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Review

Voltammetric Electrochemical Sensor for Rapid and Convenient Morphine Detection: A Review

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Abstract- Morphine is a non-synthetic narcotic that derived from opium; it is used for the treatment of pain and it is toxic during overdose or when abused. In comparison to conventional analytical techniques, like HPLC, electroanalytical methods have advantages like simplicity, ease of operation, and miniaturization. Today, electroanalytical sensors are used in agriculture, food, oil, and biomedical applications. In addition to the versatility of reporting signals, such as voltages, currents, power outputs, or electrochemical impedances, electrochemical sensing has low theoretical detection limits due to differences between Faradaic and non-Faradaic currents. In this review, different electrochemical sensing modification-based techniques for determining the morphine content of samples have been investigated. Furthermore, we present the performance of reported electrochemical sensors toward morphine detection, including their detection range (LDR), detection limit (LOD), and modification of electrodes. It is our belief that the information in this manuscript can serve as a platform for future research on developing sensitive electrodes for morphine and other drugs.

Keywords- Morphine; Electroanalysis; Sensor; Nanoparticles; Biomedical Application

1. INTRODUCTION

Humans have reportedly used opium since the 6th millennium BCE. There were a number of opium products created during the Renaissance period, which led to its widespread administration and dependence in ancient societies. The potency of opium was not fully realized. In the early 1800s, Wilhelm Sertürner isolated morphine, opium's active ingredient [1]. In 1847, August Lauren (1809-1853) developed morphine salts through de-moisturizing and determined its chemical formula to be $C_{34}H_{38}N_2O_6$ by careful calculations, which corresponds to the currently accepted formula i.e $C_{17}H_{19}NO_3$) [2]. The World Health Organization (WHO), has declared morphine as the most widely administered opioid for treating moderate to severe pain in serious conditions in the United States. The opioid directly affects the pain-modulating receptors of the nervous system, that are also referred to as opioid receptors [3]. In comparison with other painkillers, morphine is considered the classic opioid analgesic. Morphine, like its other equivalents, targets delta, kappa, and above all, mu opioid receptors of the central and peripheral nervous systems [4-6].

No need to mention that like every other opioid, morphine causes dependency, its overdose is dangerous and it can even cause death, apart from affecting different immune functions, respiration rate and lowering blood pressure. Center for Disease Control and Prevention has reported over 46,000 deaths due to opioid-overdose in the US in 2018 [7,8]. Morphine is also used to synthesize hydromorphine, oxymorphine and heroin.

Analysis of morphine in biological samples is very common in forensics and pharmokinetics and clinical applications, which create high demand for facile, sensitive, and selective tools and techniques for its determination in various samples. However, no such tools are currently widely available.

Electrochemical sensors are valuable tools in medical diagnosis given their small dimensions, response speed, and low costs [9-11]. Recently, accurate sensors with reasonable costs have been designed and constructed for the analysis of morphine, and in the light of the above, this text tends to provide a comparative review of the different electrochemical sensors for the analysis of morphine, in terms of their performance including their detection range (LDR), detection limit (LOD), and modification of electrode [12].

2. METHODS FOR MORPHINE DETECTION

Opioid concentrations in biological samples are routinely determined through HPLC-MS, which is a very accurate method with very low limits of detection. The method, however, is highly costly, requires sophisticated equipment, highly trained technicians and also takes a lot of time. Electrochemical techniques, on the other hand, can be used to develop in vitro diagnostic tools for quantification of concentrations of medicines in blood samples [13-15], with short response times, low limits of detection, using simple equipment (Figure 1) [16,17]. Electrochemical sensors constitute a major field in medicine and biotechnology, environmental

and industrial applications. These devices can be tailor made through various modifications techniques. These stable and portable devices can also be miniaturized for specific purposes [18].

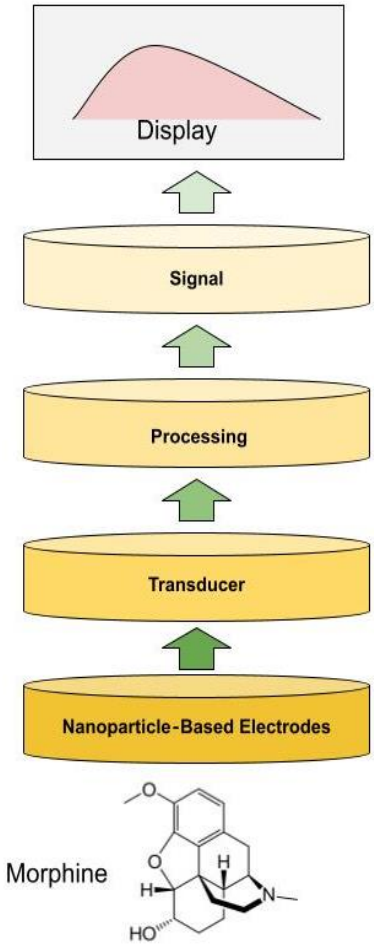

Figure 1. Schematic illustration of the major elements of a standard electrochemical sensor

