

Review

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Review

Recent Advances on Electrochemical Sensors for Detection of Contaminants of Emerging Concern (CECs)

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Abstract: Contaminants of Emerging Concern (CECs), a new category of contaminants in limelight, is a major issue of global concern. The pervasive nature of CECs and its harmful effects, such as cancer, reproductive disorders, neurotoxicity etc., make the situation alarming. The perilous nature of CECs lies in the fact that even very small concentration of CECs can cause great impacts on living beings. It also has a nature of bioaccumulation. Thus, it is the ultimate need to have efficient sensors for detection of CECs to ensure safe living environment. Electrochemical sensors are an efficient platform for CEC detection as they are highly selective, sensitive, stable, reproducible, and prompt, and can detect very low concentrations of analyte. Major classes of CECs are pharmaceuticals, illicit drugs, personal care products, endocrine disruptors, newly registered pesticides, and disinfection by-products. This review focusses on CECs, including their sources and pathways, health effects caused by them, and electrochemical sensors as reported in literatures under each category for the detection of major CECs.

Keywords: contaminants of emerging concern; electrochemical sensors

1. Introduction

Population booming and the ever-increasing demands of products and facilities have induced the massive growth of industries and production. With industrialization and technological advancements, pollution of natural resources has become much evident on air, water, and soil. Unheard diseases are being reported along with new chemicals getting added to the list of harmful contaminants.

Contaminants of Emerging Concern (CECs), Emerging Contaminants (ECs), Persistent Organic Pollutants (POPs), Persistent Bioaccumulative toxic (PBT) are some of the novel terms that have recently attracted much attention. Contaminant of Emerging Concern (CEC) was defined by Diamond et al. as a chemical for which there are increasing concerns regarding its potential risks to humans and ecological systems, including endocrine disruption and neurotoxicity [1]. Sauve et al. defined CECs as naturally occurring, manufactured or manmade chemicals or materials which have now been discovered or are suspected to be present in various environmental compartments and whose toxicity or persistence is likely to alter the metabolism of a living being significantly [2]. To solve the ambiguity between emerging contaminants (EC) and CECs, ECs are those contaminants which have appeared recently. It is more of a relative term as those contaminants regarded as ECs a decade ago may longer be one in the presence. However, CECs are contaminants which have been in environment for a while but concerns are raised recently. Also, a well-known, already regulated contaminants may reattain the emerging position when new information about the contaminant emerges and regulations need to be revised [3]. At times, ECs and CECs are used interchangeably. Pharmaceuticals, personal care products (PCPs), endocrine disruptors (EDs), illicit Drugs, newly registered pesticides, and disinfection by-products (DBPs) are commonly reported CECs.

Although CECs have been polluting the ecosystem for a long time, the amounts and types of CECs have shown a tremendous increase in the past 50 years [4]. CECs are identified at very low concentrations of ng/L to µg/L in surface water, waste water, and drinking water [5]. However, the nature of bioaccumulation of CECs makes the scenario alarming. Bioaccumulation is the build-up of CECs in an organism. Some mixtures of CECs bioaccumulated are more toxic than single CECs [6]. Unlike conventional pollutants, CECs are rarely globally regulated and another risk related to CECs is associated with endocrine disruption [7], carcinogenicity [8], neurotoxicity and genotoxicity [9]. Traditional water treatment methods, like activated carbon treatment and reverse osmosis, failed to remove CECs from water [10]. Thus, it is very important to develop an efficient technique to sense CECs and provide safe food and drinking water. Electrochemical sensors, which can detect analytes at a very low concentration like picomolar range, are a better option for CEC detection as they are present in very low concentrations in environment. Also, electrochemical sensors are a cost-effective option while being rapid to detect CECs [11]. Electrochemical sensors provide a sensitive, selective, and effective platform to detect CECs in water, food, and serum samples. This review aims to explain about CECs, their sources, toxic effects of different CEC classes, and electrochemical sensors developed for the detection of these CECs.

2. Sources and Toxicity of Contaminants of Emerging Concern (CECs)

Industrial wastewater, agriculture/ livestock runoff, household and hospital effluents are the major sources of CECs, whereas water and soil are the sinks of CECs. The primary sources of CECs include ineffective waste water treatment plants, industrial waste water, agriculture and livestock runoff, hospital and domestic effluents [12]. Pharmaceuticals, personal care products, pesticides are the common CECs found in industrial effluents. Pesticides used to improve crop productivity are the common CEC found in agriculture runoff. Livestock runoff include pharmaceuticals and steroid hormones used in livestock. Hospital and domestic effluents might contain pharmaceuticals, endocrine disruptors and disinfection by-products [13]. Depending on the physicochemical properties of CECs, they seep into the soil, pollute ground water or adsorb into the soil thus contaminate the soil and ground water. Some other CECs get discharged into surface water, leading to surface water contamination [14]. Plants are the major transporters of CECs as they are the primary producers in the food chain [15]. From plants, they get transferred to higher trophic levels and may lead to bioaccumulation.

Unfortunately, CECs have become an integral part of our daily lives. Unregulated use of pharmaceuticals and personal care products have resulted in release of large number of CECs. Unchecked use of insecticides and pesticides to improve crop yield have added to the situation. Rebellious use of illicit drugs has also increased. They can cause reproductive damage, endocrine disorders [16], allergies, neurotoxicity, bioaccumulation effects, growth inhibition effects, hormone interference, disrupted physiological process, increased cancer risks, affected metabolism, transport and synthesis of endogenous hormones and specific receptors [17], and biochemical toxicity to invertebrates, amphibians and fish [18]. Major classes of CECs, representative compounds, sources, and effects are shown in Table 1.

With the increasing awareness of the toxicity of CECs hidden in our daily use products, its detection is also growing importance. EPA has developed criteria to assess and manage the risks of CECs in aquatic environment due to its extensive use, chemical persistence, consequences in nature and concerns of public [19]. Safe drinking water act (SDWA), toxic substance control act (TSCA), comprehensive environmental response, compensation and liability act (CERCLA), and clean water act (CWA) rose the interest of authorities to address the CECs issue [20]. CECs may be present in very low concentrations, but can cause serious effects on living beings and nature [21]. For example, fluoxetine at a concentration of 546 ng/L can be fatal to aquatic species [22] and ibuprofen at a minimum concentration of 250 ng/L can cause endocrine disruptions to aquatic species [23]. Therefore, a quick, selective, and sensitive method is necessary for the detection of CECs.

Table 1. Classes of CECs, their sources and toxic effects.

Classes of CECs	Description/ Categories	Representative Compounds	Sources	Effects
Pharmaceuticals	Anti-inflammatory, analgesics, antibiotics, antidepressants, Lipid lowering agents, antihistamines, β -blockers	Diclofenac, Norfloxacin, Acetylsalicylic acid, sodium salicylate, oxaprozin, ibuprofen, indomethacin, Acetaminophen, Carbamazepine, Promethazine hydrochloride, Norfloxacin	Over the counter (OTC) Pharmaceuticals, Prescribed Pharmaceuticals	Cancer, liver and kidney failure [34], neurotoxicity, cardiovascular risks, Drug induced hepatotoxicity [35]
Illicit Drugs	Non-prescribed Drugs	Cocaine, Morphine, Codeine, Amphetamine, MDMA, 6-acetylmorphine	Psychotropic drugs	Neurotoxic effects [53], Hypertension [54], low blood pressure, Respiratory problems [56], coma [64]
Personal Care Products (PCPs)	Cosmetics, Daily care products and fragrances, plasticizers, synthetic musks, UV-filters, preservatives	Methyl paraben, Ethyl paraben, Triclosan, Vanillin, Triclosan	Toothpastes, Lotions, fragrances, cosmetics, soaps, shampoos	Endocrine effects, reproductive malfunctions [67], Contact dermatitis, breast cancer [69], affects CNS [76]
Endocrine Disrupting Chemicals (EDCs)	Bisphenols (BPs), polychlorinated biphenyls (PCBs), phthalate esters, alkylphenols, natural and synthetic estrogens	BPA, BPS, Diethylstilbestrol, Estradiol, Phthalates	Chemicals used as solvents or lubricants, their by-products, plasticizers, electronic materials	Reproductive issues, neurological damages, cardiovascular diseases, diabetes [84], fertility defects, sexual abnormalities, cancer [96]
Newly Registered Pesticides	New pesticide or chemicals or different uses of existing chemicals	Imidacloprid (IDP), Thiamethoxam (TMX),	Pesticides used to improve the crop yield	Cancer, Effects on immune system, reproductive system, respiratory system [101]
Disinfection By-products (DBPs)	Chlorates, chlorites, bromates, Trihalomethanes (THMs) and Haloacetic acids (HAAs)	Chlorites, bromates, Trichloroacetic acid (TCAA), Trichloroacetamide (TCAM)	Disinfectants react with organic matter or manmade contaminants during water disinfection	Genotoxicity, carcinogenicity [114], Acquired methemoglobinemia [115], Cancer, reproductive defects [121]

3. Electrochemical sensors for detection of CECs

Electrochemical sensor is an efficient technique for CEC sensing. Conventional methods, such as liquid chromatography [24], inductively coupled plasma mass spectroscopy (ICP-MS) [25], atomic absorption spectroscopy (AAS), atomic emission spectroscopy (AES) [26], atomic fluorescence spectroscopy [27] and inductively coupled plasma-optical emission spectrometry (ICP-OES) [28], are quite expensive, time consuming, and highly skilled professionals demanding. Compared to conventional techniques, electrochemical methods are more sensitive, selective, stable, portable, and economical, and allow simultaneous detection of multiple analytes and environmentally benign [29]. Voltammetry is the most commonly used electrochemical technique to detect CECs. Voltammetric techniques measure current with corresponding variation in potential [30]. Linear sweep voltammetry (LSV), cyclic voltammetry (CV), differential pulse voltammetry (DPV), anodic stripping voltammetry (ASV), differential pulse anodic stripping voltammetry (DPASV), square wave anodic stripping voltammetry (SWASV), and linear sweep anodic stripping voltammetry (LSASV) are the subclasses of voltammetric technique based on the mode of voltage variation [31].

