentration. The Clark electrode's innovation demonstrated the feasibility of integrating biological recognition elements with physicochemical transducers for specific and accurate analyte measurements. This invention laid the groundwork for modern biosensor technology, influencing the development of a wide range of biosensors used today in various applications, including medical diagnostics, environmental monitoring, and food safety testing (Tetyana et al., 2021).

A biological event can be immediately converted to an electrical signal with the help of electrochemical biosensors. However, the interface architecture is largely responsible for the signal transduction and the electrochemical sensor's performance. Electron production or consumption is the basis of electrochemical biosensing. Either current is produced by electron transport across the working electrode's electric double layer, or voltage is produced by a

change in the double layer potential. The bio-electrochemical sensor is gaining popularity due to several benefits, including its low cost of development, time-saving nature, basic electrochemical instrumentation requirements, and ease of use without the need for specialized training. They are also adaptable to downsizing and have excellent selectivity, sensitivity, stability, and quick response times (Shanbhag et al., 2023).

Each biosensor has three key components, the bioreceptor, the transducer, and the electronics. The bioreceptor is the biological element of the biosensor, responsible for the specific interaction with the target analyte. Bioreceptors can be enzymes, antibodies, nucleic acids, cells, or other biological molecules capable of recognizing the substance of interest. The selection of the bioreceptor depends on the nature of the target analyte and the desired specificity and sensitivity of the biosensor. For example, enzymes are commonly used in glucose sensors due to their ability to catalyze specific reactions with glucose. Antibodies are used in immunosensors for their high specificity in binding to particular antigens. The bioreceptor's primary function is to provide a selective and sensitive recognition of the analyte, ensuring accurate measurement (Wang et al., 2024).

The transducer is the component that converts the biorecognition event into a measurable signal. It acts as a bridge between the biological and electronic parts of the biosensor. Transducers can operate through various mechanisms, including electrochemical, optical, piezoelectric, and thermal methods. Electrochemical transducers, for instance, measure changes in current, voltage, or impedance resulting from the biochemical interaction at the bioreceptor. Optical transducers detect changes in light absorption, fluorescence, or reflectance. Piezoelectric transducers sense changes in mass or acoustic waves on a surface. The choice of transducer depends on the nature of the bioreceptor-analyte interaction and the required sensitivity and detection range. The efficiency and accuracy of the transducer are crucial for translating the biorecognition event into a quantifiable signal (Yadav et al, 2023).

The signal processor is the final component of a biosensor, responsible for amplifying, processing, and displaying the signal generated by the transducer. This component includes the electronic circuitry and software necessary for signal amplification, noise reduction, data processing, and result presentation. In modern biosensors, signal processors are often integrated with microcontrollers and digital interfaces to enable real-time data analysis and connectivity with other devices, such as computers or smartphones. The signal processor ensures that the output signal is clear, accurate, and interpretable, allowing for precise quantitative analysis of the target analyte. Advanced signal processing algorithms can also

enhance the sensitivity and specificity of the biosensor by filtering out background noise and correcting for potential interferences (Mohanty & Kougianos, 2006). **Figure 7** depicts the configuration and working principle of an electrochemical sensor as described above.

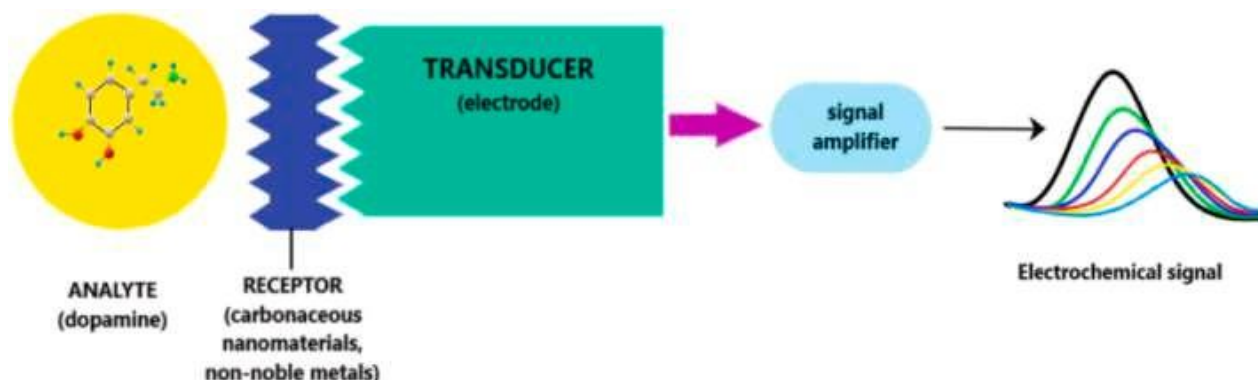


Figure 7. Principle of operation of a dopamine electrochemical biosensor (Balkourani et al., 2023)

The key characteristics of a biosensor include specificity, sensitivity, range, accuracy, precision, response time, stability, reproducibility, portability, and cost-effectiveness. Sensitivity is crucial for detecting low-abundance analytes, while a broad dynamic range allows for accurate measurement across various concentrations. High accuracy and precision ensure reliable and reproducible results, with fast response times enabling real-time monitoring. Stability and reproducibility guarantee consistent performance over time, while portability and cost-effectiveness enhance their usability in diverse settings, including field and point-of-care applications. User-friendliness, miniaturization, and connectivity further improve their practicality, allowing for easy operation, integration into compact systems, and real-time data sharing via digital platforms (Bhalla et al, 2016; Naresh& Lee, 2021).

3.3.2. Types of biosensors

3.3.2.1. Electroanalytical

Since dopamine is rapidly oxidized, it results in the creation of dopamine-o-quinone by a two-electron mechanism, a variety of electrochemical techniques have been developed for the measurement of dopamine. To be noted, the oxidation peak of dopamine occurs at 150 mV (vs Ag/AgCl). Examples of these methods include amperometry, cyclic voltammetry, and differential pulse voltammetry. The measurement of these substances is made possible by the electrons generated by dopamine during its oxidation, which produces currents that may be linearly dependent on the concentration of the electroactive dopamine biomolecules (Lakard et al., 2021).

Electroanalytical methods offer numerous advantages for dopamine detection, making them a valuable tool in both research and clinical applications. These methods are highly sensitive, capable of detecting extremely low concentrations of dopamine, and they provide rapid response times, allowing for real-time monitoring of neurotransmitter dynamics. The equipment used is relatively low-cost and straightforward, which, combined with the potential for miniaturization, supports the development of portable and implantable devices for continuous monitoring. Additionally, electroanalytical techniques can be tailored for high selectivity, ensuring accurate detection of dopamine even in complex biological samples. They also facilitate quantitative analysis and are versatile, and compatible with various formats such as cyclic voltammetry and amperometry. Furthermore, electroanalytical methods are environmentally friendly, as they typically require fewer reagents and solvents, making them an efficient and sustainable choice for dopamine detection (Lakard et al., 2021).

3.3.2.2. Voltametric

Voltametric biosensors are sophisticated electrochemical instruments that identify and measure target analytes using voltammetric methods. These sensors measure the current that results from applying a different voltage to an electrode, which represents the analyte's electrochemical characteristics. Important techniques for measuring voltage include square wave, differential pulse, cyclic, and linear sweep voltammetry. The selection of electrode materials, which can include conducting polymers, metal electrodes, and carbon-based electrodes, has a significant impact on the performance of these sensors. To improve selectivity and sensitivity, electrode surfaces are frequently modified with biological recognition components such as enzymes, antibodies, aptamers, or molecularly imprinted polymers (Wuet al., 2023).

Carbon-based electrodes have been extensively used in voltametric studies due to several advantages, such as low cost, availability, stability, and the ease with which carbon's morphology can be modified. Various types of carbon-based electrodes exist, including glassy carbon (GC), polycrystalline boron-doped diamond (pBDD), carbon nanotubes (CNTs), and, more recently, graphene. The use of carbon as an electrode tip has numerous advantages including mechanical strength and inhibition of water electrolysis (Kimmel et al., 2012).

A voltametric dopamine biosensor can work in numerous modalities, including differential pulse voltammetry, linear sweep voltammetry, square wave voltammetry, and cyclic voltammetry. The corresponding current–potential characteristics and the sensitivities of each

type are different. Most voltametric dopamine biosensors measure current–potential responses based on variations in dopamine concentration. For instance, dopamine oxidation using cyclic voltammetry results in an oxidation peak, which is unique to a particular biomolecule. It is feasible to create calibration curves and measure this substance by examining the development of the oxidation peak's strength in cyclic voltammograms for various dopamine concentrations (Ding et al., 2020).

Typically, the binding kinetics of dopamine to the active sites of the immobilized bioreceptor and the mass transfer rate of the dopamine to the working electrode surface significantly affect the current–potential behavior of the dopamine biosensor. The mass transfer limiting current can be achieved by bulk transfer, diffusion, and ionic migration. The kinetics of the reaction between dopamine and the biorecognition element and the change in the mass transfer process of dopamine on the bioreceptor-modified electrode give rise to the faradaic process in an electrochemical system (Zhao et al., 2019).

For the *in vivo* monitoring of dopamine in the brain, fast-scan cyclic voltammetry is a widely used and effective voltammetric approach. High temporal resolution is provided by this method without compromising its exceptional selectivity. By utilizing this method, one may examine fast fluctuations in dopamine levels in the brain by increasing the applied potential at a high scan rate ($>100 \text{ Vs}^{-1}$) over the potential range of interest. Using a triangle waveform formed by a linearly ramping electrode potential swept in both directions over time, fast-scan cyclic voltammetry records the resultant current, which is directly proportional to the quantity of dopamine present in the sample. When outfitted with high temporal and spatial resolution, fast-scan cyclic voltammetry might provide (Venton et al., 2020).

This rapid scanning method effectively minimizes membrane fouling and limits the molecular diffusion distance to the electrode surface. To accurately isolate the faradaic signal from the capacitive component that forms at the electrode-electrolyte interface, careful background subtraction is necessary. This is usually achieved through techniques such as principal component analysis, Hilbert transforms, or the asymmetric least squares smoothing method. Among the materials used for modified electrodes, carbon nanofibers, and polypyrrole are currently the most prevalent nanomaterials for detecting and quantifying dopamine in biological samples (Koehne et al., 2011).

By superimposing a sequence of consistent voltage pulses on top of the potential linear sweep, differential pulse voltammetry, DPV, is a variation of linear sweep voltammetry. The electrode in DPV is subjected to a base potential that is selected so that there is no faradaic

reaction. Equal increments are made to the base potential in between pulses. Before every potential shift, the current is measured, and the difference between the current and the potential is shown. To lessen the impact of the charging current, sample the current right before the potential is changed. An oxidation process creates a wave in the voltammogram when using the linear sweep approach, but it creates a peak when using the DPV technique (Westbroek, et al, 2005). This is especially helpful for electrochemical biosensors because, when using the DPV approach, it is simpler to distinguish between the sharp peaks that are thought to be caused by dopamine, ascorbic acid, or uric acid than it is to distinguish between the vast waves that are produced by linear voltammetry (Venton et al., 2020).