3. ELECTROCHEMICAL SENSORS

Electrochemical sensors are dependable tools for biological, environmental, industrial, and pharmacological analyses with various figures of merit like durability, considerable sensitivity, accuracy. For over 20 years different nano-scale metal and metal-oxides, conductive organometallic compounds, and carbonous materials and polymers have been used in constructing modified electrochemical assays [19,20] to further enhance their loading of enzymes, antibodies, and aptamers and induce specificity to target species [21-23]. Modification of surface and structure helps to lower the electrical resistance and surface area, and enhance the sensitivity of the resulting analytical devices.

Further advantages of electrochemical sensors include low detection limits, and quick analysis which makes them ideal choices for flow analyses and alerting systems [24-27].

4. MODIFICATION OF ELECTROCHEMICAL SENSORS WITH NANOPARTICLES

Nanoparticles have unique properties, including their high surface/volume ratios, which make them suitable for modification of sensors and biosensors [28-40].

Researchers have also tried to control the dimensions and morphologies of various nanoparticles, given the role these factors can play on the applications in various areas, including the construction of sensing devices [39,41-43].

The role of nanoparticles in the modified sensing instruments include immobilizing biomolecules, electrocatalysis and the improved electron transfer between electrodes and target species, labeling biomolecules or direct involvement in reactions [44-50] Various nanoparticles, e.g., metallic, polymeric or composite nanostructures, fullerene, graphene, carbon nanotube have been applied in constructing sensing and biosensing electrodes (Figure 2).

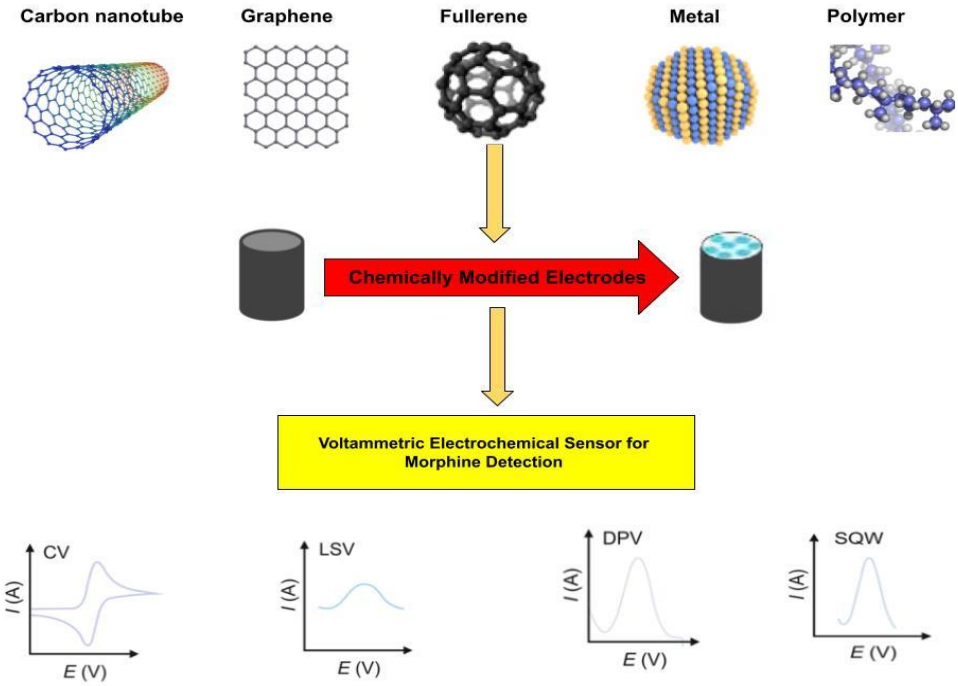

Figure 2. Schematic representation of a chemically modified electrode, interaction with the voltametric electrochemical sensor, and conversion of these interactions into measurable signals.