3.1. Electrochemical sensors for detection of Pharmaceuticals

Pharmaceuticals are one of the most widely occurring CECs. Pharmaceuticals are substances that are used for therapeutic, preventive, and diagnostic purposes and do not include the recreational drugs like cocaine [32]. Anti-inflammatories, analgesics, antibiotics, antiepileptics, antidepressants, lipid lowering agents, antihistamines, β -blockers belong to the class of pharmaceuticals [33,34]. The presence and distribution of pharmaceuticals have become an issue of major concern [35,36]. The global medicine usage achieved 4.5 trillion doses by 2020, when half of the world population consumed >1 dose/person/day of drugs [37].

Pharmaceutical products have contaminated water sources, soil, and sediments [38]. They have the nature of persistent contaminants, which are resistant to completely degrade [39]. This leads to pollution of water resources and soil, thus harming the aquatic life and other organisms. Presence of pharmaceuticals in soil can affect the biological balance in soil [40] and thus threaten the food safety [41]. Pharmaceutical products in aquatic environment have caused health effects, like cancer, liver, or kidney failure [42]. "Drug induced hepatotoxicity" implies liver damage driven by drugs, which can directly or indirectly damage liver cells, which is a vital organ for carbohydrate, protein and fat metabolism, detoxification, drug transformation, bile secretion, and vitamin storage [43]. Therefore, a number of electrochemical sensors have been developed for detection of pharmaceutical products.

Acetaminophen[N-(4-hydroxyphenyl)acetamide], commonly known as paracetamol (PA), is a painkiller used to reduce fever and pain worldwide. Excess use of PA can affect liver and kidney. Spinel vanadium nano ferrite (VFe_2O_4) modified GCE was developed for electrochemical sensing of PA in pharmaceutical and biological samples with a limit of detection of 8.20 nM [44]. Simultaneous femtomolar detection of PA, Diclofenac[2-[2-(2,6-dichloroanilino)phenyl]acetic acid,(DIC)], and Orphenadrine[(RS)-N,N-Dimethyl-2-[(2-methylphenyl)-phenyl-methoxy]-ethanamine (ORP)] was achieved using COOH-CNTs/ZnO/NH₂-CNTs modified on GCE, where ZnO nanoparticles were trapped in between the COOH-CNT and NH₂-CNT [45]. DIC, an antihistamine, may affect central nervous system and ORP, an antirheumatic drug, can cause heart attack. 46.8, 78, and 60 fM, respectively, were the limit of detection of PA, DIC, and ORP by COOH-CNTs/ZnO/NH₂-CNTs/GCE. Detection of DIC in human urine samples was reported using exfoliated graphene supported cobalt ferrite (EGr-Co_{0.2}Fe_{1.8}O₄) modified SPCE, synthesized by an ultrasonication method as shown in Figure 1(a) [46]. EGr-Co_{0.2}Fe_{1.8}O₄/SPCE outperformed Co_{0.2}Fe_{1.8}O₄/SPCE, Gr-Co_{0.2}Fe_{1.8}O₄, and bare SPCE in CV analysis for detection of DIC as shown in Figure 1(b). With increase in concentration of DIC, the peak current value also increases (Figure 1(c)) in DPV analysis and shows a linear relationship between concentration of analyte and current value from which a limit of detection of 1 nM and sensitivity of 1.059 $\mu A \mu M^{-1} cm^{-2}$ were deciphered. Copper-aluminium layered double hydroxide homogeneously dispersed over oxidized graphitic carbon nitride modified GCE (oxidized g-C₃N₄/Cu-Al LDH/GCE) was fabricated for the detection of diclofenac sodium (DS). Due to the combined effect of oxidized g-C₃N₄ and Cu-Al LDH, a good electrochemical response for DS

oxidation and enhanced current was obtained. The sensor could achieve a detection limit of 0.38 μM and linear range of 0.5–60 μM using DPV technique [47].

Ibuprofen [(RS)-2-(4-(2-methylpropyl)phenyl)propanoic acid, (IBU)] is the third highly consumed non-steroidal anti-inflammatory drug used to relieve pain, arthritis, and inflammatory diseases. Over dose of IBU can raise the risk of heart attack and due to its antiplatelet outcome, it is known as blood- thinning drug. For the detection of IBU, copper tellurate (Cu_3TeO_6) with a 3D-stone like morphology was developed by a wet-chemical method [48]. $\text{Cu}_3\text{TeO}_6/\text{GCE}$ reported a detection limit of 0.017 μM and linear range of 0.02–5 μM and 9–246 μM . Nitrofurazone [(2E)-2-[(5-Nitro-2-furyl)methylene]hydrazine carboxamide, (NZ)] is an antibiotic used to treat ulcers, bladder cancer, skin inflammation, and gastrointestinal infections. NZ has genotoxic, mutagenic, and carcinogenic effects. Sulfur doped graphitic carbon nitride with copper tungstate hollow spheres (Sg- $\text{C}_3\text{N}_4/\text{CuWO}_4$) was synthesized by ultrasonic method [49]. S/g- $\text{C}_3\text{N}_4/\text{CuWO}_4/\text{GCE}$ exhibits a detection limit of 3 nM and sensitivity of 1.24 $\mu\text{A}\mu\text{M}^{-1}\text{cm}^{-2}$ towards detection of NZ in human urine and serum samples. Norfloxacin [1-ethyl-6-fluoro-4-oxo-7-piperazin-1-yl-1H-quinoline- 3-carboxylic acid (NFX)] is a fluoroquinolone used to treat human and veterinary infections. NFX can disrupt the endocrine of aquatic organisms. $\text{CaCuSi}_4\text{O}_{10}/\text{GCE}$ was developed for the detection of NFX. The inter layer spaces of layered silicate structure offered suitable adsorption sites. In order to improve conductivity, MnO_2 was also added. The sensor showed a detection limit of 0.0046 μM and linear range of 0.01–0.55 and 0.55–82.1 μM [50].

Naproxen sodium (NAP) and Sumatriptan (SUM) are drugs which have shown anti-viral effects against COVID-19. NAP [(+)-(S)- 2-(6-methoxynaphthalene-2-yl) propanoic acid] is used to relieve pain and rheumatic disorders. Overdose of NAP can lead to kidney or liver disease. SUM [1-[3-(2-dimethyl aminoethyl)-1 H-indol-5-yl]-N-methylmethanesulfonamide] is a tryptamine-based medicine used to treat migraine headaches. SUM overdose can narrow blood vessels leading to heart issues like heart attack. Auxillary use of NAP and SUM are associated with serotonin syndrome. Multiwalled carbon nanotube decorated with ZnO, NiO, and Fe_3O_4 nanoparticles on glassy carbon electrode (GCE) ($\text{ZnO}/\text{NiO}/\text{Fe}_3\text{O}_4/\text{MWCNTs}$) was developed for the detection of NAP and SUM with a detection limit of 3 nM and 2 nM, respectively [51]. The high electrical conductivity and electroactive surface area of the composite improved the electrooxidation of NAP and SUM. The linear range of detection were 4.00 nM to 350.00 μM for NAP and from 6.00 nM to 380.00 μM for SUM. Yet another electrode developed for the simultaneous detection of NAP and SUM was a carbon paste electrode modified with peony like $\text{CuO}-\text{Tb}^{3+}$ nanostructure (P-L $\text{CuO}:\text{Tb}^{3+}$ NS/CPE) [52]. P-L $\text{CuO}:\text{Tb}^{3+}$ NS/CPE gave a linear range of 0.01-800 μM and 0.01–700 μM and detection limit of 3.3 nM and 2.7 nM for detection of SUM and NAP.