3.3.2.3. Amperometric

Amperometric biosensors are a particular type of voltammetric biosensors, where the current is measured during the time at a constant potential. Modern electrochemical devices known as amperometric biosensors detect current flow in response to an applied voltage that is created when a target analyte interacts with a biological recognition element. The fundamental idea is to provide a constant voltage to an electrode, which will cause the analyte to undergo oxidation or reduction in an electrochemical process. The quantity of the target material is found by measuring the resultant current, which is directly proportional to the analyte's concentration (Kalyakin et al., 2016).

Kalyakin et al., designed a sensor for oxygen detection that is made up of two main parts: (i) an electrochemical cell that acts as an amperometric sensor, designed to pump out oxygen from the sensor chamber, and (ii) a second electrochemical cell that measures the oxygen concentration in the chamber, functioning in potentiometric mode. The gas being analyzed serves as the test gas for the second cell, which operates under a potentiometric regime. This second cell also allows the monitoring of the sensor's internal oxygen levels. The presence of the potentiometric cell improves the sensor's reliability by enabling simultaneous measurement of both the limiting current in the pump cell and the electromotive force in the potentiometric cell. The sensor, as constructed, is highly effective, delivering quick and precise responses for gas mixtures with oxygen concentrations between 0.8% and 88% at operating temperatures from 400 - 700 °C (Kalyakin et al., 2020).

These sensors make use of a variety of electrode materials, including metal electrodes like platinum and gold as well as carbon-based electrodes like glassy carbon and carbon nanotubes. Certain biorecognition components, such as enzymes, antibodies, or aptamers, are

frequently added to electrode surfaces to improve selectivity and guarantee accurate interaction with the target analyte (Hammond et al., 2016).

Amperometry records the current as a function of time and can be used to determine the amount of dopamine in a sample by applying a constant potential between the working and counter electrodes that is sufficient for dopamine oxidation. This means that the quantity of dopamine is directly proportional to the amplitude of the observed amperometric current. Amperometric current may be generated on time scales less than milliseconds using electrodes modified with bioreceptors with relative ease, as the common sampling rate is in the range of kHz (Rahman et al., 2016).

Chronoamperometry, an advanced form of conventional amperometry, involves applying a square-wave potential to a bioreceptor-modified electrode and measuring the resulting current generated from the oxidation or reduction of target chemical species over time. This electroanalytical technique has been extensively used in studies to measure dopamine levels in the cerebral fluid of the brain. Despite its widespread application, chronoamperometry faces significant challenges related to chemical selectivity (Lakshmanakumar et al., 2021).

In a study by Bavatharania et al., a chronoamperometric biosensor was developed to quantify dopamine using a combination of silica, polydiphenylamine, and graphene nanosheets as biorecognition elements. This biosensor successfully differentiated dopamine from other electroactive interferences, such as acetaminophen and glucose. With a regression coefficient (R^2) of 0.98, the nanohybrid electrode responds linearly to DA (1–5 μM) in the presence of phosphate buffer solution (PBS at pH 7.0). Dopamine was shown to have a low detection limit of 0.1 μM and a superior sensitivity of 0.66 $\mu\text{A } \mu\text{M}^{-1}\text{cm}^{-2}$, respectively (Bavatharani et al., 2020).

3.3.2.4. Potentiometric

Potentiometric sensors operate by measuring the open circuit voltage (OCV, E), where the electrolyte with reversible electrodes functions as a concentration cell. This cell is exposed to a gradient in the chemical potentials of the ionic species (Volkov et al., 2017). Up to now, potentiometry has become the standard technique in the clinical analysis of ions, particularly the determination of the biologically relevant electrolytes in physiological fluids or even in cells such as sodium, potassium, and calcium (Ding & Qin, 2020).

With potentiometric sensors, quasi-null current flow conditions are reached to detect the potential difference between an indicator electrode and a reference electrode using a very

high impedance voltmeter (Yunus et al. 2012). The potential of the indicator electrode fluctuates based on the activity of an ion in the sample to be analyzed, while the reference electrode maintains a constant potential. The Nernst equation, which links the potential difference between reference and indicator electrodes, E , to the actions of the species causing the response, contains the fundamentals of potentiometric measurements (Yunus et al., 2012). In hydrogen sensors, the difference in partial pressures of hydrogen in each compartment results in varying chemical potentials due to the differing concentrations of hydrogen ions. This generates an electromotive force (EMF) within the cell. The EMF, representing the sensor's open circuit voltage (OCV), is the potential difference between the SE and RE and can be determined using the Nernst equation (Kalyakin et al., 2014).

The main challenge with potentiometric biosensing is achieving selectivity. There has been a great deal of recent advancement in biosensing, from the novel use of materials both old and new to improved potentiometric sensors that expand the range of available analytes to the use of new sensing devices to drastically improve sensitivity. An important trend has emerged in the push toward portable, self-powered, and wearable biosensors for use in technologically limited locations or point-of-care devices (Walker et al., 2021).

Wearable potentiometric sensors (WPIs) offer a novel approach to physiological and clinical monitoring by integrating chemical, material, and electronic features for on-body measurements. These electrochemical systems, based on ion-selective electrodes, are designed to measure the concentration and activity of critical ions in body fluids. WPIs are versatile and can be configured as sweatbands, textiles, or epidermal patches, providing comprehensive data on the body's physiological activities. Their ability to continuously monitor biomarkers in real time makes them a valuable tool for health management and clinical diagnostics. Parrilla et al. reported a novel biomarker detection tool for ion sensing, the ion patches in the human body (Sweat in this case) using WPIs technology. The device could detect Cl^- , K^+ , and Na^+ ions as well as the pH activity can be all detected by the proposed WPIs. (Parrilla et al., 2019).

Molecularly imprinted polymers (MIPs) have emerged as an innovative approach to enhance the performance of analytical sensors. These sensors utilize a "lock and key" mechanism where MIPs are designed with specific cavities tailored to fit particular target molecules. This customization ensures a high level of selectivity and affinity for the target, which significantly improves the sensitivity of the sensing system. The polymeric structures of MIPs contain active sites that interact specifically with the target molecules, thereby enhancing the accuracy and efficiency of biosensing applications (Gui & Jin, 2019).

4.1. General information

4.1.1. Metabolism

The biosynthesis of dopamine initiates from tyrosine. Tyrosine hydroxylase (TH) hydroxylates L-tyrosine at the phenol ring to produce DOPA, which is the first step in the two-step production of DA that occurs in the cytoplasm of CAergic neurons. This oxidation is tightly controlled and requires the cofactor tetrahydrobiopterin (BH₄), which is produced by GTP cyclohydrolase. Aromatic amino acid decarboxylase then converts DOPA to DA. The vesicular monoamine transporter 2 (VMAT2), easily sequesters DA into synaptic vesicles. The acidic pH inside these vesicles stabilizes oxidation-prone DA and avoids oxidative stress in the cytosol (Meiser et al., 2013).

Catechol-O-methyl transferase (COMT) and monoamine oxidase (MAO) are the enzymes responsible for the enzymatic breakdown of DA to its inactive metabolites. The MAO isoforms MAO-A and MAO-B are capable of carrying out this degradative function. Whereas MAO-A is mostly present in catecholaminergic neurons, such as the cells of the SN, MAO-B is primarily located in astrocytes. Dopamine is broken down by MAO into 3,4-dihydroxyphenylacetaldehyde (DOPAL), which is then broken down by the enzyme aldehyde dehydrogenase to generate 3,4-dihydroxyphenylacetic acid (DOPAC). It should be mentioned that glial cells are the primary source of COMT expression. This enzyme is either absent from neurons or present in extremely small amounts. The enzyme COMT is involved in another process in which dopamine (DA) is metabolized, yielding 3-methoxytyramine (3-MT). After that, 3-MT is converted to HVA by MAO and excreted in the urine (Juárez Olguín et al., 2016).

4.1.2. Role in health and disease

Under physiological conditions in nervous and bodily fluids, the normal concentration of dopamine is between 10 and 1000nM. In the brain, dopamine is central to the reward and pleasure system. It is released in response to rewarding stimuli such as food, sex, and social interactions, thereby reinforcing behaviors that are beneficial for survival and reproduction. Key areas involved in this system include the ventral tegmental area (VTA), which produces dopamine and releases it into other brain regions, the nucleus accumbens, which is critical for

experiencing pleasure and reward, and the prefrontal cortex, which is involved in decision-making and behavioral regulation in response to rewards (Lewis et al., 2021).

Motivation and drive are also significantly influenced by dopamine. This neurotransmitter drives goal-directed behaviors and the willingness to exert effort for potential rewards. Abnormal dopamine function can lead to conditions like depression, where motivation is diminished, or addiction, where motivation is pathologically heightened. In addition to its role in motivation, dopamine is essential for motor control, primarily through the nigrostriatal pathway. Neurons in the substantia nigra produce dopamine and send it to the striatum, where it modulates motor activity. Deficits in this pathway are a hallmark of Parkinson's disease, characterized by tremors, rigidity, and bradykinesia (Bromberg-Martin, et al., 2010).

Cognitive processes, including attention, memory, and problem-solving, are also affected by dopamine. It modulates synaptic plasticity, which is the ability of synapses to strengthen or weaken over time and is essential for learning and memory formation. Additionally, dopamine influences emotional regulation- imbalances in dopamine levels are implicated in psychiatric conditions such as schizophrenia and mood disorders like bipolar disorder (Westbrook& Braver, 2016).

In the body, dopamine has significant effects on the cardiovascular system. It can act on different receptors to either dilate or constrict blood vessels, influencing blood pressure and heart function. At low doses, dopamine can lead to vasodilation, particularly in the kidneys, enhancing renal blood flow and increasing urine output. At high doses, it tends to cause vasoconstriction, increasing blood pressure. Dopamine also plays a role in hormonal regulation by inhibiting the release of prolactin from the anterior pituitary gland, thus influencing reproductive and metabolic processes. It impacts the release of other hormones, such as growth hormone and thyroid-stimulating hormone (TSH) (Klein et al., 2019).

In the gastrointestinal tract, dopamine helps regulate motility by modulating the activity of the enteric nervous system, affecting gut motility and function. Dopaminergic drugs are sometimes used to treat conditions like gastroparesis, which is delayed stomach emptying. Emerging evidence also suggests that dopamine may play a role in modulating immune responses, with dopamine receptors present on immune cells influencing their activity and impacting inflammation and immune responses (Serio et al., 2023).

Clinically, dopamine's roles have significant implications. There are several key dopaminergic pathways in the brain, each associated with different functions. The mesolimbic pathway is involved in reward, pleasure, and reinforcement learning, with dysfunction in this

pathway linked to addiction. The mesocortical pathway plays a role in cognition, executive function, and emotional regulation, with dysregulation contributing to conditions like schizophrenia. The nigrostriatal pathway is critical for motor control, and degeneration of neurons in this pathway leads to Parkinson's disease. The tuberoinfundibular pathway regulates the release of prolactin, with dysregulation leading to hyperprolactinemia, affecting reproductive and metabolic functions.

Parkinson's disease, characterized by the degeneration of dopamine-producing neurons in the substantia nigra, leads to motor deficits, with treatments often involving dopamine precursors or agonists (Latif et al., 2021). Schizophrenia is associated with dysregulation of dopamine in the mesolimbic and mesocortical pathways, with antipsychotic drugs typically targeting dopamine receptors to mitigate symptoms. Imbalances in dopamine are also implicated in mood disorders such as depression and anxiety, with treatments aimed at influencing dopamine levels. Additionally, drugs of abuse often hijack the dopamine reward system, leading to reinforced drug-seeking behavior and addiction, with treatment strategies aiming to normalize dopamine function (Franco et al., 2021).