5. VOLTAMETRIC TECHNIQUES FOR DETECTION OF MORPHINE

Voltammetry is a common analytical technique, which is based on recording electrical current behavior upon altering the applied potential. Cyclic voltammetry (CV) is a very fundamental voltametric techniques, used for studying electrochemical characteristics like stability of final products, intermediates species, reaction reversibility, and electron transfer.

Quantitative applications of the method involve using the concentration of analytes through its correlation to the current in reversible reactions [51]. Another voltametric technique is the linear sweep voltammetry (LSV), in which potential scan is only in one direction, as opposed to CV where the potential sweep is also reversed which makes CV applicable to reversible reactions. LSV, on the other hand is more suitable for irreversible reactions, which cannot respond to reversal of scan direction. The maximum (peak) current is an indication of oxidation or reduction of the analyte and can be used for determination of its concentration. The measurements can be made more sensitive through altering the potential scan rate [51]. Another derivative of CV is differential pulse voltammetry (DPV), in which a set of potential pulses are accompanied with a the linear sweep during which the current reading is performed before the potential change, and its difference is also recorded during potential change, to eliminate the share of non-faradic charging current, enhancing sensitivity [52]. Another alteration is the quicker, more sensitive square wave voltammetry (SWV). The potential waveform in SWV includes a pulse amplitude and staircase waveform to eliminate the disturbing charging current for more sensitive analyses [51,53]. All these techniques have been applied to the determination of morphine and the following lines cover examples of each set of cases.

5.1. Cyclic voltammetry based electrochemical sensors for detection of morphine

There are many reports on the electrochemical detection of morphine using cyclic voltammetry. Table 1 lists these reports. Xu et al. reported a modified electrode with a cobalt hexacyanoferrate and used it for the CV analysis of morphine. They reported that the peak current had a linear correlation with the concentration of morphine from 1.0×10^{-6} M to 5.0×10^{-4} M at +0.60 V (*vs.* Ag/AgCl). The limit of detection was 5.0×10^{-7} M (S/N of 3) [54].

In another study a gold electrode modified with a 2-aminoethanethiol self-assembled monolayer was developed for the CV analysis of morphine. The electrode was reported to possess great electrocatalytic activity for the oxidation of morphine which was reflected by the enhanced more negative oxidation peak potential compared to the non-modified gold electrode. This behavior was attributed to the interaction of the amino group of the modifying group with the phenolic group of the analyte [55].

In another work, a film of indium tin oxide (ITO) electrode was modified with gold nanoparticles to construct an electrode for the CV analysis of morphine. The preparation of the electrode was carried out through electrochemical deposition. The electrode was reported to possess considerable electrocatalytic activity in oxidizing morphine, which produced a linear current/concentration correlation from 8.0×10^{-7} to 1.6×10^{-5} M (detection limit: 2.1×10^{-7} M) [56].

Yang et al. reported constructed an electrochemical sensor for morphine by depositing arrays of gold nanotubes on a glassy carbon electrode, and reported that te electrode could be

used for the determination of morphine concentration from 1.22×10^{-7} to 7.44×10^{-4} M (limit of detection: 4.06×10^{-8} M) [57].

In another study a sol-gel method was used to prepare a film of Alumina nanoparticles, which were thermally grown on the surface of silica. Analyses proved that the Al/O ratio in the final product was 60:45indicating the absence of intermediate structures between the alumina and silicon particles. The product was reported to possess high electron conductivity when deposited on a carbon paste electrode. The resulting modified electrode was used for the analysis of morphine from 0.1 to 550, with a detection limit of 0.03 μM [58].

A glassy carbon electrode was modified for the simultaneous determination of morphine and ondansetron using a composite of multi-walled carbon nanotubes and Nafion. The electrode revealed enhanced response in voltametric analysis due to the synergy between the carbon-based nanostructure and Nafion. The electrode showed to distinct CV signals for ondansetron and morphine with a potential difference of 430 mV, and produced linear response from 1.0×10^{-7} to 4.0×10^{-6} M for morphine, with a detection limit of 3.1×10^{-8} M [59].

Wester et al. anodically treated an electrode composed of titanium/tetrahedral amorphous carbon and evaluated the electrode in the electrochemical analysis of morphine and paracetamol. The anodic treatment was reported to lead to the oxidation of the carbonous material, and exposure and oxidation of the titanium layer below, discriminating the peaks of the two analytes, by 2.5 V through shifting the oxidation potential of paracetamol while that of morphine did not change. The electrode could determine concentrations as low as 9.8 nM and it produced a linear response from 0.1 to 10 μM in the presence of 100 μM of paracetamol [60].