Vortioxetine (VOR) (1-[2-(2,4-dimethyl-phenylsulfanyl)-phenyl]-piperazine- hydrobromide) is an antidepressant used in the treatment of major depressive disorder. VOR is associated with sexual dysfunction, liver injury, anxiolytic like behavior in children, histopathological damage, nausea and vomiting [53]. Vortioxetine (VOR) detection was made possible using an electrochemical sensor based on gold nanoparticles/graphene ($\text{AuNP}_s@\text{GRP}$) modification on GCE [54]. $\text{AuNP}_s@\text{GRP}$ synthesized by a one pot method enhanced the sensitivity of the sensor for selective detection of VOR. The electrochemical sensor could detect VOR at a detection limit of 50 nM and linear range of 0.1–1.0 and 1.0–6.0 μM in pharmaceutical samples. Promethazine hydrochloride (PMHC) [(N, N-dimethyl-1-phenothiazin-10-yl-propan-2-amine hydrochloride) is another drug commonly prescribed for mental illness. PMHC overdose is related to cardiac problems and reproductive dysfunction, which can be fatal. A hybrid of barium tungstate with functionalized carbon black modified on SPCE ($\text{BaWO}_4/\text{f-CB}/\text{SPCE}$) was fabricated for the detection of PMHC [55]. $\text{BaWO}_4/\text{f-CB}$ was synthesized by co-precipitation followed by ultrasonication method and then drop casted on SPCE for PMHC sensing as shown in Figure 1(D). As compared to bare SPCE, $\text{BaWO}_4/\text{SPCE}$ and $\text{f-CB}/\text{SPCE}$, $\text{BaWO}_4/\text{f-CB}/\text{SPCE}$ gave better oxidation and reduction peak currents (Figure 1(E)) and the efficiency of the electrode for PMHC sensing was proved. A detection limit of 29 nM was inferred from the DPV analysis with increase in concentration of PMHC (Figure 1(F)) and the resulting linear graph of current and concentration. Carbamazepine (CBZ) [5H-dibenzo [b,f]azepine-5-carboxamide] is an

anticonvulsant medicine used for treatment of bipolar disease, mental disorder, and post-traumatic stress disorder. Growing awareness about the toxicity of CBZ is due to the harmful consequences like neurotoxicity. Gadolinium vanadate nanostructure decorated on functionalized carbon nanofiber on glassy carbon electrode ($GdVO_4/f\text{-CNF}/GCE$) was developed for the detection of CBZ in pharmaceuticals and human urine samples [56]. The synergistic effect between $GdVO_4$ and $f\text{-CNF}$ enhanced the electrochemical performance of the sensor. The detection limit of $0.0018 \mu\text{M}$ and linear range of $0.01\text{--}157 \mu\text{M}$ were achieved using the electrochemical sensor for CBZ detection.

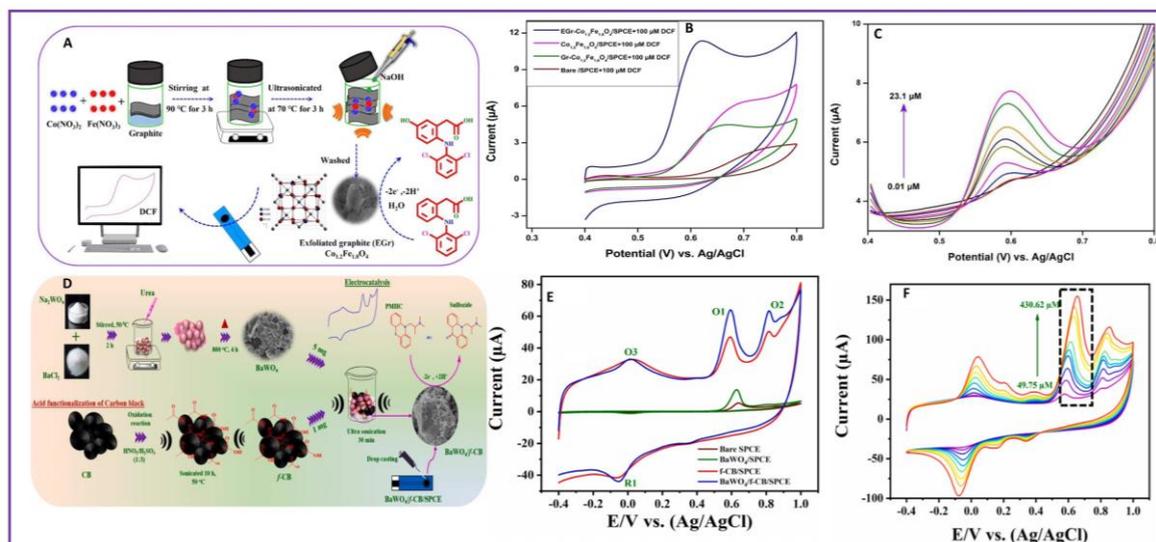


Figure 1. Scheme of fabrication of (A) $EGr\text{-}Co_{1.2}Fe_{1.8}O_4/SPCE$ (D) $BaWO_4/f\text{-CB}/SPCE$; Comparison of CVs of different electrode for detection of (B) DIC (D) PMHC (C) DPV of $EGr\text{-}Co_{1.2}Fe_{1.8}O_4/SPCE$ for detection of DIC at different concentrations; (E) CV of $BaWO_4/f\text{-CB}/SPCE$ for detection of PMHC at different concentrations.

3.2. Electrochemical sensors for detection of illicit drugs

Illicit drugs are non-prescribed drugs or psychotropic substances [57]. International Drug Control Conventions prohibit the production, sale, and use of illicit drugs [58]. Cocaine, benzoylcegonine, morphine, codein, 6-acetylmorphine, methadone, amphetamine, methamphetamine, and 3,4-methylenedioxyamphetamine are common illicit drugs. According to World Drug Report 2023 launched by the UN Office on Drugs and Crime (UNODC), over 296 million people used drugs in 2021, which accounts to be 23 percentage greater than the previous decade. The number of people suffering from drug disorders shot up to 39.5 million, with 45 per cent increase over 10 years [59]. These drugs are consumed by means of ingestion, inhalation, absorption, injection, smoking, dissolution under tongue or skin patching. Due to the stimulus that these drugs provide, people use them even though they are dangerous and become addicted to these drugs. These drugs affect both mentally and physically. Driving Under Influence of Drugs (DUID) has led to many road accidents [60]. Drug-assisted sexual harassment is another issue of major concern. The misuse of illicit drugs can have severe neurotoxic effects [61].

Cocaine (Methyl (1R,2R,3S,5S)-3-benzoyloxy-8-methyl-8-azabicyclo [3.2.1] octane-2-carboxylate) is a Central Nervous System (CNS) stimulant. Risks associated with cocaine include hypertension, altered mental status, seizure, chest pain, headache, neurological defects, corneal ulceration and vision loss, HIV, and hepatitis [62]. Octahedral palladium doped cobaltite composite modified GCE ($Oh\text{-Pd}^{2+}: Co_3O_4\text{-C}/GCE$) was fabricated for cocaine detection in biological specimens [63]. $Oh\text{-Pd}^{2+}: Co_3O_4\text{-C}$ composite was synthesized by hydrothermal method. The large surface area and higher electrocatalytic activity of the composite gave a detection limit of 1.3 nM and linear range of $0.01 \mu\text{M}\text{--}900.0 \mu\text{M}$.

Morphine is an opioid used to relieve moderate to severe pain in cancer patients. Like other opioids, morphine also cause addiction, low blood pressure, and respiratory problems [64].

Polydopamine-functionalized multiwalled carbon nanotube modified GCE (PDA-f-MWCNT/GCE) was used for the detection of morphine [65]. The PDA-f-MWCNT catalyst has raised the electrochemical activity for morphine oxidation. The detection limit obtained using this sensor was 0.06 μM and linear range of detection of morphine in human plasma and urine samples was 0.075–75.0 μM .

Amphetamine-type stimulant (ATS) is a group of synthetic drugs, chemically derived from β -phenethylamine, that stimulates the central nervous system. Amphetamine (A), methamphetamine (MA), 3,4- methylenedioxyamphetamine (MDA), 3,4-methylenedioxymethamphetamine (MDMA), and 3,4-methylenedioxyamphetamine (MDEA, MDE or "Eve") are the common drugs in this category [66]. MDMA is a psychoactive recreational hallucinogenic abuse drug used worldwide. Long term effects of MDMA include kidney failure, hepatotoxicity, decreased immunity [67]. An electrochemical sensor was developed for the simultaneous detection of morphine and MDMA simultaneously using carbon nanohorns-chitosan decorated with Pt nanoparticles (CNH-CHI@PtNPs) [68]. Schematic representation of the electrode fabrication for sensing morphine and MDMA is given in Figure 2(A). CNH-CHI@PtNPs/GCE showed a better catalytic performance than CNH-CHI/GCE and bare GCE [Figure 2(B)]. A linear range of 0.05-25.4 μM and detection limit of 0.02 $\mu\text{mol/L}$ and 0.018 $\mu\text{mol/L}$ were obtained using DPV analysis [Figure 2(C)] for detection of morphine and MDMA, respectively. MDEA has similar effects as that of MDMA. Fast, on-site detection of MDEA was made possible with an electrochemical sensor using carbon-screen printed electrode(C-SPE) which yielded a detection limit of 0.03 $\mu\text{mol L}^{-1}$ and linear range of 2.5 to 30.0 $\mu\text{mol L}^{-1}$ [69].

Oxycodone [14-hydroxy-7,8dihydrocodeinone, OXY] and codeine [7,8-didehydro-4,5-epoxy-3-methoxy-17methylmorphinan-6-ol monohydrate, COD] belong to the class of opiod analgesics used for pain relief. The use of opiod analgesics is controversial due to issues like dependence, tolerance, addiction, and abuse [70]. CoFe_2O_4 modified carbon paste electrode was used for the detection of OXY and COD simultaneously in human plasma and urine samples [71]. The enhanced electrocatalytic activity of the modified electrode provided a good platform for oxidation of OXY and COD. The sensor yielded a detection limit of 0.050 $\mu\text{mol L}^{-1}$ for OXY and 0.02 $\mu\text{mol L}^{-1}$ for COD. Clonazepam (CNZ), (5-(2-Chlorophenyl)-1, 3-dihydro-7-nitro-2H- 1, 4-benzodiazepin-2-one), also known as Klonopin, belongs to the group of benzodiazepines. CNZ is used for the treatment of seizure, anxiety, insomnia, and amnesia. CNZ is misused criminally and recreationally. It can cause impaired coordination, distress, and coma [72]. A hybrid nanocomposite of cobalt oxyhydroxide nanoflakes and rGO nanosheet as the support (CoOOH/r-GO) was used for detection of CNZ [73]. The synthesis scheme of CoOOH nanoflakes and CoOOH-rGO nanocomposite is given in Figure 2(D). CoOOH/r-GO/SPCE outperformed r-GO/SPCE, CoOOH/ SPCE and bare SPCE, which is evident from the DPV curve as shown in Figure 2(E). CoOOH/r-GO/SPCE was used to analyze CNZ by DPV technique with variation in CNZ concentration of [Figure 2(F)], giving a linear relationship between current signal and CNZ concentration. From this, the detection limit was found to be 38 nM and the sensor performed well in the linear range of 0-350 μM . Five illicit drugs, including cocaine, heroin, MDMA, 4-chloro-alpha-pyrrolidinovalerophenone, and ketamine, were detected simultaneously with SDS functionalized SPE with square-wave adsorptive stripping voltammetry (SWAdSV) [74]. The SDS adsorbed at the electrode surface provided adsorption sites for illicit drugs and hence improved the electrochemical output. The sensor yielded a detection limit of 0.7 μM , 1.8 μM , 0.9 μM , 1.6 μM , and 1.1 μM for cocaine, heroin, MDMA, Cl-PVP, and ketamine, respectively.