Sweat, blood, and urine are easily accessible fluids that are easier to test, but they might have low and fluctuating concentrations and give less accurate results than measures taken at the manufacturing site. Dopamine levels in the brain may be measured in vivo using implantable biosensors equipped with minimally invasive microelectrodes, which can also be used to track the development of neurodegenerative illnesses (Lakardet al., 2021).

4.2. Traditional methods for dopamine detection

Most detection methods rely on the different chemical characteristics of dopamine. Dopamine can be difficult to detect since it has many analogues and certain possible interferences. Dopamine (pKa 8.9) has a positive charge at pH 7.0 and is easily oxidized, which is why several electrochemical techniques have been developed. Polydopamine may be synthesized via self-oxidation and polymerization in an alkaline environment, which turns the compound brown (Liu & Liu, 2021). Dopamine's UV-Vis absorption peak is located at 300 nm, and after oxidation, a new peak at about 350 nm. appears. Dopamine's fluorescence was examined between pH 2.0 to 12.0, with a greater emission noted at pH values below 6-7. At the ideal pH of 3.6, the fluorescence emission was observed at 315 nm with excitation occurring at 279 nm. Recently, dopamine was imaged using multi-photon microscopy with an excitation wavelength of 540 nm (Maity & Maiti, 2018).

A dialysis microprobe in the targeted brain area collects chemicals diffusing across a semi-permeable membrane and constantly perfuses a solution resembling cerebrospinal fluid. Mass spectrometry (MS) and high-performance liquid chromatography (HPLC) can then be used to separate and evaluate the gathered materials. Microdialysis has two key features: high molecular specificity that allows DA to be distinguished from other NMs with a similar structure, such as norepinephrine (NE), and high sensitivity that allows DA to be detected at nanomolar quantities. Microdialysis does, however, have many drawbacks, such as a low sample rate (on the order of minutes) that makes it difficult to record quick DA release events and a constrained spatial resolution due to the size of the probe (Zheng & Li 2023).

To assess DA release, Millar and Wightman devised and popularized the FSCV in the 1980s. It is predicated on the redox characteristics of DA, which at the surface of carbon-fiber microelectrodes may be reversibly oxidized to dopamine-o-quinone, producing an electrochemical current with a distinct form (Venton and Cao, 2020). In contrast to microdialysis, FSCV employs tiny probes with a diameter of less than 10 μm , which avoids tissue damage, and offers outstanding temporal precision on the scale of sub-seconds. Recently, tissue-like graphene-elastomer composite and FSCV have been combined to create a novel sensor known as Neurostring. FSCV has limited applicability in brain areas where DA and NE are interlaced due to its weak molecular specificity, which makes it ineffective in differentiating between the two chemicals (Schwarz et al., 2015). Furthermore, FSCV does not offer precise spatial information, in contrast to imaging-based methods as they are explained below (Schwarz et al., 2015).

Sulzer's fluorescent false neurotransmitters (FFNs) provide a less direct way to gauge the release of DA. As a fluorescent substrate for the neuronal vesicular monoamine transporter 2 (VMAT2), which may be taken up into the vesicles and expelled during exocytosis, FFN511 is the first FFN that has been described. FFNs have been enhanced with various characteristics. For instance, the signal-to-noise ratio (SNR) of pH-sensitive FFN102 is noticeably higher (Meszaros et al., 2018). According to Pereira et al. (2016), FFM200 is more selective when labeling dopaminergic neurons and can image exocytosis from a single presynaptic bouton of a dopaminergic neuron. Although FFNs have a relatively modest temporal resolution (seconds), they offer great spatial resolution for tracking DA release. Especially, the *in vivo* use of these dyes presents significant challenges because of the intricacy of dye transport and their less-than-ideal spectrum, which is mostly found in the blue-green region (Pereira et al., 2016).

Chromatography is a commonly utilized technique for separation, in which a mobile phase is used to dissolve the sample solution to be tested for DA, and then the stationary phase is passed through it. There will be a separation in the sample because various compounds will travel at different rates. Mass spectrometry is frequently used in conjunction with chromatography to identify substances, such as DA and determine their amount based on the mass-to-charge ratio. The most dependable, accurate, and exact combination is high-performance liquid chromatography-mass spectrometry (HPLC-MS), however, it is costly and time-consuming to use (Burns et al., 2023).

4.3. Catalytic biosensors

Enzymes or enzyme-mimetic catalysts are used in catalytic biosensors for dopamine to selectively identify dopamine and catalytically convert it to dopamine-o-quinone by speeding up the rate of dopamine oxidation. The majority of interfering chemicals that are not oxidized or reduced in that low potential range are less effective when the enzymatically produced dopamine-o-quinone is detected electrochemically at a low applied potential.

The biological or biomimetic receptor is integrated by catalytic biosensors onto the surface of an electrochemical transducer, which is usually a glassy carbon electrode (GCE), screen-printed electrode (SPCE), carbon fiber membrane electrode (CFME), or other kinds of microelectrode platforms. Open-cavity carbon-based nanopipettes, which are capable of obtaining a detection limit of 56 nM (LOD) on brain sections, can be utilized in addition to solid electrodes to increase the spatial resolution of dopamine assays (Yang, et al., 2019).

Traditionally, the transformation of phenolic substances into their corresponding quinone counterparts is catalyzed by tyrosinase and laccases. Polyphenol oxidase, also known as tyrosinase, is a copper-containing metalloenzyme that catalyzes the o-hydroxylation of monophenols to o-diphenols and then to o-quinones. The laccase family of copper-based enzymes, also referred to as multi-copper oxidases (MCOs), is specific to phenolic catechol amines. With the use of these enzymes, dopamine may be indirectly detected by measuring the reduction of o-dopaquinone back to dopamine by a $2e^-/2H^+$ redox process at a voltage of around -0.1 V versus Ag/AgCl reference under physiological conditions (Agarwal et al., 2019; Janusz et al., 2020).

4.4. Electrochemical sensors for dopamine detection

Based on size, electrodes are often divided into two categories: standard electrodes and microelectrodes, each having unique benefits and drawbacks. Because they are bigger,

conventional electrodes frequently have limited sensitivity for dopamine detection and may unintentionally change or destroy dopamine in blood serum, rendering them inappropriate for in vivo monitoring. On the other hand, because of their small size, microelectrodes that have at least one dimension less than 25 μm provide better performance. Their reduced time constant allows for quicker reaction times; higher signal-to-noise ratios yield more readable readings; better mass transfer rates lead to more effective reactions; and larger steady-state current densities yield stronger sensitivity. Microelectrodes are more useful for accurate and consistent detection of neurotransmitters like dopamine due to these features (Chen, et al., 2020).

One major issue in developing electrochemical sensors for detecting dopamine in biological samples is interference from other electroactive substances, such as ascorbic acid (AA) and uric acid (UA). Both compounds are present in body fluids and can complicate dopamine quantification. Ascorbic acid has a similar oxidation potential to dopamine and can even regenerate dopamine by converting its oxidation products back to dopamine. Additionally, ascorbic acid is found in much higher concentrations than dopamine, further complicating detection (Kocot et al., 2017). Uric acid, another commonly found substance, is electrochemically active and present in higher concentrations than dopamine. It can overlap in potential with dopamine at higher concentrations and its antioxidant properties can inhibit the oxidation of dopamine, leading to inaccurate measurements. To address these challenges, sensor development often involves surface modifications using biological molecules like enzymes, antibodies, or aptamers to enhance dopamine detection. However, these modifications can be time-consuming and slow down the production process (de Oliveira Filho et al., 2023).

Despite advancements, existing electrochemical sensors still face challenges with selectivity and high limits of detection (LOD). To address these issues, the study conducted by He et al., introduced a heterogeneous electrode interface designed to enhance sensor selectivity through distinct charged regions. The electrode, fabricated on a flexible polyolefin substrate, is modified with negatively charged graphene (GP) and neutral gold nanoparticles (Au NPs). Experimental results demonstrate that the optimized electrode, composed of five layers each of GP and Au NPs (GP5AuNPs5), effectively detects dopamine (DA) and uric acid (UA) in solutions containing ascorbic acid (AA) with minimal interference. The GP5AuNPs5 sensor achieves low LODs ($3\sigma/S$) of 10 nM for DA and 1.47 μM for UA, making it suitable for detecting low concentrations found in the human body. Additionally, the presence of Au NPs improves the

separation of current peaks, likely due to electrostatic force differences and enhanced diffusion effects provided by the Au NPs (He et al., 2021).

4.4.1. Transistors

Organic Field-Effect Transistors (OFETs) operate on the same fundamental principle as conventional Field-Effect Transistors (FETs), including MOSFETs, but with an organic semiconductor instead of an inorganic one. In OFETs, the channel charge density is regulated by the gate bias, similar to traditional FETs. However, unlike MOSFETs, which use an inversion channel, OFETs utilize an accumulation channel, often composed of holes in a p-type semiconductor activated by a negative gate voltage. In an OFET biosensor, the transistor's function is influenced by a constant external gate bias, and the variations in electric charge on the gate are directly proportional to the concentration of the analyte. These changes cause deviations in the drain-source current and the threshold voltage of the transistor. Often, the FET component is physically separated from the receptor membrane, which is either placed over the gate insulator or substrate, functioning as an Extended Gate OFET (Ravariu, 2023).

A study by Ohshiro et al., presented an extended-gate organic field-effect transistor (OFET)-based biosensor designed for detecting dopamine in human urine. The sensor is functionalized with a laccase-linked mediator, specifically an N-ethylphenazonium moiety, which oxidizes dopamine, triggering an electron relay on the extended-gate electrode. This process causes measurable changes in the transistor's characteristics. The OFET sensor, enhanced with an enzyme-linked self-assembled monolayer, demonstrates selective detection of dopamine, even in the presence of interferents. The sensor's limit of detection is 0.029 ppm (0.19 μM), indicating its potential for effectively monitoring dopamine levels in urine, with the typical urinary dopamine concentration being below 0.39 ppm (2.5 μM) (Ohshiro et al., 2023).

Another study presented a sensor array of microscale OECTs utilizing PEDOT/PSS as the channel material, which demonstrated high sensitivity and selectivity for detecting DA with a platinum (Pt) pseudo-reference gate electrode. The OECTs achieved an impressive limit of detection as low as 1 nM and exhibited high selectivity against uric acid and ascorbic acid. Furthermore, the researchers improved the selectivity of the OECTs at high concentrations (up to 100 μM) by modifying the surface of the Pt gate electrode with chitosan, thereby expanding the detection range. The biosensor is given below in **Figure 8** (Li et al., 2024).