Li et al., reported the electrochemical pretreatment of a glassy carbon electrode and its application for the analysis of morphine with a detection limit of 0.2 μM even in the presence of codeine [61]. In another report a composite film of electrochemically reduced MWNTs-doped graphene oxide (ER-MWNTs-doped-GO) composite film was used for constructing a modified morphine electrode [62].

Kumary VA et al. reported using electropolymerizing copper coordinated amino acid on reduced graphene oxide via a two-step process and using the electrode for the detection of morphine with a detection limit of 47 nM and a linear response from 50 nm to 80 μM. [63].

Maccaferri et al. reported a modified screen-printed electrode with graphene oxide for morphine with a high sensitivity of 2.61 nA ppb^{-1} and detection limit of 2.5 ppb [64].

Cordova-Mateo E et al. reported evaluation of electrodes for the analysis of morphine via evaluating different electroactive polymers, namely poly(3,4-ethylenedioxythiophene) (PEDOT), poly(3-methylthiophene) (P3MT), polypyrrole (PPy), poly(N-methylpyrrole (PNMPy) and poly[N-(2-cyanoethyl)pyrrole] (PNCPy), based on a theoretical calculations on model complexes and voltametric analyses. They reported the prediction of the quantum mechanical calculations to show the binding strength of morphine and the polymers to be in

the order of PEDOT < PNMPy < Py < < P3MT ≈ PNCPy. Experimental data on the changes in the electroactivity and the anodic current at the reversal potential, on the other hand, indicated that the CV response to morphine is much higher for P3MT and PNCPy in acidic media where the former is stronger than the latter, and neutral pH where both electrodes have similar responses [65].

Atta NF et al reported a modification of a carbon paste electrode with gold nanoparticles modified with phthalocyanine complexes of Co, Ni, Cu, and Fe to construct a morphine electrode, that can analyze morphine in the presence of ascorbic and uric acids and evaluated the effect of the metal ions on the electron transfer properties of the electrode, and discovered the Co phthalocyanine based electrode to have the highest sensitivity, a detection limit of 5.48×10^{-9} M and an applicability window of 4.0×10^{-7} to 9.0×10^{-4} M [66].

In another work a voltametric immunosensor was constructed for the analysis of morphine self-assembling cysteamine modified gold particles on a graphene screen printed electrode (GSPE). This was done based on thiol interactions, to create amino groups on the surface of the electrode. Then antibodies were then covalently immobilized on the electrodes to construct morphine biosensors, which functions based on the competition of morphine and morphine-bovine serum albumin conjugate. The immunosensor produced linear response from 0.1 to 100 $ng.mL^{-1}$, and had a detection limit of 90 $pg.mL^{-1}$ [67].

Mokhtari A et al developed a modified morphine CPE based on vinyl ferrocene/MWCNT. The proposed electrode could separately measure morphine and diclofenac and discriminate them by around 300 mV in SWV. The linear response for morphine was linear from 0.2 to 250.0 μM , with a detection limit of 0.09 μM [68].

Li F et al. reported a morphine electrode based on a glassy carbon electrode modified with mesoporous carbon modified, and reported an overpotential drop of around 82 mV and an 80-fold sensitivity increase (1.74 μA/μM) compared to unmodified GCE. The electrode produced linear response from 0.1 μM to 20 μM and a detection limit of 50 nM [69].

In 2008 a a highly selective gold disk micro electrode was developed for the analysis of methyl morphine in flow systems. To enhance the sensitivity the peak currents were integrated through a set of potential The calibration plot of the peak currents vs the concentration was linear from 0.02–1.1 μM and the LOQ was reported to be 0.01 μM [70].

Pournaghi-Azar at al reported modified an Al electrode covered with metallic Pd using Prussian blue and suggested the electrooxidation of morphine on the electrode to occur through 2 distinct mechanisms at various pH. They also reported the thermodynamic and kinetic factors for the electrode [71].

Atta et al. reported a facile technique for direct electrodeposition of Au particles on a CPE and used the electrode in the analysis of morphine. The calibration plot of the method was reported to be linear from 4.0×10^{-7} to 2.0×10^{-4} M and its LOD was reported as 4.21×10^{-9} M [72].