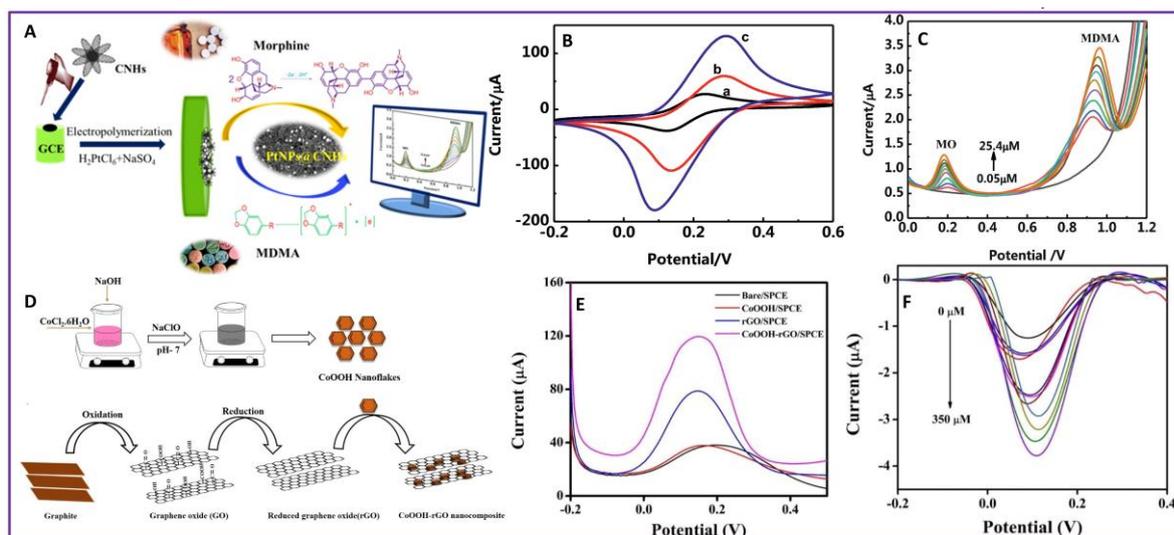


Figure 2. Scheme of fabrication of (A) CNH-CHI@PtNPs/GCE (D) CoOOH/r-GO/SPCE; (B) Comparison of CVs of different electrodes for detection of MO and MDMA; (C) DPV of CNH-CHI@PtNPs/GCE at different concentrations of MO and MDMA (E) Comparison of DPVs of different electrodes for detection of CNZ; (F) DPV of CoOOH/r-GO/SPCE at different concentrations CNZ.

3.3. Electrochemical sensors for detection of Personal Care Products (PCPs)

PCPs are an important category of CECs. PCPs include a number of chemicals used in cosmetics and daily care products including UV-filters, fragrances, plasticizers and preservatives. PCPs are detected in aquatic environment worldwide [75]. Since PCPs are mostly used on human epidermis, they have a higher chance to spread through the atmosphere. PCPs may cause endocrine effects, bioaccumulate in aquatic organisms, cause antibiotic resistance, reproductive malfunctions such as reduction in semen quality or changes in normal development of male genitals [76].

Parabens (alkyl-p-hydroxybenzoates) are the commonly used antimicrobial preservative used in cosmetics, toiletries, pharmaceuticals and food to increase the shelf life of products. Benzyl, butyl, ethyl, isobutyl, isopropyl, methyl, and propyl parabens are the common parabens in use. Of these, methyl paraben and propyl parabens are commonly used in cosmetics. Methyl parabens can cause allergic contact dermatitis and breast cancer [77]. Two electrochemical sensors based on gold nanoparticles decorated over activated carbon modified pencil graphite electrode (AuNPs@AC/PGE) and gold nanoparticles decorated over graphene oxide modified pencil graphite electrode (AuNPs@GO/PGE) were developed and their ability to sense methyl paraben in cosmetic samples were compared [78]. AuNPs@GO/PGE showed a better performance by giving a low detection limit of 2.02 μM against the 2.17 μM given by AuNPs@AC/PGE. Zinc oxide nanoparticles of different morphologies-ZnO nanowires (ZA 8), nanocuboids(ZA 10) and nanospheres (ZA 12) were synthesized by varying the pH and their sensitivity towards detection of methylparaben were compared[79]. GCE/ZA 8 exhibited better performance than GCE/ZA 10 and GCE/ZA12 towards methyl paraben detection. GCE/ZA 8 demonstrated a linear range of 0.02–0.12 mM and detection limit of 7.25 μM. The electrode also proved to be an attractive candidate for antifouling applications.

Dihydroxybenzenes have two hydroxyl groups (-OH) on the benzene ring. The three structural isomers of dihydroxybenzene are 1,2-dihydroxybenzene (catechol, CC), 1,3- dihydroxybenzene (resorcinol, RS), and 1,4- dihydroxybenzene (hydroquinone, HQ). They are widely used in cosmetics, especially in whitening creams and hair dyes. They are associated with carcinogenesis, allergies, and DNA damage [80]. Nitrogen doped nickel carbide spheres (N-NiCSs) were synthesized by varying the type of surfactant, surfactant-to-Ni molar ratio, reaction temperature, and reaction time [81]. Scheme for synthesis of N-NiCS with its fabrication over GCE and electrochemical detection of CC, RS, and HQ is shown in Figure 3(A). On comparison of the electrochemical activity of bare GCE, N-doped nickel oxide spheres/GCE (N-NiO/GCE) and N-NiCS/GCE, N-NiCS/GCE appeared to have better sensitivity for detection of HQ, CC, and RS from the DPV analysis as shown in Figure 3(B). The

DPV analysis with increase in concentration of the three dihydroxybenzene isomers exhibited an increase in peak current value (Figure 3(C)). A linear relationship was established between the concentration of the HQ, CC, and RS and current value. The detection limits of HQ, CC, and RS were inferred from the graph as 0.00152 μM , 0.015 μM , and 0.24 μM , respectively, and linear range of detection as 0.005-100 μM , 0.05-200 μM , and 5-500 μM for HQ, CC, and RS, respectively.

Tert-butylhydroquinone (TBHQ) is a synthetic phenolic antioxidant used as an additive to prevent the oxidation of food, cosmetic, oil, and fats during storage and processing. TBHQ overuse can lead to stomach, DNA and liver damage. GCE modified with MnO_2 electrodeposited on electrochemically reduced graphene oxide ($\text{MnO}_2/\text{ERGO}/\text{GCE}$) was developed for the detection of TBHQ [82]. The sensor has demonstrated a linear range of 1-50 μM and 100-300 μM and detection limit of 0.8 μM . Vertically-ordered mesoporous silica films were fabricated on graphene ($\text{VMSF}/\text{ErGO}/\text{GCE}$) for electrochemical detection of TBHQ in cosmetics and edible oils [83]. GO was reduced by cathodic reduction and vertically-ordered mesoporous silica films were grown over ErGO by electrochemical assisted self-assembly (EASA) method as shown in Figure 3(D). The electrochemical performance of $\text{VMSF}/\text{ErGO}/\text{GCE}$ was better than bare GCE, ErGO/GCE , and GO/GCE , which was evident from the CV curves (Figure 3(E)). Using DPV technique, $\text{VMSF}/\text{ErGO}/\text{GCE}$ could detect TBHQ at a detection limit of 0.23 nM and linear range of 0.001-0.5 and 0.5-120 μM using DPV technique (Figure 3(F)). Another electrochemical sensor based on $\text{ZnO}/\text{ZnNi}_2\text{O}_4$ @porous carbon@covalent-organic framework ($\text{ZnO}/\text{ZnNi}_2\text{O}_4$ @porous carbon@ COF_{TM}) was constructed for the detection of paracetamol and TBHQ [84]. The formed core-shell $\text{ZnO}/\text{ZnNi}_2\text{O}_4$ @porous carbon@ COF_{TM} nanocomposite had an active $\text{ZnO}/\text{ZnNi}_2\text{O}_4$ @porous carbon as the core and porous N, O-rich COF_{TM} as the shell. The detection limit for paracetamol and TBHQ was 12 nM and 15.95 nM, respectively.