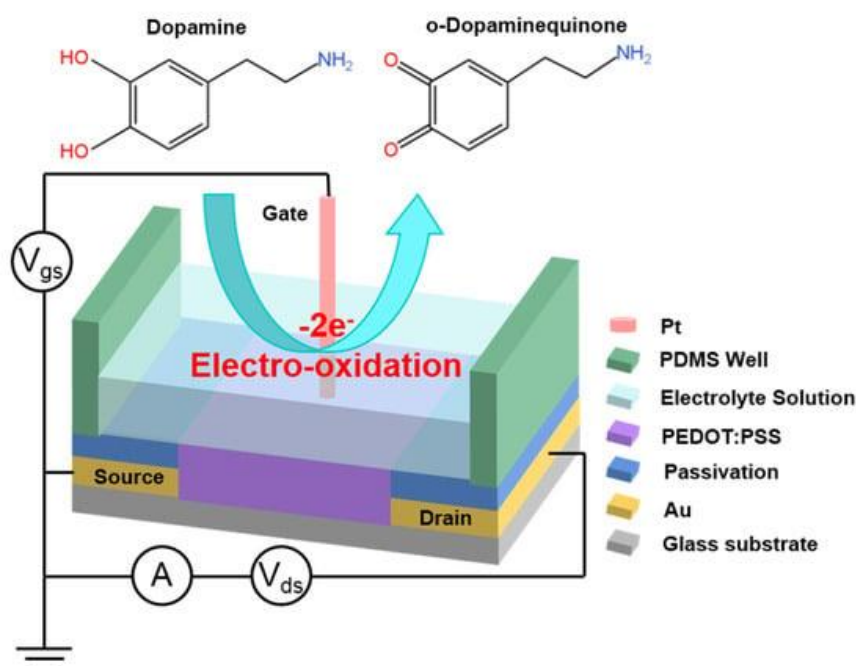


Figure 8. Microscale OECTs for DA detection (Li et al., 2024).

Li et al., reported an ultrasensitive (sub-ppb level) sensor that exploits n-type monolayer molecular crystals (MMCs) with porous two-dimensional structures. Thanks to the monolayer crystal structure of $\text{NDI}_3\text{HU-DTYM}_2$ (NDI) and the controlled formation of porous structure, a world-record detection limit of NH_3 (0.1 ppb) was achieved. Moreover, the MMC-OFETs also enabled direct detection of solid analytes of biological amine derivatives, such as dopamine at an extremely low concentration of 500 ppb (Li et al., 2020).

Xi et al., designed an all-carbon solution-gated transistor (AC-SGT) for dopamine (DA) sensing, utilizing Nafion-coated nitrogen and oxygen-co-doped carbon yarn (Nafion/NOCY) as the gate electrode and graphene as the channel. The AC-SGT's carbon materials allow for various strategies to enhance electrochemical sensing, including modifications to the gate electrode and adjustments to the operating voltage. Operating at a low gate-source voltage of 0.02 V and a drain-source voltage of 0.05 V, the AC-SGT demonstrated exceptional DA sensing capabilities, including high sensitivity, selectivity, a limit of detection of 3 nM long-term stability over 30 days, and a preconditioning time of 60 seconds. Additionally, the researchers integrated the AC-SGT into a smartphone-controlled portable sensing system, showcasing the effective potential of all-carbon transistors for smart wearable biosensor applications. The developed sensor is depicted in the next figure, **Figure 9** (Xi et al, 2023).

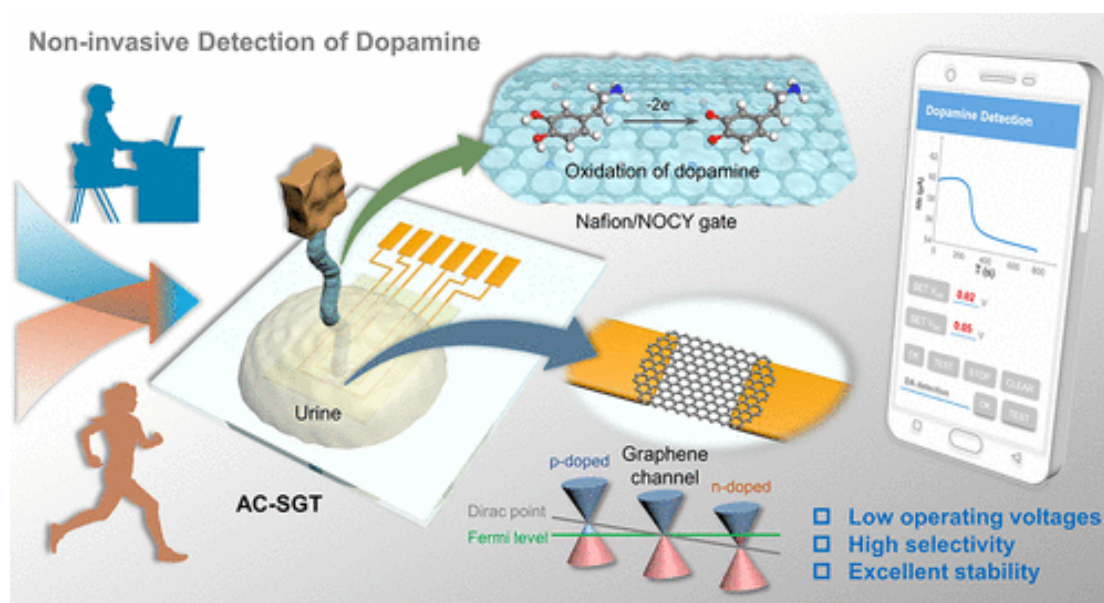


Figure 9. The wearable biosensor for DA detection (Xi et al, 2023).

A promising approach was proposed by Casalini et al. They described a potentiometric sensor based on an Electrolyte-Gated Organic Field-Effect Transistor (EGOFET) designed for the "in vitro" detection of dopamine. The device's sensing element was located at the interface between the gold gate and the aqueous solution, utilizing a self-assembled monolayer composed of cysteamine and 4-formylphenyl boronic acid. The covalent and selective adsorption of DA generated a surface dipole potential, which shifted the electrode's work function and modulated the double-layer capacitance. As a result, the device was able to detect DA at concentrations as low as the pico-molar level, demonstrating greater sensitivity compared to other methods (Casalini et al., 2013).

4.4.2. Polymers

Conducting polymers are affordable, biocompatible, and offer excellent electrical conductivity and high electron affinity. Conducting polymers have attracted significant interest in the health sector, especially after the remarkable conductivity of polyacetylene was discovered. Due to polyacetylene's instability in air, various CPs such as polyaniline (PANI), polypyrrole (PPy), polythiophene (PTh), and polyfluorene (PF) were developed. The chemical properties of CPs are determined by their chain sequences, while their physical characteristics are influenced by chain length. CPs are synthesized through the oxidation of monomers using oxidizing agents or by applying current through electrodes, with their conductivity and other properties adjustable by varying the electrochemical synthesis parameters. Furthermore, the

integration of graphene-based electrodes with CPs has been widely studied for electrochemical sensing applications (Balkourani et al., 2023).

The molecularly imprinted polymer (MIP) technique for electrode patterning shows great potential for the selective detection of target analytes in real sample matrices. This method, widely used to functionalize many electrodes, involves copolymerizing template molecules (the target analytes) with functionalized monomers. Once the template molecules are removed from the polymer matrix, specific recognition sites or cavities are left behind that are tailored to the template molecules (Burns et al., 2023). A sensor was created by electrodepositing a poly(melamine)-poly(aspartic acid) copolymer on a carbon paste electrode (CPE). The sensor's combination of semi-conducting and organic polymer properties increased the surface area for analyte interaction. The limit of detection for DA in serum, was measured at 780 pM using differential pulse voltammetry (Bonyadi& Ghanbari, 2021).

Conducting polymer nanowires hold significant potential for biosensor development due to their high surface area and electrical conductivity. For this reason, 3D structures consisting of polypyrrole nanowires and molecularly imprinted polymers (MIPs) were created by electropolymerizing DA with o-phenylenediamine using the cyclic voltammetry technique on glassy carbon electrode surfaces. The resulting biosensor exhibited both a large surface area and excellent electrocatalytic activity for dopamine oxidation, resulting in high sensitivity and a low detection limit of 33 nM. Additionally, the electropolymerized MIP provided numerous accessible surface imprints, enhancing the biosensor's selectivity. A similar dopamine electrochemical biosensor was also developed using MIP arrays modified on vertically aligned ZnO nanotubes (Lakard et al., 2021).

Polymer-based substrates are attractive materials for developing wearable sensors due to their low cost and flexibility, making them suitable for real-time disease monitoring. Although few sensors have utilized polymer substrates, materials like paper, SU-8, and polydimethylsiloxane pyramids have been employed as working electrodes. One example involved a paper electrode coated with nickel-iron phosphide/phosphate (NiFeP), which achieved a limit of detection of 300 pM. The NiFeP was applied to the Whatman filter paper via drop-casting, and its low LOD was attributed to the high charge-transfer conductivity of NiFeP, facilitating rapid electron transfer kinetics for DA electro-oxidation. (Kafle et al., 2022).

Another sensor, using neodymium, iron, and boron (NiFeB) with carbon paste, demonstrated a higher LOD but a broader linear range compared to a NiFe bimetallic compound-modified paper-based sensor. In this case, Vulcan carbon cellulose paper was prepared by

coating Whatman paper with carbon paste, followed by drop-casting NiB and FeB onto the modified electrode. This fabricated electrode exhibited a high surface area due to the carbon paste's porosity and maintained flexibility thanks to the paper substrate. Bimetallic phosphides showed superior catalytic activity compared to bimetallic oxides, and the electrode's flexibility suggests potential for wearable applications. The sensor effectively detected DA with minimal interference from uric acid, and both sensors were validated using dopamine-spiked real samples to confirm their practical performance (Thakur et al., 2020). The electrode that had been developed in the context of this study is shown in the next figure, **Figure 10**.

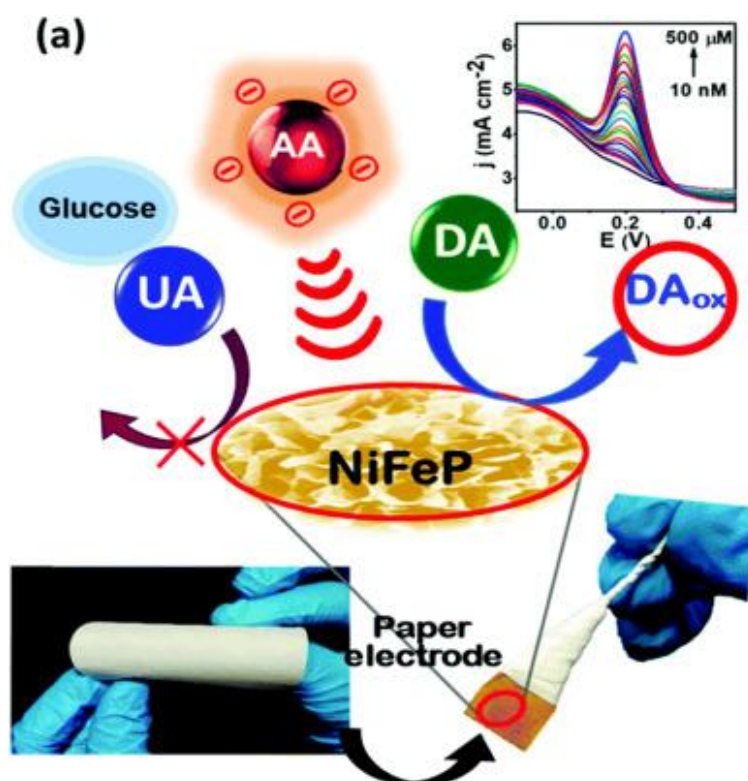


Figure 10. The NiFe bimetallic compound-modified paper-based sensor (Thakur et al., 2020).