Table 1. Cyclic voltammetry based electrochemical sensors performance for morphine

Target Analyte	Electrode	Medium	Types Of Sample	Linear range	Limit of detection	Ref
Morphine	Mesoporous carbon/GCE	0.05 M PBS (pH 7.0)	Urine	0.1–20 μM	10 nM	[69]
Morphine Paracetamol	Ti tetrahedral amorphous carbon	PBS (pH 7.4)	-	0.1−10 μM	9.8 nM	[60]
Morphine	Cobalt hexacyanoferrate chemically modified electrode	0.1 M phosphatebuffered saline (pH 4.5)	Brain Dialysis Samples	1.0×10^{-6} M to 5.0×10^{-4} M	5.0×10^{-7} M	[54]
Morphine	2-aminoethanethiol self-assembled monolayer (sam)-modified gold electrode	0.2-M PBS (pH 6.0)	-	-	-	[55]
Morphine	Gold nanoparticles modified indium tin oxide film	0.2 M PBS (pH 7)	Human Urine	8.0×10−7 to 1.6×10−5 M	2.1×10^{-7} M	[56]
Morphine	Electrodepositing gold nanotube arrays onto an anodic aluminum oxide template	0.1 M disodium hydrogen phosphate-citric acid buffer solution (pH 6.1)	Patient's Blood or Urine	1.22×10^{-7}–7.44×10^{-4} M	4.06×10^{-8} M	[57]
Morphine	Al_2O_3/NP/CPE electrode	IL/CNTPE in 0.1 M phosphate buffer solution (pH 7.0)	Human Urine and Drug	0.1–550 μM	0.03 μM	[58]
Ondansetron Morphine	Carbon nanotubes/Nafion polymer composite/GCE	0.1 MH_2SO_4	Human Serum	1.0×10^{-7}–5.0×10^{-6} M and 1.0×10^{-7}–4.0×10^{-6} M	3.1×10^{-8} and 3.2×10^{-8} M	[59]
Morphine Paracetamol	Titanium/tetrahedral amorphous carbon electrod	PBS of pH 7.4	-	0.1–10 μM	9.8 nM	[60]
Morphine Codeine	Glassy carbon electrode	0.05M PBS (pH 7.4)	Urine Samples	-	0.2 μM	[61]
Morphine Dopamine, Uric Acid Codeine	Mwnts-doped graphene oxide composite	pH 4.5 phosphate buffer solution	Urine Serum	3×10^{-7} to 1×10^{-5} M	2×10^{-7} M	[73]
Morphine Diclofenac	RGO/Cu-poly (Ala)/GCE	0.1 M PBS (pH=7) containing 0.1 mg mL^{-1} graphene oxide (GO)	Blood Serum	50 nM-80 μM	-	[63]
Morphine	Graphene oxide modified screen-printed electrodes	PBS, at pH 7.0.	Urine	2.5 ppb	-	[64]
Morphine	Gold nanoparticles/ Metalphthalocyanine modified cp-electrodes	B-R buffer pH 7.4	Urine	4×10^{-7} M to 9×10^{-4} M	5.48×10^{-9} M	[66]
Morphine	Graphene screen printed electrode modified aunp	PBS buffer pH 7.4	Saliva Samples	0.1 to 100 ng·mL−1	90 pg·mL−1	[67]
Morphine Diclofenac	Vfmcpe Vfmcntpe	PBS (pH 7.0)	Urine	0.2–250.0 μM, and 5.0–600.0 μM, respectively	0.09 and 2.0 μM respectively	[68]
Morphine	Mesoporous carbon modified/GCE	0.05 M PBS (pH 7.0)	Urine	0.1 to 20 μM	10 nM	[69]
Methyl Morphine	Flow injection analysis and fast Fourier transform cyclic voltammetry	0.05 M H_3PO_4	Human Urine Plasma	0.02–1.1 μM	0.008 μM	[70]
Morphine	PB/Pd–Al-modified electrode	0.5 M KNO_3+0.2 M acetate solution		-	-	[71]
Morphine	Gold Nanoparticles modified carbon paste electrode	B-R buffer (pH 7.4)	Urine	4.0×10^{-7} to 2.0×10^{-4} M	4.21×10^{-9} M	[72]

5.2. DPV analysis of morphine using modified electrodes

Differential pulse voltammetry is a highly sensitive technique, which as described above, tends to reduce the effect of non-faradic currents in the voltametric analyses [74]. Table 2 lists the reports on detection of morphine by DPV method.

Bagheri et al reported a developing a method for the analysis of morphine and codeine through a using Zn_2SnO_4–graphene nanocomposite for modification of a CPE which was used in the DPV analysis of the species. The response calibration plot was linear for both species from 0.020 to 15 μM and the respective detection limit for morphine was 0.011 μM. Species including Ca^{2+}, glucose, lactose, sucrose, ascorbic acid, acetaminophen, ethanol, noscapine were reported to cause no significant interference [75].