Ultraviolet filters are considered as CECs. They are found in PCPs and industrial products like plastics, paints, rubber, etc. which require photo degradation protection. With the rising awareness of the skin cancer and photoaging associated with sun radiation, the use of sun protection factor (SPF) has become inevitable. However, the organic ultra-violet filter (UVF) tends to bioaccumulate and thus get transferred to the offspring. They also show damaging effects on central nervous system and reproductive organs [85]. Oxybenzone and octocrylene are two commonly used UV filters. 2-hydroxy-4-methoxybenzophenone (oxybenzone/benzophenone-3 or BP3) and octocrylene (2-Ethylhexyl 2-cyano-3,3-diphenylprop-2-enoate, OC) were simultaneously detected using SPCE [86]. The electrochemical sensor could sense BP3 at a detection limit of 1.9 μmolL^{-1} and OC at a limit of detection of 4.1 μmolL^{-1} .

Triclosan (2,4,40-trichloro-20-hydroxydiphenyl ether, TCS) is an antibacterial agent used widely in toothpaste, soaps, detergents, deodorants, sanitizers, and cleansers [87]. TCS can prevent fatty acid formation and damage chlorophyll in algae. On exposure to light, TCS break down releasing chlorophenols and low chlorinated dioxins, which are poisonous [88]. A ternary nanocomposite of rGO modified porous Cu-benzene tricarboxylic acid metal organic framework (Cu-BTC MOF) decorated NiCo bimetallic nanoparticle modified GCE was fabricated for the detection of TCS [89]. High electrical conductivity of rGO, greater surface area of Cu-BTC MOF, and the electrocatalytic nature of NiCo bimetallic nanoparticles resulted in a low detection limit of 0.23×10^{-12} M and linear range of 49×10^{-6} M to 0.39×10^{-12} M.

Vanillin (4-hydroxy-3-methoxybenzaldehyde) is an aromatic compound used as a fragrance constituent used in cosmetics and as a food additive in chocolates, ice creams, and beverages. Synthetic vanilla, if consumed in unchecked quantities, can cause headaches, nausea and may affect kidney functions [90]. Manganese dioxide nanowire functionalized reduced graphene oxide modified GCE ($\text{MnO}_2\text{NWs-rGO}/\text{GCE}$) was effective for detection of vanillin [91]. The unique electrochemical properties of r-GO and MnO_2 NWs resulted in significant improvement in oxidation peak of vanillin. The sensor could yield a detection limit of 6 nM and linear ranges of 0.01-20 μM and 20-100 μM from SDLSV curve.

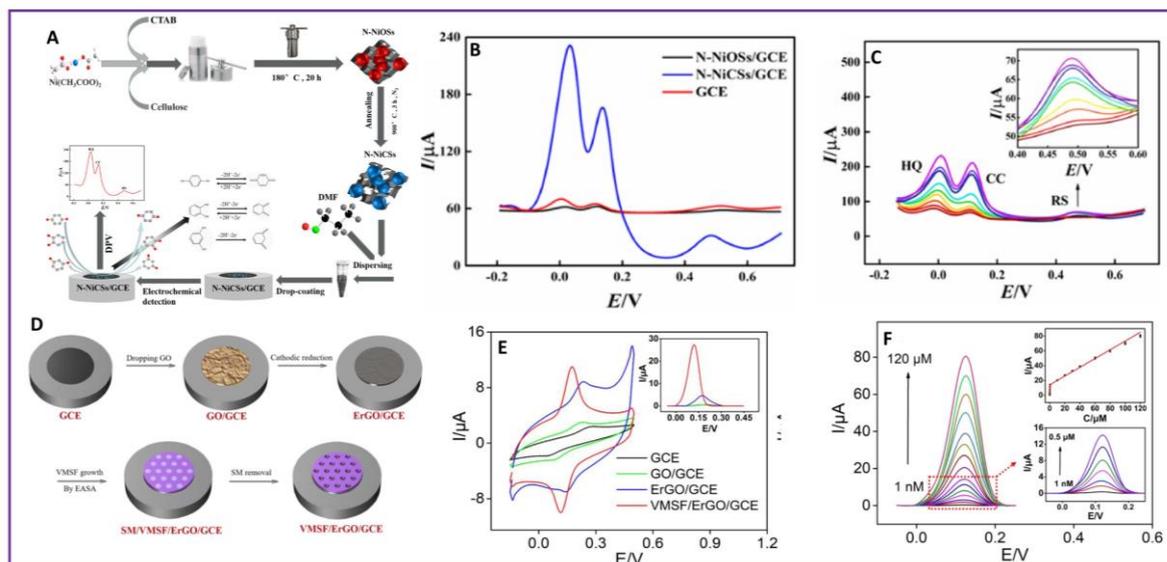


Figure 3. Scheme of fabrication of (A) N-NiCS/GCE (D) VMSF/ErGO/GCE; Comparison of (B)DPVs of different electrodes for detection of HQ, CC, and RS; (E) CVs of different electrodes for detection of TBHQ; DPV curves of (C) N-NiCS/GCE at different concentrations of HQ, CC and RS; (F) VMSF/ErGO/GCE at different concentrations of TBHQ.

3.4. Electrochemical sensors for detection of Endocrine Disrupting Chemicals (EDCs)

According to WHO, Endocrine disrupting chemicals (EDCs) are exogenous substances that can interfere with human and animal endocrine systems and thus adversely affect organisms and even their progeny [92]. Bisphenols (BPs), polychlorinated biphenyls (PCBs), phthalate esters, alkylphenols, natural and synthetic estrogens are common EDCs as reported. Even minute concentration of EDCs in water can have acute health impacts on living organisms. Reproductive issues, neurological damages, cardiovascular diseases, diabetes and many other diseases may be caused by the exposure to EDCs [93].

Bisphenol A (4,4'-(propane-2,2-diyl) diphenol, BPA) is a component widely used in plastics, baby bottles, storage containers, and epoxy coatings in metal cans. BPA belongs to the class of EDCs and can lead to reproductive issues, infertility, early puberty, cardiovascular diseases, cancer, neurological and metabolic diseases. Nanocomposite of MAX phase material, $\text{Mo}_2\text{Ti}_2\text{AlC}_3$ with MWCNT ($\text{Mo}_2\text{Ti}_2\text{AlC}_3/\text{MWCNT}$) was synthesized for sensing BPA in milk pack, plastic bottle, and metal cans [94]. The schematic representation of the detection mechanism is given in Figure 4(A). The synergistic effects of $\text{Mo}_2\text{Ti}_2\text{AlC}_3$ and MWCNT improved the electrocatalytic activity of the electrode compared to $\text{Mo}_2\text{Ti}_2\text{AlC}_3/\text{GCE}$ and MWCNT/GCE as depicted by the CV curves in Figure 4(B). With the use of DPV technique, $\text{Mo}_2\text{Ti}_2\text{AlC}_3/\text{MWCNT}/\text{GCE}$ was used for sensing BPA by increasing the concentration of BPA as shown in Figure 4(C). Then, a linear relationship was obtained for concentration of BPA and current value, from which a limit of detection of 2.7 nM and linear range of 0.01–8.50 μM were figured out. Recently, another electrochemical sensor using a composite of 2-dimensional layered titanium carbide ($(\text{Ti}_3\text{C}_2\text{T}_x)$ MXene with V_2O_5 modified GCE was fabricated for the detection of BPA [95]. DPV technique exhibited a detection limit of 87 nM and linear range of 414 nM–31.2 μM for BPA sensing. Bisphenol S (Bis(4-hydroxyphenyl) sulfone, BPS) is similar in structure to BPA, except for the sulfone (SO_2) group present in the central linker instead of dimethylmethylene group ($\text{C}(\text{CH}_3)_2$) as in the case of BPA. BPS is often used as a substitute for BPA. BPS also shows adverse effect on reproductive health, heart, and neurobehaviors. Due to similarities in structure and health effects, BPA and BPS need to be determined simultaneously. A highly porous covalent organic framework, CTpPa-2 modified GCE was constructed for the simultaneous detection of BPA and BPS in bottle samples [96]. The sensor could detect BPA and BPS at a detection limit of 0.02 μM and 0.09 μM and linear range of 0.1–50 μM and 0.5–50 μM , respectively. Dimethyl bisphenol A (2,2-bis(4-hydroxy-3-toluene), DM-BPA), is another EDC, which has similar adverse effects. Simultaneous

detection of BPA and DM-BPA was reported with SPCE modified with a composite of Pt nanoparticles modified on single-walled carbon nanotube (Pt@SWCNT), MXene (Ti_3C_2), and graphene oxide (GO) (Pt@SWCNTs- Ti_3C_2 -rGO/SPCE) [97]. The detection limit for of BPA and DM-BPA obtained with DPV technique were 2.8 nmolL^{-1} and 3 nmolL^{-1} respectively. Tetrabromobisphenol A (4,4'-(Propane-2,2-diyl) bis(2,6-dibromophenol), TBBPA) is another EDC. A conductive composite of carboxylic carbon nanotube and cobalt imidazole framework (CNTs@ZIF-67) modified on acetylene black electrode (CNTs@ZIF-67/ABE) was developed for TBBPA detection and the composite showed excellent adsorption capacity for TBBPA detection [98]. The excellent adsorption capacity of the composite resulted in a low detection limit of 4.23 nmolL^{-1} and linear range of $0.01\text{--}1.5 \text{ }\mu\text{molL}^{-1}$.