4.4.3. Nanomaterials

Because of their special qualities, sensors based on nanomaterials have become highly effective instruments for the detection of dopamine, providing increased sensitivity and selectivity. These sensors use a variety of nanomaterials, including carbon-based materials, nanoparticles, and nanocomposites, which have good electrical conductivity, a large surface area, and the capacity to functionalize with certain proteins (Lakard et al., 2021). These characteristics make it possible to detect dopamine at extremely low concentrations, which makes them perfect for use in neurological research and medical diagnostics. Rapid, precise, and dependable dopamine detection is achievable by incorporating nanomaterials into sensor systems.

4.4.3.1. Carbon-based

Recent research has brought attention to the special physicochemical characteristics of carbon nanomaterials, such as graphene, graphene oxide, and carbon nanotubes (CNTs), to create a new class of controlled, enhanced electrochemical sensors for the detection of norepinephrine, dopamine, and adrenaline. These nanoparticles have been widely applied as signal enhancers in various methods to offer a successful platform for increasing sensitivity. One of the primary developments in the field of selective electrochemical sensors has included the application of conductive polymer-coated carbon nanotubes. Various techniques have been developed for the ultrasensitive detection of catecholamines on carbon nanotubes coated with a polymer matrix (Fredj et al., 2023; Brouzgou et al., 2019).

Carbon nanofibers (CNFs), another structure based on carbon, have been employed for many years in a variety of applications. The first carbon fiber microelectrode (CFM) with an active surface consisting of carbon fibers (8 μm apiece) was created in 1980 to detect DA in rats. Since then, CFMs have been used extensively for neurotransmitter detection because of their high specific strength, good conductivity, and biocompatibility. In addition, carbon nanohorns (CNHs), also known as carbon nanocones, are carbon cages with a conical form. CNHs, which are composed of sp^2 -hybridized carbons with high specific surface area and edge planes, are classified as 1D carbon nanostructures, just as SWCNTs. Among other characteristics, they have a strong resistance to oxidation and semi-conductivity (Balkourani et al., 2023; Balkourani et al., 2021).

A straightforward approach was employed by Elugoke et al., to fabricate an electrochemical sensor using a nanocomposite of carbon quantum dots (CQDs) and copper oxide (CuO) for the detection of dopamine. The sensor's components and the composite material were thoroughly characterized using a variety of analytical techniques, including UV-visible spectroscopy, Fourier-transform infrared (FT-IR) spectroscopy, energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD). Additionally, the structural and morphological properties were examined using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The nanocomposite (CQDs/CuO) was used to modify a glassy carbon electrode (GCE). Electrochemical analysis of dopamine (DA) at the CQDs/CuO-modified GCE demonstrated that optimal dopamine detection occurs at approximately 0.3 V. The sensor exhibited a detection limit of 25.40 μM , determined by square wave voltammetry (SWV), across a broad linear range of 1–180 μM . Although the sensor showed excellent reproducibility, it had relatively low stability. The percentage recovery of dopamine from a

dopamine hydrochloride injection, measured with this sensor, confirmed its effectiveness for DA detection in pharmaceutical samples and suggests potential applicability for other dopamine-containing real samples (Elugoke et al., 2022).

A composite material consisting of reduced graphene oxide (rGO) combined with nickel (Ni) and cobalt (Co) was synthesized through a hydrothermal process followed by high-temperature annealing. This composite was utilized to fabricate an electrode for dopamine (DA) detection. The material's surface and structural properties were characterized using scanning electron microscopy (SEM), transmission electron microscopy (TEM), Fourier-transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD). Electrochemical testing revealed that the rGO/Ni/Co-modified glassy carbon electrode exhibited excellent electrocatalytic performance for DA detection. The sensor demonstrated a broad linear detection range from 10 $\mu\text{mol/L}$ to 2250 $\mu\text{mol/L}$ with a high sensitivity, achieving a detection limit of 0.119 $\mu\text{mol/L}$. Interference tests indicated that common ions had minimal impact on DA detection, highlighting the sensor's high selectivity. Additionally, the composite material's magnetic properties facilitated easy sample separation and recovery, enhancing the practicality of the sensor (He et al., 2020).

Anshori et al., developed a nanocomposite using functionalized multi-walled carbon nanotubes and silver nanoparticles (f-MWCNT/AgNP) as the biosensing material for dopamine (DA) detection. Characterization techniques like SEM, EDS, and TEM confirmed the successful functionalization, with MWCNT serving as the base material. Differential Pulse Voltammetry (DPV) measurements provided key performance metrics, including a limit of detection (LOD) of 0.2778 μM within a linear range of 0–8 μM , which is below the concentration required to detect DA in human urine (0.3–3 μM). The biosensor also demonstrated high selectivity for DA even in the presence of other biofluids (Anshori et al., 2021).

An ultrasensitive electrochemical sensor based on a composite of graphene quantum dots (GQDs) and multi-walled carbon nanotubes (MWCNTs) was developed for detecting DA. The use of GQDs, known for their large surface area, enhances the electrode's conductivity, while MWCNTs serve as exceptional electrode materials. The sensor demonstrated outstanding selectivity for dopamine, effectively distinguishing it from other interfering bioanalytes. Under optimized conditions, the sensor achieved excellent performance in dopamine detection, with a broad linear range from 0.005 to 100.0 μM and a detection limit as low as 0.87 nM. Additionally, this sensor was successfully applied to detect dopamine in

human serum and was the first to measure dopamine secretion from live PC12 cells, delivering impressive results (Huang et al., 2020).

The Li_2TiO_3 -multiwalled carbon nanotube (LTO-MWCNT) nanocomposite, synthesized by Lakshmi Narayana, et al., using a one-step hydrothermal method, has been examined for its use as an electrochemical sensor in pharmaceutical formulations. Characterization techniques including X-ray diffraction, X-ray photoelectron spectroscopy, Raman spectroscopy, and transmission electron microscopy reveal that MWCNTs are encapsulated within well-crystallized, cubical-shaped LTO nanoparticles (with a monoclinic structure, C2/c space group, and 53 nm size), creating an electrode material with an efficient conductive network. BET analysis indicates that the LTO-MWCNT nanocomposite has a high specific surface area of 32 m^2/g and an average pore diameter of 16 nm, providing numerous electroactive sites and maintaining structural stability, which contributes to its excellent electrochemical performance. When applied to a glassy carbon electrode, the LTO-MWCNT composite was tested for its electrocatalytic activity towards DA using CV and DPV. The LTO-MWCNT/GC electrode shows remarkable sensitivity and selectivity for detecting acetaminophen and tyrosine. The sensor demonstrated a LOD of 1.71 $\mu\text{mol/L}$ and a limit of quantification of 5.7 $\mu\text{mol/L}$, with a dynamic range of 5.0–1000 $\mu\text{mol/L}$. The LTO-MWCNT/GC sensor provides good accuracy and precision, achieving recovery rates of 98% in dopamine injection formulations (Lakshmi Narayana et al., 2022). **Figure 11** shows the overall process for the synthesis of the Li_2TiO_3 -multiwalled carbon nanotube (LTO-MWCNT) nanocomposite and the DA detection.

In another study, electrochemical DA sensors were created using reduced graphene oxide coupled with gold or platinum nanoparticles. These sensors were developed through co-electrodeposition onto a flexible substrate, with a systematic investigation of electrodeposition parameters (such as precursor concentration, deposition time, and potential) conducted to optimize DA detection sensitivity. Square wave voltammetry, an electrochemical technique, was utilized to achieve highly sensitive detection in the nanomolar (nM) range.

The sensors were tested against synthetic urine to mimic real sample conditions, where dopamine concentrations typically fall below 600 nM. The sensors demonstrated minimal interference from uric and ascorbic acids, which did not impact performance. They also exhibited a wide linear detection range with high sensitivity and a low limit of detection. When tested with real urine samples, the dopamine concentrations detected were consistent with

results obtained through conventional laboratory methods. Also, the sensor fabrication process was cost-effective and compatible with flexible substrates, indicating potential for integration into wearable sensing devices (Patella et al., 2021).

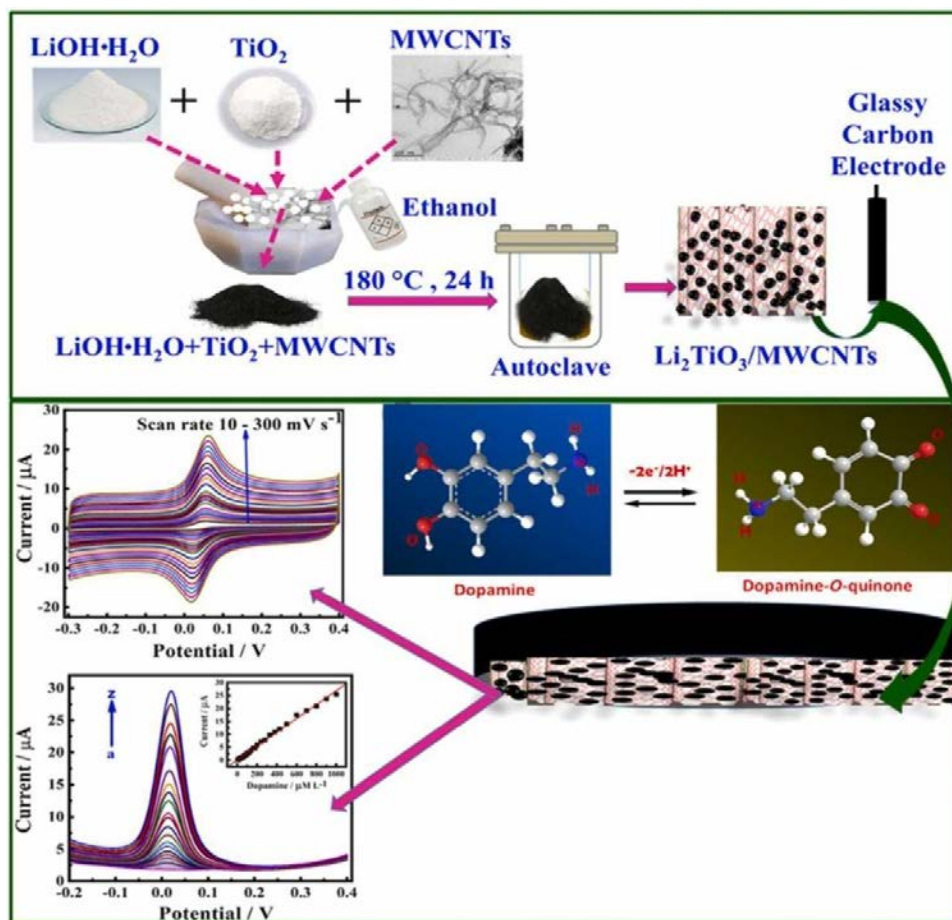


Figure 11. The Li_2TiO_3 -multiwalled carbon nanotube (LTO-MWCNT) nanocomposite synthesized for DA detection (Lakshmi Narayana et al., 2022).