Jahani et al. used graphene nanoribbons to modify a screen printed electrode (SPE) which proved to be applicable in the concentration range of 0.07- 600.0 μM, with an LOD of 20.0 nM [76].

Aliabadi et al used a modified CPE electrode for the analysis of morphine in small volumes of samples. They used a hydrogel in the carbon paste composition. During the analyses, a droplet of the sample was absorbed into the CPE surface before immersion into the analytical cell. The electrode containing the absorbent polymeric matrix (hydrogel) had linear response from 5.0 to 200 μM (LOD: 1 μM) [77].

Verrinder et al. reported using a disposable electrochemical sensor strip for the analysis of morphine in untreated blood samples. To this end they prepared SWCNT networks on a polymer matrix, together with integrated reference and counter electrodes, and covered the strips with a thin film of Nafion. They reported a linear range of 0.5 to 10 μM and a detection limit of 0.48 μM [78].

Salajegheh et al. used modified a GCE covered with sodium alginate using a molecularly imprinted composite film, through electropolymerizing L-lysine, together with morphine (as a template). The electrode was reported to be applicable from 0.1 to 1000.0 μM and its LOD was 48 nM [79].

Navaee et al reported a graphene based electrode for the concurrent analysis of morphine, noscapine and heroin through DPV and reported respective linear response ranges up to 65, 40 and 100 μM, and LODs of 0.4, 0.2 and 0.5 μM [80].

Atta et al. used a composite of reduced graphene oxide and palladium to construct an electrode for DPV analysis of morphine, and reported the optimal electrode composition to lead to two linear ranges of 0.34 to 12 μM and 14 to 100 μM, with and LOD of 12.95 nmol L^{-1} for the former. No significant interference was reported for dopamine, ascorbic and uric acids [81].

Basiri et al. developed and electrode for morphine and diclofenac using a graphite paste electrode containing $MgFe_2O_4$. Two distinct sharp peaks were recorded in the DPV analyses for the electrooxidation of diclofenac and morphine at +0.370 and 0.540 V (*vs.* Ag/AgCl) in

neutral pH. The response was linear in the concentration window of 50 nM to 920 μM for morphine and the detection limit was reported to be 10 nM [82].

Table 2. differential pulse voltammetry based electrochemical sensors performance for morphine

Target Analyte	Electrode	Medium	Types of sample	Linear range	Limit of detection	Ref
Morphine	Chitosan/Fe_3O_4/carbon paste electrode	0.1 M PBS (pH 7)	Serum Urine	0.01–720 μM	3 nM	[89]
Morphine	Graphene/pd/GCE	0.1 M PBS (pH 7.4)	Urine	0.34–12 μM	12.9 nM	[81]
Morphine	Exfoliated graphene oxide/SPE	PBS	Urine	0−300 μM	2.5 μM	[64]
Morphine Diclofenac	$Mgfe_2o_4$/graphite paste electrode	PBS (0.1 M, pH 7.0)	Serum	0.05–920 μM	10 nM	[82]
Morphine	Ydrogel/CPE	PBS pH 7.4	Urine	5–200 μM	1μM	[77]
Morphine	RGO/Cu-poly (Ala)/GCE	0.1 M (pH 8) PBS	Serum	50nM-80 μM	78 nM & 47 nM	[63]
Dopamine Morphine	Mwcnts/Chitosan/ GCE	0.1 M PBS (pH 7.0)	Serum Urine	2–100 μM	0.24 μM	[86]
Morphine	Gold nanoparticle/CPE	B-R buffer (pH 7.4)	Urine	0.4–200 μM	4.21 nM	[72]
Heroine, Morphine Noscapine	Graphene nanosheet/GCE	0.1 M PBS (pH 8)	Serum and Urine	0.01–720 μM	0.4 μM	[80]
Morphine	NiO/ MWCNT paste electrode	0.1 M PBS (pH 7.0)	Urine	0.34–12 μM	0.14 μM	[73]
Morphine	Gold nanoparticle metal phthalocyanine carbon paste Electrode	B-R buffer (pH 7.4)	Urine	0.4–0.09 μM	5.9 nM	[66]
Morphine	Gold nanoparticle/Nafion/CPE	B–R buffer, pH 7.4	Urine	0.2–240 μM	1.3 nM	[83]
Morphine	Palladised Aluminum electrode	0.1 M PBS (pH 7.2)	Nil	2–50 μM	0.8 μM	[71]
Morphine	Ds-DNA modified Au electrode	0.1 M phosphate buffer solution (pH 5.0)	Serum Urine	0.05–500 μM	0.01 μM	[85]
Morphine	Pt nanoparticle porous Si/IL/CPE	0.2 M phosphate buffer solution (pH 6.50)	Serum	0.1–25 μM	30 nM	[90]
Morphine Codeine	$Mwcnts/sno_2$-Zn_2SnO_4/CPE	PBS (pH 6.0)	Urine	0.1–310 μM	0.009 μM	[91]
Morphine	Spe	-	Urine	0.005–2 μM	0.005 μM	[84]
Morphine in the presence of Paracetamol	Ds-DNA/SPE	PBS with ta-C	Urine	0.7–40 μM	0.07 μM	[60]
Morphine	Pretreated GCE	0.05 M PBS (pH 7.4)	Urine	4–100 μM	0.2 μM	[61]
Morphine Codeine	Zn2SnO4/graphene/CPE	B-R buffer solution (pH 7.0)	Urine	0.020–15 μM	0.011 μM	[75]
Morphine	PEDOT/Pt electrode	BR (pH 7.4)	Urine	0.3–8 μM and 10–60 μM	50 nM 68 nM	[92]
Morphine	Au np/ferrocene/cpe	B-R buffer (pH 7.4)	Urine	1–180 μM	3.5 nM	[72]
Morphine	Poly (CTAB)/GO	0.1 M PBS (pH 8)	Serum Urine	50 nM–60 μM	0.36 μM	[63]