Bisphenol F (4,4'-Methylenediphenol, BPF), structural analogue of BPA, is used as an alternative for BPA in the market. BPF has the potency to cause fertility defects and adverse effects on brain development in mammals, and deplete glutathione levels in humans, resulting in an increased oxidative stress to raise cancer incidences [99]. An electrochemical sensor with carbon paste electrode modified with zinc oxide reduced graphene oxide and cetyltrimethylammonium bromide (ZnO/G/CTAB/MPCE) was used to detect BPF in water samples, human body fluids, and canned drinks [100]. BPF was recognized with DPV technique and it yielded a detection limit of $0.06 \text{ }\mu\text{M}$ and linear range of $0.5\text{--}10 \text{ }\mu\text{M}$.

BPSIP(4-(4-isopropoxy-benzenesulfonyl)-phenol) was introduced as an alternative dye developer to BPS. BPSIP also has endocrine disrupting effects, such as estrogenicity and antiandrogenicity [101]. BPSIP sensing in river water samples was implemented by a nanocomposite of graphitic carbon nitride and copper coordinated dithioamide metal organic framework modified on GCE (g- C_3N_4 /Cu-DTO MOF/GCE) [102]. A detection limit of $0.02 \text{ }\mu\text{M}$ and sensitivity of $0.5675 \text{ }\mu\text{A}\mu\text{M}^{-1}\text{cm}^{-2}$ was achieved using the sensor with DPV technique. Recently, another electrochemical sensor based on graphene modified SPCE (g- C_3N_4 @GN/SPCE) was developed for the detection of BPSIP [103]. g- C_3N_4 @GN/SPCE showed a good electrochemical response to BPSIP oxidation due to the synergistic effect of g- C_3N_4 and GN/SPCE. The sensor offered a detection limit of $0.02 \pm 0.01 \text{ }\mu\text{M}$ and linear ranges of $1\text{--}100 \text{ }\mu\text{M}$ and $100\text{--}1000 \text{ }\mu\text{M}$.

o-phenylphenol (OPP) and butylparaben (BP) are endocrine disrupting chemicals. Mn_2O_3 with various morphologies, including spherical, dumbbell-like, cubic, and ellipsoidal (S- Mn_2O_3 , D- Mn_2O_3 , C- Mn_2O_3 , E- Mn_2O_3), were synthesized and their activity towards OPP and BP sensing were evaluated [104]. Of these, E- Mn_2O_3 showed a better performance. In order to improve the performance of the sensor, a GO-wrapped E- Mn_2O_3 modified GCE was developed (E- Mn_2O_3 @GO/GCE), which yielded a detection limit of 0.63 nM and 0.88 nM for OPP and BP, respectively.

17α -Ethinylestradiol (EE2) is a synthetic hormone used widely as contraceptives, for the treatment of menopausal and postmenopausal syndrome, breast cancer in postmenopausal women. However, higher amount of EE2 intake can cause sexual abnormalities, decrease fertility, and cause cancer [105]. Carbon black supported palladium nanoparticles (CB/Pd NPs) were synthesized for the electrochemical detection of EE2 [106]. The synergistic effect of CB and Pd NPs enhanced the electrooxidation of EE2. The detection limit attained with CB/PdNPs/GCE was 81 nmolL^{-1} and linear range of $0.5\text{--}119 \text{ }\mu\text{molL}^{-1}$. 17β -estradiol (17β -E2) is a natural steroid hormone existing in mammals. Side effects of 17β -E2 include endocrine disorders, cancer, obesity, diabetes, and neurological disorders [107]. Wrinkled mesoporous carbon synthesized from wrinkled silicon nanoparticle as a sacrificial template with modified GCE ($w\text{MC}_{0.67}$ /GCE) was used for the detection of 17β -E2 [108]. The step-by-step procedure for synthesis of $w\text{MC}/\text{GCE}$ and the mechanism for detection of 17β -E2 is given in Figure 4(D). $w\text{MC}_{0.67}/\text{GCE}$ was demonstrated to have a better electrocatalytic activity and electron transfer compared to bare GCE and other modified electrodes as shown in the CV curves of Figure 4(E). With increase in concentration of 17β -E2 added, the peak current value showed an increase in Figure 4(F). From the linear relationship drawn between the concentration of 17β -E2 and current value, a detection limit of 8.3 nM and linear ranges of $0.05\text{--}10$ and $10\text{--}80 \text{ }\mu\text{M}$ by increasing the concentration of 17β -E2 in real samples, such as milk and river water, were accomplished using the

sensor. Diethylstilbestrol (DES) is another hormone that has similar side effects as 17 β -E2. An electrochemical sensor for the simultaneous analysis of 17 β -E2 and DES using Fe₃O₄-doped nanoporous carbon (Fe₃O₄-NC) synthesized by carbonization of Fe-porous coordination polymer (Fe-PCP) was developed [109]. The strong adsorptive and catalytic performance of Fe₃O₄-NC granted a detection limit of 4.6 nmolL⁻¹ and 4.9 nmolL⁻¹, and linear range of 0.01-12 μ molL⁻¹ and 0.01-20 μ molL⁻¹ for sensing DES and 17 β -E2.

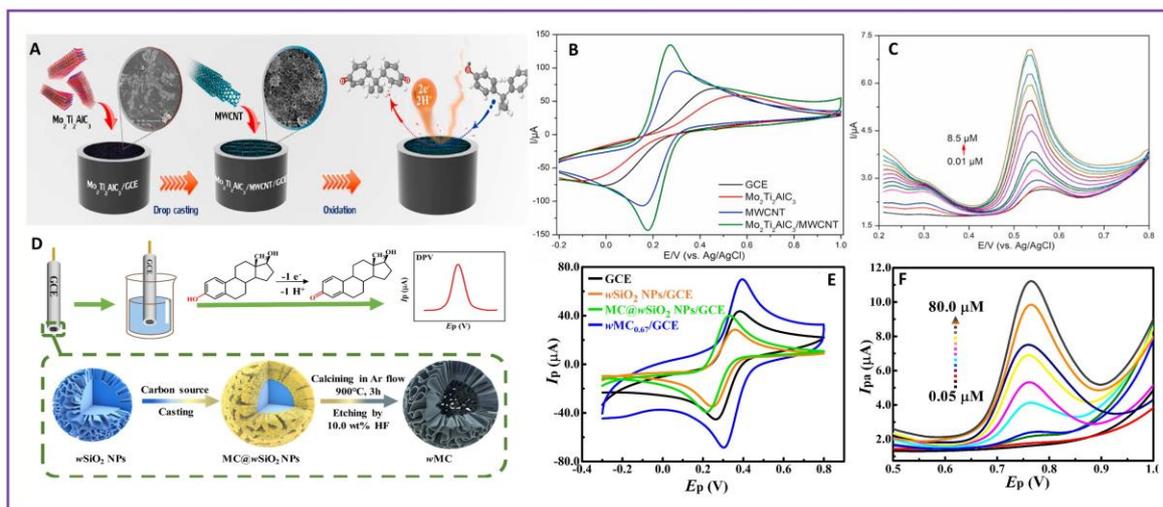


Figure 4. Scheme of fabrication of (A) Mo₂Ti₂AlC₃/MWCNT/GCE(D) wMC/GCE; Comparison of CVs of different electrodes for detection of (B) BPA, (E) 17 β -E2; DPVs of (C) Mo₂Ti₂AlC₃/MWCNT/GCE at different concentrations of BPA(F) wMC/GCE at different concentrations of 17 β -E2.

3.5. Electrochemical sensors for detection of Newly Registered Pesticides (NRPs)

Pesticides are a group of chemicals and microorganisms for the eradication of insects, fungi, weeds, and bacteria [110]. Different classes of pesticides include organophosphates, organochlorines, carbamates, pyrethrin, neonicotinoids, sulfonylureas, and triazines. Newly registered pesticides are new pesticide chemicals or different use of the existing chemicals as pesticide. In order to cater with the demands of food production worldwide, the agriculture sector is largely dependent on pesticides. Even though pesticides increase the crop, vegetable, and fruit yields to several folds, the havoc they cause is matter of serious concern. Soil and water contamination, bioaccumulation, carcinogenic effects, damage to immune system, reproductive system, nervous system, and respiratory system are the dangerous impacts of the use of pesticides [111].

Neonicotinoids is a group of pesticides which are highly effective against insects. Imidacloprid (IDP), thiamethoxam (TMX), and dinotefuran (DNF) are common neonicotinoid pesticides. IDP[1-(6-chloro-3-pyridymethyl)-N-nitroimidazolidin-2-ylideneamine] is a neonicotinoid pesticide. IDP binds irreversibly to the nicotinic acetylcholine receptors which are responsible for the functioning of central nervous system. This hinders the neural transmission, eventually leading to the paralysis and then the death of the insect [112]. IDP may also cause liver failure and affect neural tissue development in infants and cancer [113]. An electrochemical sensor for the detection of IDP using Ag nanoparticles deposited on mesoporous carbon and naturally extracted hematite ore (Ag@Meso-C/Hematite Ore) nanocomposite resulted in a detection limit of 0.257 μ M [114]. An organic/inorganic composite of f-MWCNT/EDTA integrated electrochemical sensor was used for the detection of IDP [115]. This electrochemical sensor provided a detection limit of 3.1×10^{-3} pM. Tungsten sulfide (WS₂) nanosheets were fabricated for the detection of IDP in water and soil samples [116]. Fast electron transfer on WS₂ nanosheets resulted in electrochemical reduction of the aromatic nitro group in IDP at a detection limit of 0.28 μ M and linear range of 10-90 μ M. A hydrothermally synthesized Fe-rich FeCoNi-MOF in-situ modified nickel foam working electrode was utilized for sensing IDP [117] as shown in Figure 5(A). An optimized ratio of Fe:Co:Ni (5:1:1) exhibited a better electrocatalytic activity for detection of IDP in Figure 5 (B). The exposed rich active sites in Fe-rich FeCoNi-MOF resulted in

a low detection limit of 0.04 pmol/L and linear range of $1\text{--}1.2\times 10^8$ pmol/L using DPV technique as shown in Figure 5(C).