The weak interfacial forces between graphene and conductive substrates pose a challenge in creating stable graphene electrodes. In a study, few-layer graphene was directly synthesized on a high-pressure high-temperature (HPHT) diamond substrate through sp^3 -to- sp^2 conversion using catalytic thermal treatment, with the diamond itself serving as the carbon source. This hybrid electrode demonstrated high conductivity and exhibited a linear electrochemical response to dopamine in the concentration range of $5 \mu\text{M}$ to 2 mM , with a detection limit as low as 200 nM . However, prolonged exposure to DA led to electrode fouling and a reduction in sensitivity. Due to the strong interfacial bonding between graphene and HPHT diamond, the fouled electrode could be regenerated and its performance fully restored through ultrasonic cleaning (Yuan et al., 2018). This ability is also shown in the diagram below, in **Figure 12**.

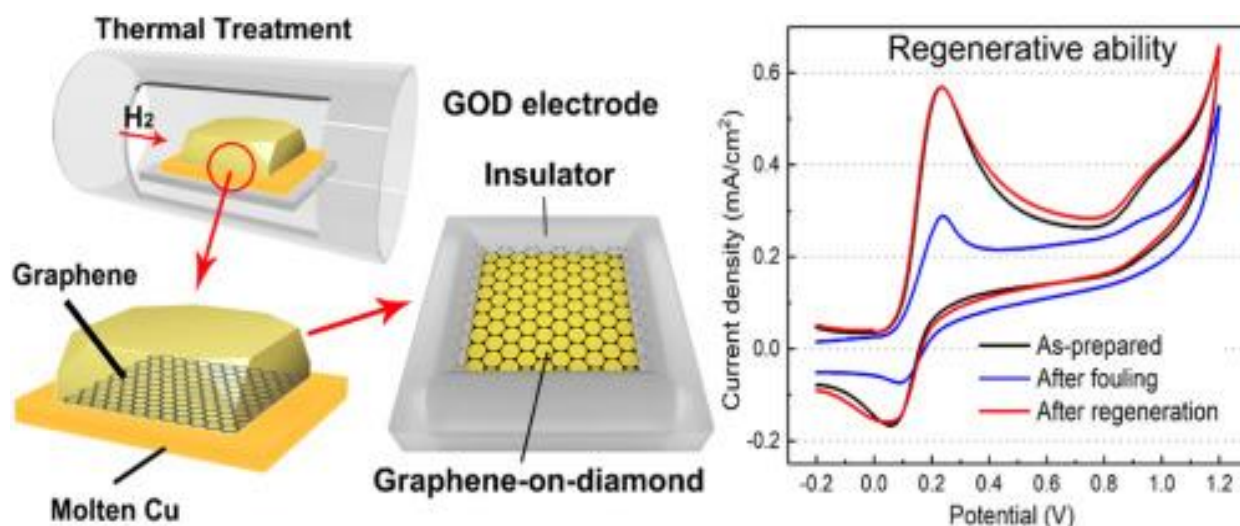


Figure 12. The regenerative electrode developed by Yuan et al. (Yuan et al., 2018).

Doping carbonaceous materials with heteroatoms like nitrogen, phosphorous, and boron can improve, as was previously indicated, by enhancing the analyte molecules' bonding sites, their electrochemical activity. In order to evaluate a nitrogen-doped rGO nanocomposite (N-rGO) against DA detection, Zhang et al. constructed a modified GCE. Poly (p-phenylenediamine)-rGO (PpPD-rGO) hybrids were Pyrolyzed to provide a nitrogen-doped reduced graphene oxide nanocomposite. The sensing performance of the N-rGO sensor in physiological settings (pH = 7) was evaluated utilizing the techniques of CV and DPV. As per their findings, N-rGO demonstrated efficacious sensing capabilities in the simultaneous identification of DA, AA, and UA, exhibiting linear voltametric responses within the respective ranges of 1.0–60.0 μM , 0.1–4.0 mM, and 1.0–30 mM (Zhang & Liu, 2020).

Anbumannan et al. created a new GO/tungsten trioxide (GO/ WO_3) nanocomposite using a two-step procedure. WO_3 NPs modified GO via a co-precipitation approach after GO was synthesized using the modified Hummers' process. When the as-synthesized WO_3/GO composite was examined physically and chemically, the 100.0–200.0 nm nanocube-shaped WO_3 was found to be well distributed and to cover the GO surface uniformly. The electrode's capacity for sensitive and selective DA detection was examined using the chronoamperometry as shown in **Figure 13**.

Metal–organic frameworks (MOFs) hold significant potential for detecting heavy-metal cations, as they serve as substrates onto which metals and other nanoparticles can be deposited. Additionally, MOFs can enhance the accuracy of identifying cells and tissues. The biosensing capabilities of MOFs are attributed to their low toxicity, exceptional water stability, good biodegradability, and biocompatibility. MOFs have garnered considerable attention due

to their highly organized structures, excellent electrical conductivity, and unique catalytic activity. Research indicates that MOFs can be effectively employed to create biosensors capable of detecting a wide range of small molecules in complex mixtures, capitalizing on these advantageous properties (Dourandish et al., 2022).

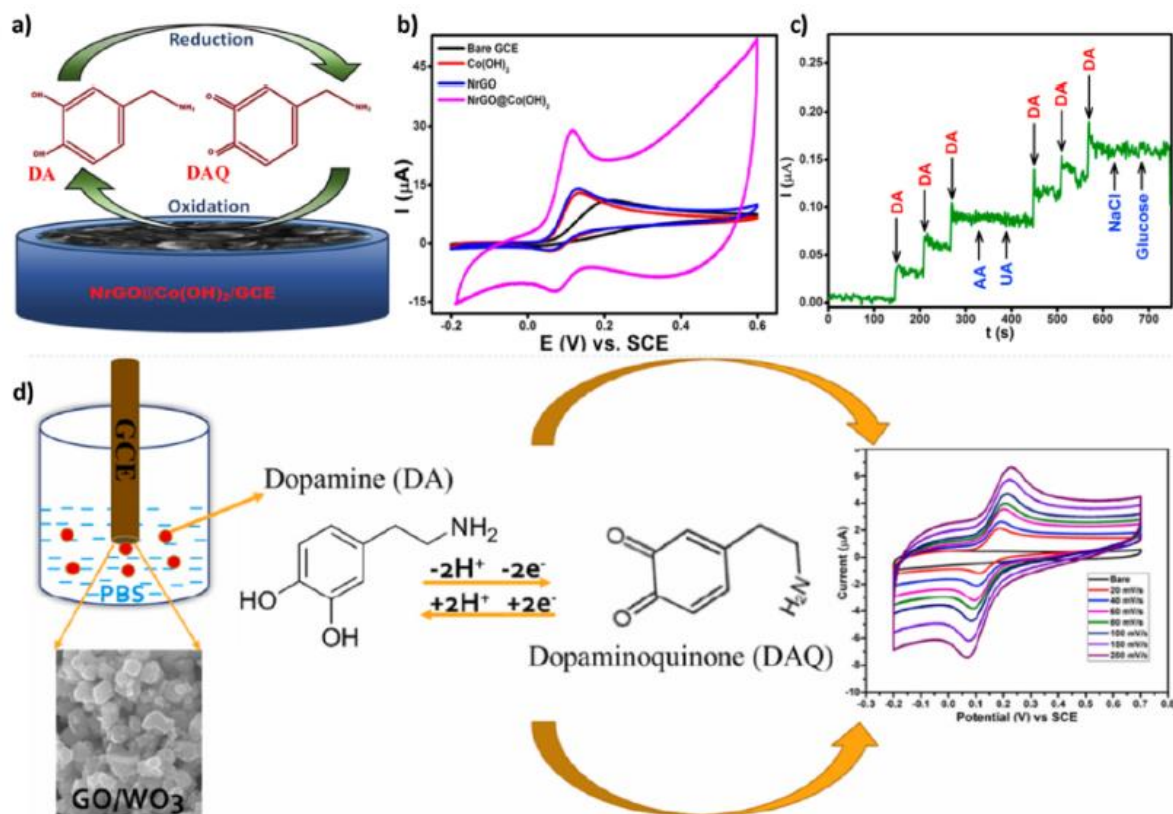


Figure 13. The GO/tungsten trioxide (GO/WO₃) nanocomposite used to detect DA (Anbumannan et al., 2021).

MOFs are a type of solid porous material composed of metal ions or metallic clusters, which serve as connection points, and polydentate organic ligands that link these nodes together through coordination bonds. These metal nodes, either ions or clusters, connect the organic ligands, creating one-dimensional, two-dimensional, or three-dimensional networks. MOFs are characterized by several key structural features directly linked to their properties and applications, including high porosity, a substantial pore volume that can account for up to 90% or more of the crystalline structure, a large specific surface area (often several thousand square meters per gram), and high thermal stability (ranging from 250 to 500 °C) due to the presence of strong bonds such as C–C, C–H, C–O, and M–O (Raptopoulou, 2021).

A chemically modified carbon paste electrode (CPE) was developed using a cobalt metal-organic framework-graphene oxide (CoMOF-GO) composite combined with the ionic liquid 1-methyl-3-butylimidazolium bromide (1-M,3-BB). This modified electrode, referred to

as CoMOF-GO/1-M,3-BB/CPE, was designed for the simultaneous detection of dopamine (DA) and uric acid (UA). The CoMOF/GO nanocomposite was thoroughly characterized using several techniques, including field emission-scanning electron microscopy (FE-SEM), X-ray diffraction (XRD), nitrogen adsorption–desorption isotherm analysis, and energy dispersive spectroscopy (EDS). The electrochemical sensor demonstrated significant catalytic activity towards the redox reaction of dopamine, as evidenced by the enhanced oxidation peak current compared to that of a bare carbon paste electrode. The CoMOF-GO/1-M,3-BB/CPE sensor exhibited a broad linear response for dopamine, ranging from 0.1 to 300.0 μM , with a detection limit as low as 0.04 μM . Furthermore, the oxidation peaks for dopamine and uric acid were well-separated when both compounds were present in a mixture, indicating the sensor's effectiveness in distinguishing between the two analytes (Roostaei et al., 2022).

Moallem et al., synthesized a nanostructured Cu-BTC (Cu-benzene-1,3,5-tricarboxylic acid) metal-organic framework (MOF) using an ultrasonic-assisted hydrothermal technique. The morphology, composition, and structure of the Cu-BTC MOF were characterized using various methods, including energy-dispersive X-ray spectroscopy (EDS), Fourier-transform infrared spectroscopy (FT-IR), XRD, Brunauer–Emmett–Teller (BET) analysis, field emission-scanning electron microscopy (FE-SEM), and elemental mapping. A sensitive electrochemical sensor was fabricated by modifying a carbon paste electrode (CPE) with the synthesized Cu-BTC MOF. This sensor was used for the detection of dopamine. The sensor's analytical performance was evaluated using CV, DPV, and chronoamperometry (CHA). Under optimized conditions, the Cu-BTC/CPE sensor demonstrated a linear detection range of 0.05 to 500.0 μM with a detection limit of 0.03 μM for DA, and a range of 0.5 to 600.0 μM with a detection limit of 0.2 μM for uric acid. The sensor was also capable of simultaneously detecting dopamine and uric acid, showing two distinct peaks during the electro-oxidation process (Moallem et al., 2022).