In another work a CPE modified with Nafion was further modified using Au nanoparticles by Atta et al. During the DPV analyses the response was linear from 2.0×10^{-7} to 2.6×10^{-4} M and the LOD was 13.3×10^{-10} M [83].

Ahmar et al. reported an developing a screen-printed carbon electrode and used it together with electroextraction through a membrane for the analysis of morphine in urine specimens. The method involved the extraction of charged morphine into a 20 µL acidic media at the lumen of a hollow fiber, through a supported liquid membrane under an electrical potential. Prior to analyses the acceptor phase was mixed with 20 µL of 0.1 N sodium hydroxide and analyzed. The calibration curve was linear from 0.005 to 2.0 µg mL^{-1}. And the LOD and LOQ of the method were reported to be 0.0015 and 0.005 µg mL^{-1} [84].

Talemi et al. investigated immobilized double-stranded DNA on an Au electrode modified with mercapto-benzaldehyde and used the resulting electrode in the DPV of morphine with a linear response in the range of 0.05–500 µM and an LOD 0.01 µM [85].

Babaei et al. reported a used MWCNT and chitosan to modify a GCE and reported a linear DPV peak current/concentration plot from 2.0×10^{-6} to 1.0×10^{-4} M (LOD: 2.4×10^{-7} M) for morphine [86].

Shishehbore et al. reported modification of a CPE using 4-hydroxy-2-(triphenylphosphonio) phenolate and MWCNTs. The electrocatalytic peak current recorded during the DPV/concentration was linear from 1.0–950.0 µM (LOD: 0.066 µM). The electrode response was reported to be unaffected by the presenc3e of acetaminophen [87].

In another work an ionic liquid was used to modify a CPE composed of MWCNTs by Ensafi et al. The electrode was claimed to enhance the electrooxidation of morphine, and a linear calibration plot was recorded rom 0.45–450 µM, in DPV mode with an LOD of 0.14 µM. [88].

5.3. SWV analysis of morphine using modified electrodes

Square-wave voltammetry (SWV) is another highly sensitive electroanalytical technique for the analysis of various species with the aim of maximizing the sensitivity and accuracy [93]. Some electrodes have been developed and used to determine morphine concentration through this mode of voltammetry (Table 3).

Rezaei et al. developed a modified pencil graphite electrode using multi walled carbon nanotubes and gold nanoparticles, and reported a linear calibration plot from 0.008 to 5 µM and an LOD of 2.9 nM for morphine [94].

Sanati et al. fabricated an developed a modified carbon paste composition based on 1-methyl-3-butylimidazolium chloride [MBIDZ]Cl as an ionic liquid binder together with NiO nanoparticles and CNTs. The electrode was reported to reveal an irreversible oxidation peak for the electrooxidation of morphine at 0.61 V (vs. $Ag/AgCl_{sat}$), greatly enhanced peak currents as opposed to unmodified CPEs and an LOD as low as 0.01 µM in SWV mode [73].