TMX[(3-[(2-chloro-5-thiazolyl)methyl]tetrahydro-5-methyl-N-nitro-4H-1,3,5-oxadiazine-4-imine)] is a neonicotinoid pesticide. The food safety authority in European countries has restricted the range of TMX in agronomy products as 0.02 mg/kg to 5.0 mg/kg [118]. A composite of hydrothermally synthesized cobalt oxide in graphitic carbon nitride ($\text{Co}_3\text{O}_4@\text{g-C}_3\text{N}_4\text{NC}$) was used for sensing TMX [119] in Figure 5(D). Due to the large electroactive surface area and faster electron transfer offered by the composite, $\text{Co}_3\text{O}_4@\text{g-C}_3\text{N}_4/\text{SPCE}$ showed better electrocatalytic performance compared to $\text{Co}_3\text{O}_4/\text{SPCE}$, $\text{g-C}_3\text{N}_4/\text{SPCE}$, and bare SPCE as shown by CV analysis in Figure 5(E). DPV techniques showed a detection limit of 4.9 nM and linear range of 0.01–420 μM in Figure 5(F). $\text{Fe}_2\text{O}_3@\text{g-C}_3\text{N}_4@\text{Schiff}$ base modified GCE synthesized by a calcination method was used for sensing TMX [120]. $\text{Fe}_2\text{O}_3@\text{g-C}_3\text{N}_4@\text{MSB}/\text{GCE}$ provided a detection limit of 0.137 μM and linear range of 0.01–200 μM for sensing TMX in real samples, like potato, rice, and river water. Another electrochemical sensor was fabricated with three-dimensional nitrogen doped macro-meso-microporous carbon composite derived from nitrogen doped Cu-MOF using PVP(N/Cu-HPC) for simultaneous detection of neonicotinoids, such as IDP, TMX, and DNF [121]. Mass and charge transport between neonicotinoid analyte molecules and Cu nanoparticles and carbon atoms improved the electrocatalytic performance of N/Cu-HPC/GCE towards detection of IDP, TMX, and DNF. N/Cu-HPC/GCE provided a detection limit of 0.026 μM for IDP, 0.062 μM for TMX, and 0.01 μM for DNF and linear ranges of 0.5–60 μM for both IDP and DNF, 1–60 μM for TMX. Another electrochemical sensor using MOF-derived N-doped octahedral NiCu nanoporous carbon composite modified GCE (N/NiCu@C/GCE) was developed for the detection of neonicotinoids IDP, TMX, and DNF [122]. The porous carbon structure and NiCu nanoalloy improved the diffusion between electrolyte and active site enhancing the electrocatalytic performance of N/NiCu@C/GCE. Using DPV technique, detection limit and linear ranges for sensing IDP, TMX, and DNF were found out to be 0.017, 0.007, and 0.001 μM and 0.5–60, 1–60, and 0.5–60 μM , respectively.

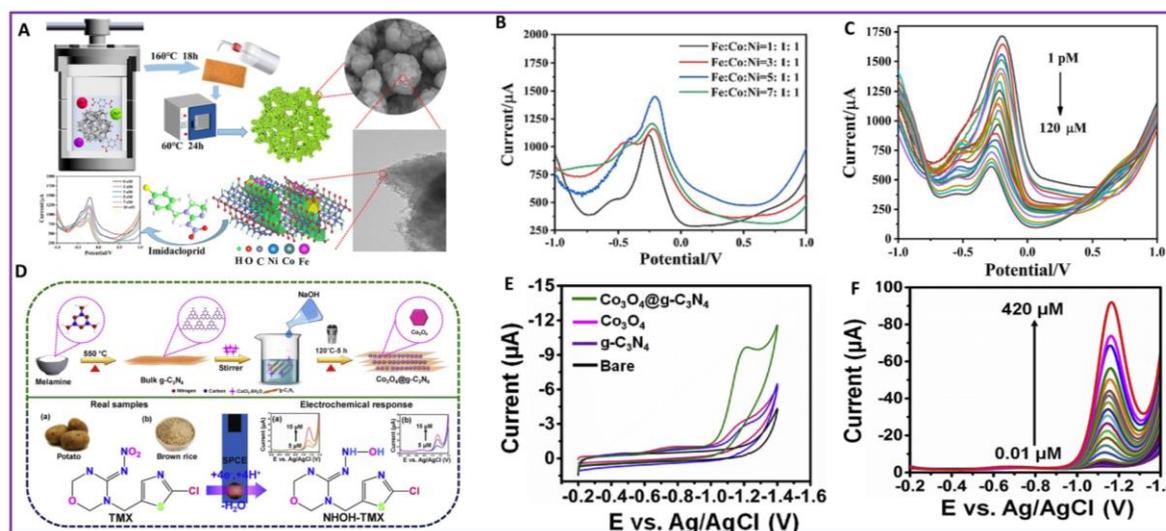


Figure 5. Scheme of fabrication of (A) FeCoNi-MOFs (D) $\text{Co}_3\text{O}_4@\text{g-C}_3\text{N}_4\text{NC}$; (B) Comparison of DPV of different electrodes of FeCoNi-MOFs for detection of IDP; (C) DPV curve of Nickel foam/Fe-rich FeCoNi-MOF at different concentrations of IDP; (E) CVs of different electrodes for detection of TMX; (F) $\text{Co}_3\text{O}_4@\text{g-C}_3\text{N}_4/\text{SPCE}$ at different concentrations of TMX.

3.6. Electrochemical sensors for detection of disinfection by-products (DBPs)

Drinking water disinfection to ensure safe drinking water was one of the greatest achievements of the past century. However,, the by-products of the process are highly toxic. Disinfection by-products (DBPs) are formed when disinfectants like chlorine, ozone, chlorine dioxide, or chloramines, react with naturally occurring organic matter, man-made contaminants, bromide, and iodide during

drinking water production [123]. DBPs are associated with genotoxicity and carcinogenicity [124]. Chlorates, chlorites, bromates, trihalomethanes (THMs) and haloacetic acids (HAAs) are common DBPs found in drinking water.

Chlorine and its oxides are the most common and cost-effective option for drinking water disinfection. Use of ClO_2^- may cause acquired methemoglobinemia, which has a life-threatening potential by reducing the oxygen capacity of haemoglobin [125]. WHO and EPA regulated the concentration of ClO_2^- should not exceed above 0.2 mg/L [126]. Fe_3O_4 nanoparticles synthesized by surfactant assisted hydrolysis of optimized amounts of Fe^{2+} and Fe^{3+} ions modified on CPE was used for electrochemical sensing of ClO_2^- [127]. The sensor could detect ClO_2^- ions in aqueous medium. The practical applicability of the sensor was tested in tap water and bottle water samples. The sensor showed a detection limit of 8.6 nM. Another electrochemical sensor with carbon black modified SPCE was used as the working electrode (CB-SPCE) for detection of ClO_2^- [128]. The sensor could detect ClO_2^- at a limit of detection of 0.01 ppm and linear range of 0.05-20 ppm. The sensor was found to be effective in sensing ClO_2^- in swimming pool water samples.

Bromate (BrO_3^-) is also a by-product of ozonation and chlorination disinfection process. Potassium bromate (KBrO_3) is used as an oxidizing agent in baking industry. Bromate is classified as a B2 carcinogen. USEPA and WHO has recommended 10 $\mu\text{g/L}$ as the maximum acceptable concentration of BrO_3^- in drinking water [129]. For the detection of BrO_3^- , a nanocomposite of electrochemically polymerized poly(aniline-co-o-aminophenol), ERGO, and Pd (PANOA/ERGO/Pd) with a caterpillar like structure modified GCE was integrated [130]. The sensor produced a detection limit of 1 μM and linear range of 4-840 μM for BrO_3^- detection. The sensor performed well in drinking water and river water samples. A lamellar MXene, $\text{Ti}_3\text{C}_2\text{T}_x$ modified GCE was fabricated for BrO_3^- in drinking water samples [131]. Figure 6(A) shows the fabrication of the working electrode by GCE and the electrochemical detection of BrO_3^- . The electrochemical activity of MXene for BrO_3^- detection was confirmed with comparison of CV of bare GCE, GCE/Graphene, and GCE/MXene as shown in Figure 6(B). MXene as a sensing platform electrocatalytically augmented the reduction of BrO_3^- , which resulted in a detection limit of 41 nM and linear range of 50 nM – 5 μM . The detection limit and linear ranges for BrO_3^- detection was obtained from the DPV curve of increasing concentration of BrO_3^- as given in Figure 6(C). The practical applicability of the MXene sensor was also tested in domestic tap water.