4.4.3.2. Inorganic

As aforementioned perovskite materials are known for their versatile electrical and ionic conductivities, including mixed electronic and ionic conductivities. These properties make them highly valuable in a wide range of technological applications such as multilayer ceramic capacitors, computer memories, sensors, wireless communication systems, pyroelectric detectors, hydrogen fuel cells, high-temperature superconductors, and other electronic devices. Among the perovskite family, barium zirconate stands out as a semiconductor proton-

conducting oxide. It is recognized for its superior thermal and chemical stability, corrosion resistance, low thermal expansion, and excellent structural compatibility. These advantageous properties arise from its structurally disordered vacancies and the rapid proton hopping between adjacent oxygen atoms (Bui & Shin, 2023).

In a study conducted by Thomas et al., BaZrO₃ perovskite crystallites were synthesized through a single-step, calcination-free, room-temperature co-precipitation method. Various techniques were employed to analyze the crystallite size, metal-oxygen bonds, thermal stability, defect structure, and surface morphology of the material. This synthesized material was then used to modify a carbon paste electrode (CPE) to create an advanced sensor. The modified electrode demonstrated lower resistance and improved charge transfer capabilities compared to the unmodified electrode. Electrochemical evaluations using CV and DPV revealed high current sensitivity for detecting DA, with an impressive detection limit of just 5 pM. The sensor also showed detection limits of 500 pM and 500 nM for co-existing molecules of uric acid and ascorbic acid, respectively. In addition, the sensor was capable of accurately quantifying DA even in the presence of the other acids. Further tests, including mixed sample analysis, interference studies, and stability assessments, confirmed the sensor's effectiveness. Additionally, the sensor's practical utility was demonstrated through real sample analysis in blood plasma and urine (Thomas et al., 2022).

A novel dopamine-imprinted sensor was developed by using a poly-ortho-phenylenediamine (P-o-PDA) coating on a titanium foil electrode, modified with a double photosystem type II heterojunction created by coupling dendrite-like CuInSe₂ (CIS) with Cu₂O nanoparticles. The sensor was fabricated through electropolymerization, combining the signal indication and functional properties of P-o-PDA with the photoelectric conversion and amplification capabilities of the CIS/Cu₂O heterojunction. Under visible light, the MIP-CIS/Cu₂O-coated Ti foil demonstrated a strong cathodic photocurrent response, which decreased in the presence of dopamine due to the hindrance effect. The sensor's performance was optimized by adjusting factors such as pH, light wavelength, and applied bias potential. It exhibited excellent sensitivity with a detection limit of 2.1 nM and reliable performance across two ranges (0.1-5.5 μM and 5.5-150 μM) for dopamine detection. The sensor showed high stability, reproducibility, repeatability, and selectivity. It was also tested on real samples, demonstrating its potential for practical applications and highlighting a new approach for designing organic-inorganic heterojunction-based photoelectrochemical sensors for biomarker detection (Dashtian et al., 2021).

Tin dioxide (SnO_2) nanoparticles were synthesized using an eco-friendly approach, employing *Nelumbo nucifera* (lotus leaf) extract as a reducing agent, without the need for additional chemicals. These nanoparticles were then applied to modify a glassy carbon electrode (GCE) for the electrochemical detection of DA. Cyclic voltammetry (CV) revealed that the modified GCE exhibited significantly better electrochemical sensing performance compared to the bare GCE electrode (Harshavardhan et al., 2023).

A simple electrochemical sensor was developed using electrochemically synthesized Fe_3O_4 nanoparticles. The sensor was constructed by preparing an ink containing the nanoparticles, isopropanol, NAFION, and carbon Vulcan, designed to detect dopamine, uric acid, and ascorbic acid. The electrocatalytic activity of the Fe_3O_4 nanoparticles for the oxidation of these analytes was evaluated using cyclic voltammetry and square wave voltammetry. Various parameters affecting the sensor's performance, such as the quantity of Fe_3O_4 nanoparticles (ranging from 1 to 10 mg), the amount of binder (5, 10, or 15 μL), and carbon Vulcan content (4, 6, or 8 mg), were optimized. The optimal conditions were found to be 8 mg of Fe_3O_4 nanoparticles, 4 mg of carbon Vulcan, and 5 μL of NAFION@117. The sensor demonstrated significant sensitivity and selectivity, with no interference from the simultaneous presence of the three analytes in solution and was successfully applied to detect these molecules in real samples (Gaya et al., 2023).

Electroanalytical techniques have demonstrated significant potential for detecting dopamine (DA); however, they often face challenges related to severe biofouling. This occurs because interfering proteins or cells present in complex biological media can non-specifically adsorb onto the sensing interface, leading to diminished sensitivity and compromised accuracy. A dependable and compact electrochemical sensing system with ultralow fouling capabilities was developed as shown in **Figure 14** to monitor targets in complex biological media, utilizing a gold nanoparticles-peptide hydrogel-modified screen-printed electrode. The zwitterionic peptide hydrogel was synthesized using a newly designed peptide sequence: Phe-Phe-Cys-Cys-(Glu-Lys) $_3$, with the N-terminal modified with a fluorene methoxycarbonyl group. The thiol groups present on the cysteine residues of the peptide self-assembled with gold nanoparticles to create a three-dimensional nanonetwork structure that exhibited excellent antifouling properties in complex biological environments, such as human serum. The resulting electrochemical sensing platform demonstrated remarkable performance for dopamine detection, featuring a wide linear range (0.2 nM to 1.9 μM), a low limit of detection (0.12 nM), and outstanding selectivity (Liu et al., 2023).

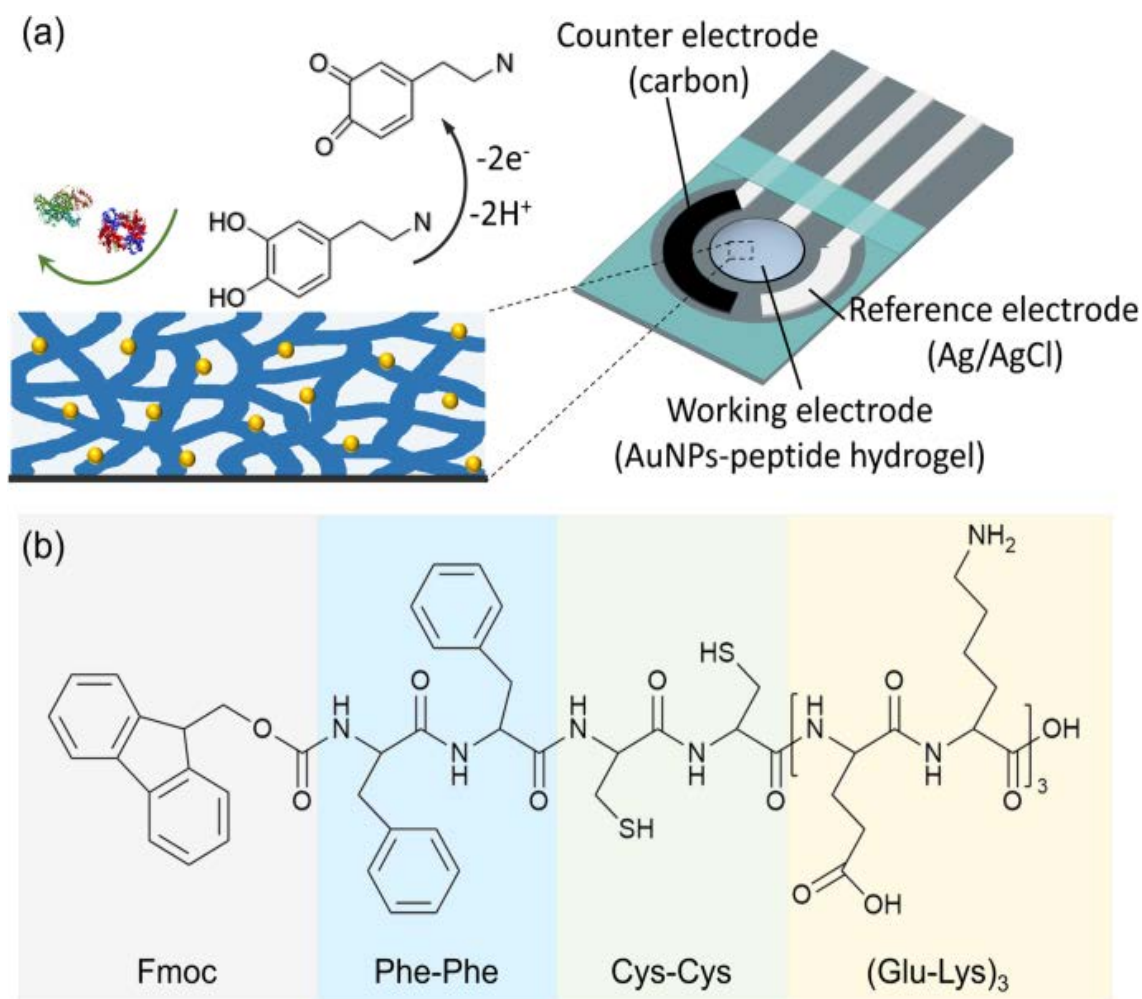


Figure 14. The AuNPs peptide biosensor (Liu et al., 2023).

4.4.3.3. Aptamer-based

Aptamers are short oligonucleic acid sequences (30 to 100 nucleotides) that can bind specifically to a wide range of target molecules, from small ions to large proteins, with high affinity and specificity. Derived from the Latin word "aptus," meaning "to fit," these molecules are selected through a process called SELEX (Systematic Evolution of Ligands by Exponential Enrichment) from diverse random sequences. Aptamers are often referred to as synthetic antibodies due to their ability to mimic antibody functions. They offer several advantages over traditional antibodies, including precise and reproducible chemical production, chemical stability across various buffer conditions, resistance to harsh treatments, and reversible thermal denaturation (Radi, 2011). An overview of aptamer-based biosensors for neurotransmitters detection can be found in the next figure, **Figure 15**.