Akbarian et al used NiO nanoparticles and SWCNTs together with another ionic liquid binder, namely 2, 4-dimethyl-N/-[1-(2, 3-dihydroxy phenyl) methylidene] aniline and reported the electrode to produce three distinct signals for the electrooxidation of diclofenac, morphine and mefenamic at around 247 mV, 445 mV and 697 mV. The calibration plot for morphine was linear in the concentration window of 0.9 – 400 μM, and a detection limit of 0.4 μM was achieved [95].

Table 3. Square wave voltammetry based electrochemical sensors performance for morphine

Target Analyte	Electrode	Medium	Types of Sample	Linear range	Limit of detection	Ref
Morphine	Gold/Graphene Screen Printed Electrode	PBS (pH 7.4)	Saliva	0.1–100 μM	90 nM	[67]
Morphine	Mwnts/MIP/Pencil Graphite Electrode	PBS (pH 6.0)	Serum Urine	0.08−5 μM	2.9 nM	[94]
Paracetamol in the presence of Morphine	Cdo/CPE	0.1 M PBS (pH 8.0)	Urine	0.5–800 μM	0.1 μM	[96]
Morphine	Nio/Swcnts/DDPM/ CPE	PBS (pH 7.2)	Serum	0.9–400 μM	0.4 μM	[95]
Morphine Diclofenac	Mwcnts/Vinyl Ferrocene/CPE	0.1 M PBS (pH 7.0)	Urine	0.2−250 μM	0.09 μM	[68]
Ondansetron Morphine	Mwnts-Nafion/GCE	BR buffer	Serum	0.1−4 μM	0.03 μM	[59]
Morphine	Gold Nanoparticle/ITO Electrode	0.2 M PBS (pH 7.4)	Urine	0.8–160 μM	0.2 μM	[56]
Morphine	Al_2O_3NP/CPE	0.1 M phosphate buffer solution (pH 7.0)	Urine	0.1–550 μM	0.03 nM	[58]

5.4. Linear sweep voltammetry analysis of morphine using modified electrodes

Table 4 list the reports on determination of morphine by linear sweep voltammetric method. Atta et al developed a morphine electrode based on poly(3,4-ethylenedioxythiophene) (PEDOT) together with sodium dodecyl sulphate (SDS). They also evaluated the effects of interfering species like ascorbic acid (AA) and uric acid (UA) on the analysis and reported linear responses from 0.3–8 μM and 10–60 μM and respective LODs of 50 and 68 nM [92].

Zare et al., reported a reported a modified carbon paste composition containing MgO nanoparticles, SWCNTs and an ionic liquid, namely 1-methyl-3-octylimidazolium tetrafluoroborate. The modified CPE produced a linear response in the range of 3.0 nM to 320 μM morphine solutions and its LOD was reported to be as low as 0.8 nM at surface of MgO/SWCNTs/MOCITFB/CPE. Moreover, the MgO/SWCNTs/MOC [97].

In a rather similar fashion, Afsharmanesh et al. developed a modified carbon paste composition containing ZnO nanoparticles, carbon nanotubes and 1-methyl-3-butylimidazolium bromide as the binder. The electrooxidation of morphine was recorded at

520 mV during the LSV analyses using the modified CPE, which corresponds to a 75 mV drop in the overpotential and the peak current was 5.5 folds that of unmodified electrode. The calibration plot was linear from 0.1 to 700 μM and the LOD was reported to be 0.06 μM [98].

Table 4. Linear sweep voltammetry based electrochemical sensors performance for morphine

Target Analyte	Electrode	Medium	Types of sample	Linear range	Limit of detection	Ref
Morphine	MWNTs/Grapheoxide/ GCE	0.1 M PBS (pH 4.5)	Serum Urine	0.07–6.5 μM	0.05 μM	[62]
Morphine	ZnO/MWCNTs/IL/CPE	0.1 M phosphate buffer (pH 8.0)	Urine	0.01–700 μM	0.06 μM	[98]

6. CONCLUSION

This manuscript provides a summary of the research conducted in thea area of voltammetry based modified electrodes for morphine detection. A significant number of research has been performed using nanomaterials to prepare electrodes with improved analytical features in morphine analysis. LOD at the level of nM were achieved which is acceptable. The stability and reproducibility of the of the sensors is still a remaining challenge that needs to be resolved. The use of the stable and electroactive polymers for fixing of nanomaterials on the surface of electrodes are also key factors for biosensors stability. Addressing this weakness shall allow morphine sensors to become robust techniques for quick determination of morphine various samples. However, the topic is still under investigation and various research studies and advances are under way. One possible scenario is that the research shall focus on the development of disposable electrodes for integration of the sensors in simple, cheap and portable systems.

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