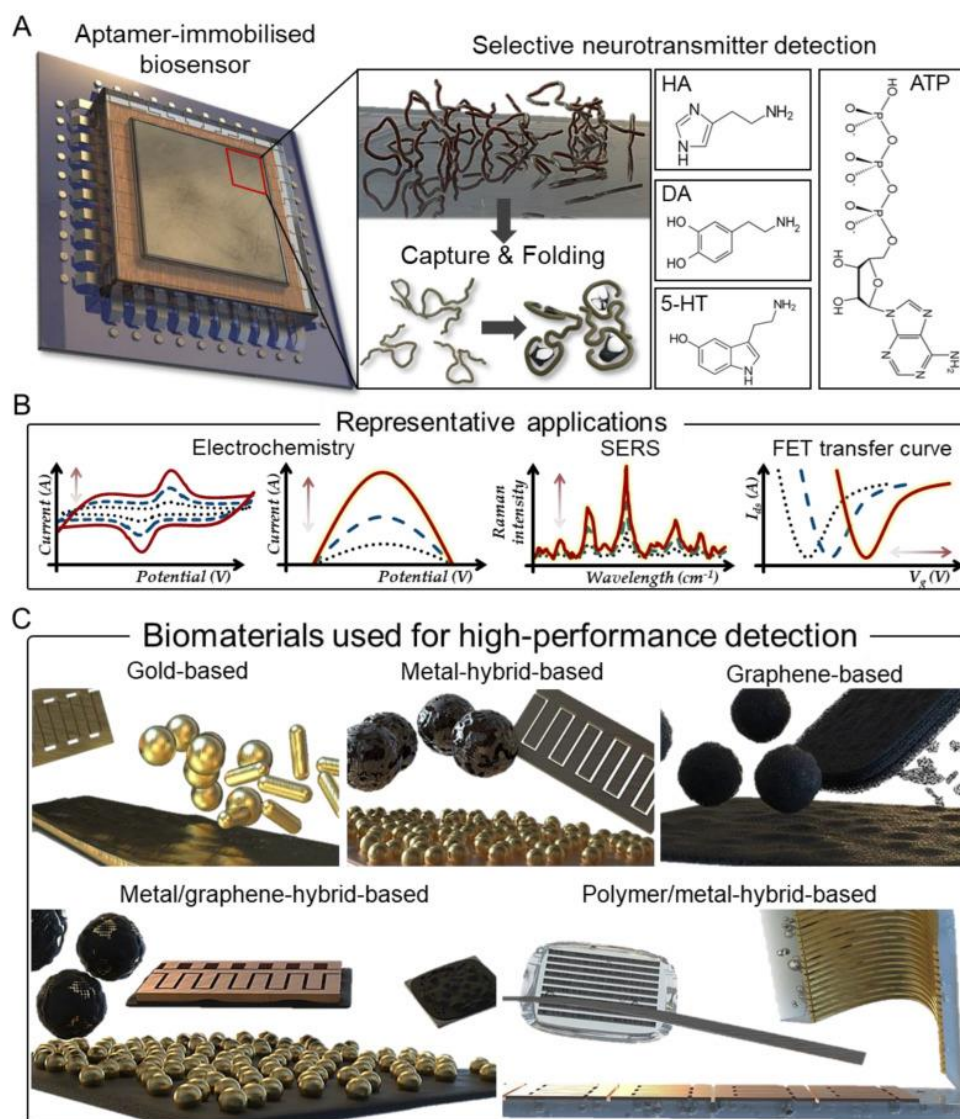


Figure 15. Aptamer-based biosensors (Park et al., 2023).

A label-free liquid crystal-based biosensor has been developed for detecting DA in aqueous solutions using dopamine-binding aptamers as recognition elements. In this biosensor, dimethyloctadecyl [3-(trimethoxysilyl) propyl] ammonium chloride (DMOAP) self-assembled monolayers are used to immobilize DBA on glass slides, with the DMOAP layers maintaining the liquid crystal molecules in a homeotropic alignment. The LC-based aptasensor demonstrated a broad linear optical response for DA ranging from 1 pM to 10 μ M and featured an exceptionally low detection limit of 10 pM ($\sim$ 1.9 pg/mL). Additionally, the biosensor showed good selectivity and has been successfully used to detect DA in human urine (Nguyen& Jang, 2020).

In 2022, researchers developed a method to quantify DA using a graphene multi-transistor array (gMTA) functionalized with a selective DNA aptamer. The micron-sized electrolyte-gated graphene field-effect transistors (EG-gFETs) were modified with DNA aptamers and integrated into the gMTA. Each chip contained 20 transistors, with groups of 10

sharing a common source and individual gold drain electrodes connected to two common source electrodes. The team created a calibration curve for detecting DA in artificial cerebrospinal fluid, demonstrating high sensitivity with a detection limit of 1 attomolar (aM) and a linear range from 1 aM to 100 μ M. The sensor was also tested for selectivity, successfully detecting dopamine in the presence of interfering molecules. Although the response to other neurotransmitters like serotonin and norepinephrine was not tested, the DNA aptamer used had low affinity for these compounds. Additionally, the sensor detected DA in the brains of rats with Parkinson's disease. The study demonstrated the repeatability and reproducibility of the sensor by synthesizing 15,680 EG-gFETs and 784 gMTAs with an 80% yield. The biosensor and the main key-findings are given in the next figure, **Figure 16** (Abrantes et al., 2022).

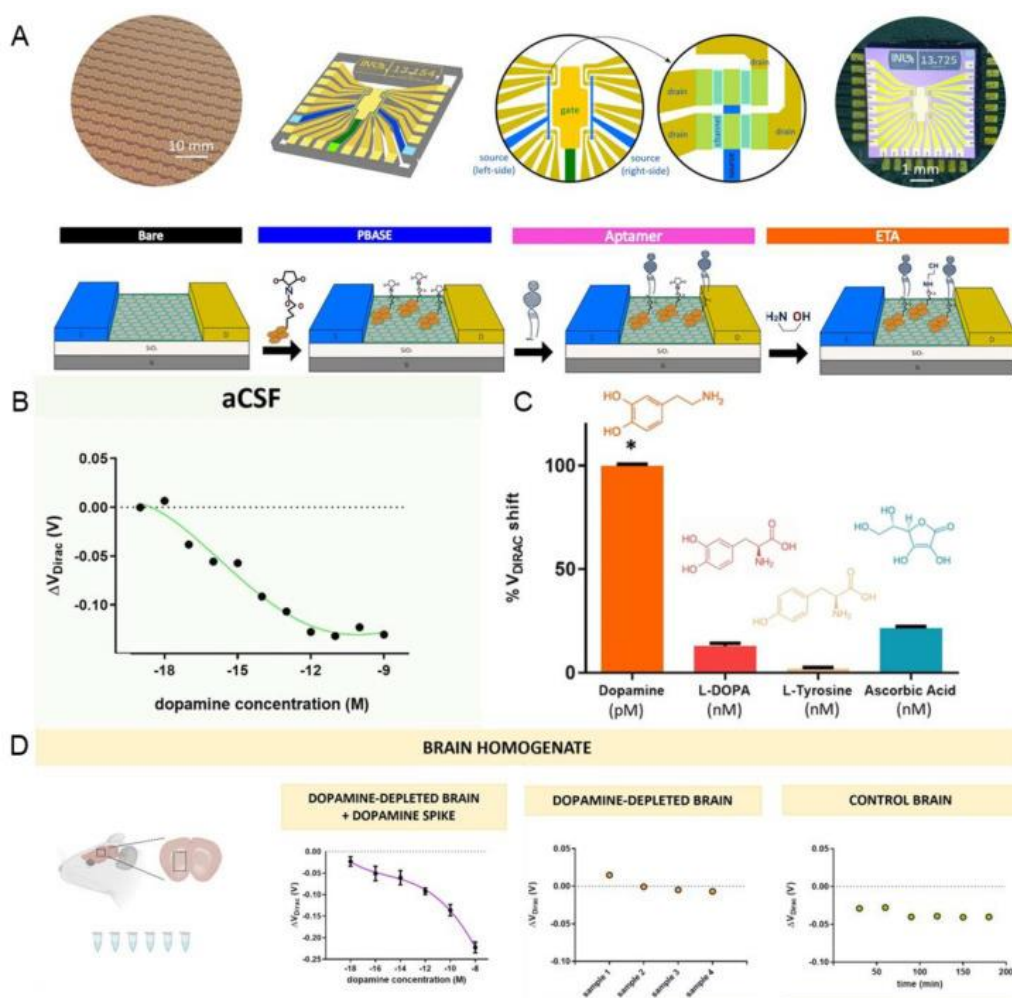


Figure 16. The graphene-based aptamer sensors developed for DA detection. (Abrantes et al., 2022).

Another study employed aptamers, nucleic acid-based recognition agents, to capture target molecules from complex samples. Following this capture, an electrochemical method known as differential pulse voltammetry was used to analyze the specific oxidation profiles of DA and serotonin (SER). The multi-detection biosensor system developed exhibited a linear response range

of 0.1 to 4 μM , with detection limits of 0.06 μM for DA and 0.12 μM for SER. Additionally, the sensor was tested with real serum samples, yielding recovery values between 86% and 92%. Overall, the study concluded that the aptamer binding process effectively addresses the sensitivity limitations typically associated with multi-detection methods (Cuhadar et al., 2024).

CHAPTER 5 CONCLUSIONS

Electrochemical sensors represent a powerful and versatile technology for detecting and quantifying chemical species across diverse applications. Their ability to deliver high sensitivity and selectivity, combined with real-time monitoring capabilities, makes them invaluable in fields such as environmental monitoring, medical diagnostics, and industrial process control. These sensors operate on the principles of electrochemical reactions, utilizing electrodes and electrolytes to generate measurable electrical signals that correspond to the concentration of target analytes.

Despite their advantages, including cost-effectiveness and portability, electrochemical sensors must be carefully designed to address challenges related to calibration, stability, and interference. Overall, the ongoing advancements in sensor materials and technology continue to enhance the performance and applicability of electrochemical sensors, paving the way for innovative solutions in both research and practical applications.

The most commonly used are carbonaceous materials and this type of materials include (i) carbon and its derivatives and (ii) graphene and its derivatives. A review article by Balkourani et al., highlighted that carbon nanotubes (both single-walled and multiwalled), graphene, and reduced graphene oxide (rGO) are key competitors, exhibiting the most favorable operational parameters, particularly low limits of detection (LOD), extended linear ranges, and strong selectivity. In this context, carbon nanotubes (CNTs), including single-walled and multi-walled (MWCNTs), are commonly favored for electrochemical sensing of dopamine.

Because of their sensitivity, specificity, and capacity for real-time analysis, electrochemical biosensors present a promising method for the detection of dopamine. However, the presence of interfering compounds like ascorbic acid and uric acid, which are abundant in bodily fluids and can reduce the precision of dopamine readings, can dramatically damage the efficiency of these sensors. There are several difficulties because of these compounds' regeneration qualities and overlapping electrochemical characteristics. Advanced surface modification methods and the use of certain biological molecules, such as enzymes, antibodies, and aptamers, are used to get around these challenges and improve the dependability and specificity of dopamine detection. Although these techniques enhance sensor performance, they may also complicate and prolong the sensor development process.

Future developments in sensor technology, such as better materials and creative design approaches, will be essential to creating electrochemical biosensors that are more dependable

and effective for precise dopamine monitoring in intricate biological contexts. In addition, the incorporation of machine learning techniques to analyze sensor data can help differentiate dopamine signals from those of interfering substances. Algorithms could be trained to recognize and correct for noise and interference, improving measurement accuracy. In parallel, predictive modeling and time-series analysis allow for forecasting future dopamine levels, which is particularly useful in clinical settings for monitoring neurological conditions. Additionally, ML can detect anomalies and unusual patterns, providing early warnings of potential issues. Personalized monitoring is enhanced through ML by tailoring detection algorithms to individual patient data, improving the relevance and accuracy of measurements. Integration with other analytical techniques and the development of smart sensors with embedded ML further advance the capabilities of dopamine detection, offering precise, reliable, and adaptive monitoring solutions.

In addition, advances in microfabrication techniques, such as photolithography and etching, allow for the precise creation of smaller sensor components, including electrodes and supporting structures. Nanotechnology further contributes by enabling the design of sensors at the nanoscale, where materials and structures can exhibit unique properties that improve sensitivity and efficiency. For instance, nanosized electrode materials can provide a larger surface area for electrochemical reactions, enhancing the sensor's performance. Also, integrated sensors can be incorporated into portable or wearable devices, such as health monitors and fitness trackers, allowing for continuous and real-time monitoring of physiological parameters, including neurotransmitters like dopamine.

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