Trichloroacetic acid (TCAA) is a kind of haloacetic acid (HAA). TCAA is associated with carcinogenicity, reproductive defects, and mutagenicity [132]. Combining the redox activity of iron phthalocyanine (PcFe) and high surface area and absorbability of ZIF-8, an efficient electrochemical sensor (PcFe@ZIF-8) for TCAA sensing was developed [133]. The sensor exhibited a detection limit of 1.89 nM. The sensor also showed good performance in real samples, such as tap water and swimming pool water samples. Haloacetamides (HAM) is a class of DBPs. Although the concentration of HAM is lower than THMs and HAAs, their cytotoxic and genotoxic effects are double fold [134]. Trichloroacetamide (TCAM), a HAM, was electrochemically detected with a heterostructure of triangular Ag nanoprism@ MoS_2 nanosheet ($\text{AgNPR@MoS}_2/\text{GCE}$) [135]. Schematics of electrochemical detection of TCAM on $\text{AgNPR@MoS}_2/\text{GCE}$ is given in Figure 6(D). The supremacy of $\text{AgNPR@MoS}_2/\text{GCE}$ for electrochemical detection of TCAM can be clearly seen from SWV curves of bare GCE, AgNPR/GCE , MoS_2/GCE , and $\text{AgNPR@MoS}_2/\text{GCE}$. The reduction peak current was better compared to other electrodes as shown in Figure 6(E). Quantitative detection of TCAM was done using DPV technique. With increase in concentration of the analyte TCAM added, the peak current value also increased as shown in Figure 6(F). Due to dechlorination reaction of TCAM catalyzed by AgNPR and accelerated by the H^+ absorption by S atoms in MoS_2 nanosheets, the sensor could provide a detection limit of 0.17 μM and linear ranges of 0.5 - 10 μM and 10 - 80 μM . The applicability of the sensor in TCAM detection in drinking water samples was also demonstrated. Table 2 summarizes the comparison of merits of electrochemical sensors for detection of different classes of CECs as discussed in the review.

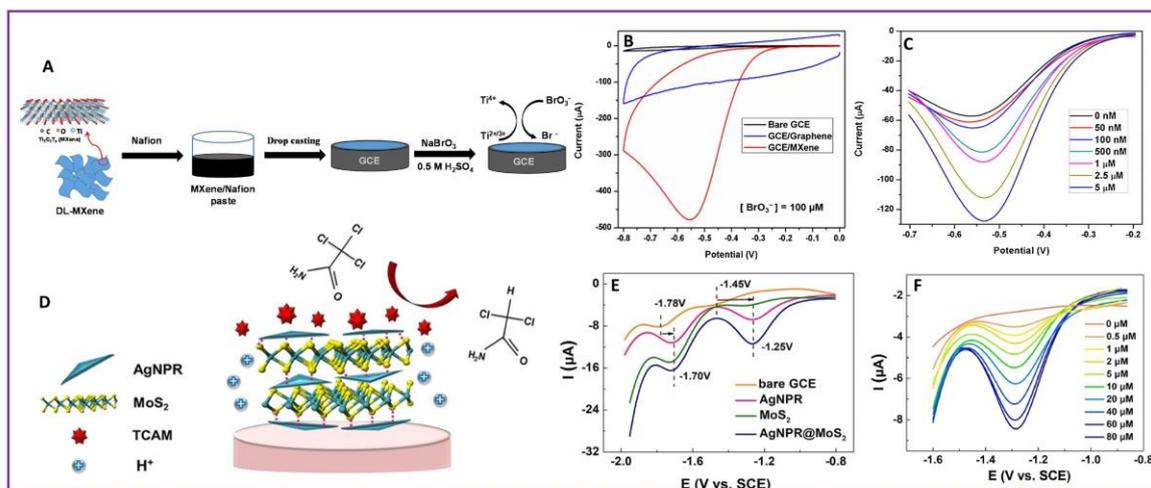


Figure 6. Scheme of electrochemical detection of (A) BrO₃⁻ at Ti₃C₂T_x/GCE (D) TCAM at AgNPR@MoS₂/GCE; Comparison of (B) CVs of different electrodes for detection of BrO₃⁻ (E) SWVs of different electrodes for detection of TCAM; (C) DPV of Ti₃C₂T_x/GCE at different concentrations of BrO₃⁻; (F) SWV of AgNPR@MoS₂/GCE at different concentrations of TCAM.

Table 2. Comparison of merit of electrochemical sensors of different classes of CECs.

Sl.No	CEC class	Analyte	Modified Electrode	LOD	Linear Range	Ref.
1.	Pharmaceuticals	PA	VFe ₂ O ₄ /GCE	8.20 nM	0.05 – 12.3 μM	44
2.	Pharmaceuticals	PA	COOH-CNTs/ZnO/NH ₂ -CNTs/GCE	46.8 fM	25pM-0.5 μM	45
		DIC		78 fM	75pM-1.5 μM	
		ORP		60 fM	75pM-1.5 μM	
3.	Pharmaceuticals	DIC	EGr-C _{0.12} Fe _{1.8} O ₄ /SPCE	1nM	0.01–23.1 μM	46
4.	Pharmaceuticals	DS	oxidized g-C ₃ N ₄ /Cu-Al LDH/GCE	0.38 μM	0.5–60 μM	47
5.	Pharmaceuticals	IBU	Cu ₃ TeO ₆ /GCE	0.017μM	0.02–5 μM and 9–246 μM	48
6.	Pharmaceuticals	NZ	Sg-C ₃ N ₄ /CuWO ₄	3 nM	0.005 μM-877 μM	49
7.	Pharmaceuticals	NFX	CaCuSi ₄ O ₁₀ /GCE	0.0046 μM	0.01–0.55 and 0.55–82.1 μM	50
8.	Pharmaceuticals	NAP SUM	ZnO/NiO/Fe ₃ O ₄ /MWCNTs	3 nM	4.00 nM to 350.00 μM	51
				2 nM	6.00 nM to 380.00 μM	
9.	Pharmaceuticals	SUM NAP	P- L CuO: Tb ³⁺ NS/CPE	3.3 nM 2.7 nM	0.01-800 μM 0.01–700 μM	52
10.	Pharmaceuticals	VOR	AuNPs@GRP/GCE	50 nM	0.1–1.0 and 1.0–6.0 μM	54
11.	Pharmaceuticals	PMHC	BaWO ₄ /f-CB/SPCE	29 nM	0.03–234.74 μM and 274.73–1314.73 μM	55
12.	Pharmaceuticals	CBZ	GdVO ₄ /f-CNF/GCE	0.0018 μM	0.01–157 μM	56
13.	Illicit Drugs	Cocaine	Oh-Pd ²⁺ : Co ₃ O ₄ -C/GCE	1.3 nM	0.01 μM–900.0 μM	63
14.	Illicit Drugs	Morphine	PDA-f-MWCNT/GCE	0.06 μM	0.075–75.0 μM	65
15.	Illicit Drugs	Morphine	CNH-CHI@PtNPs/GCE	0.02 μM	0.05-25.4μM	68

		MDMA		0.018 μM		
16.	Illicit Drugs	MDEA	C-SPE	0.03 μM	2.5 to 30.0 μM	69
17.	Illicit Drugs	OXY COD	CoFe ₂ O ₄ /C-SPE	0.050 μM 0.02 μM	0.06–38 μM	71
18.	Illicit Drugs	CNZ	CoOOH/r-GO/SPCE	38 nM	0-350 μM	73
19.	Illicit Drugs	Cocaine	SDS-SPE	0.7 μM	1-30 μM	74
		heroin		1.8 μM	2.5–30 μM	
		MDMA		0.9 μM	1–30 μM	
		CI-PVP		1.6 μM	2.5–30 μM	
		ketamine		1.1 μM	2.5–30 μM	
20.	PCPs	Methyl paraben	AuNPs@GO/PGE	2.02 μM	0.030-1 mM	78
21.	PCPs	Methyl paraben	GCE/ZA 8	7.25 μM	0.02–0.12 mM	79
22.	PCPs	HQ	N-NiCS/GCE	0.0015 μM	0.005-100 μM	81
		CC		0.015 μM	0.05-200 μM	
		RS		0.24 μM	5-500 μM	
23.	PCPs	TBHQ	MnO ₂ /ERGO/GCE	0.8 μM	1-50 μM and 100-300 μM	82
24.	PCPs	TBHQ	VMSF/ErGO/GCE	0.23 nM	0.001–0.5 and 0.5–120 μM	83
25.	PCPs	TBHQ	ZnO/ZnNi ₂ O ₄ @porous carbon@COF _{TM}	15.95 nM	47.85 nM- 130 μM	84
26.	PCPs	BP3	SPE	1.9 μM	6–200 μM	86
		OC		4.1 μM	11–300 μM	
27.	PCPs	TCS	rGO/Cu-BTCMOF/NiCo/GCE	0.23pM	0.39 pM-49 μM	89
28.	PCPs	Vanillin	MnO ₂ NWs-rGO/GCE	6nM	0.01–20 μM and 20–100 μM	90
29.	EDCs	BPA	Mo ₂ Ti ₂ AlC ₃ /MWCNT/GCE	2.7nM	0.01–8.50 μM	94
30.	EDCs	BPA	Ti ₃ C ₂ T _x /V ₂ O ₅ /GCE	87nM	414 nM-31.2 μM	95
31.	EDCs	BPA	CTpPa-2/GCE	0.02 μM	0.1-50 μM	96
		BPS		0.09 μM	0.5-50 μM	
32.	EDCs	BPA DM-BPA	Pt@SWCNTs-Ti ₃ C ₂ -rGO/SPCE	2.8 nM 3 nM	0.006-7.4 μM	97
33.	EDCs	TBBPA	CNTs@ZIF-67/PFDA/AB	4.23 nM	0.01–1.5 μM	98
34.	EDCs	BPF	ZnO/G/CTAB/MPCE	0.06 μM	0.5-10 μM	100
35.	EDCs	BPSIP	g-C ₃ N ₄ /Cu-DTO MOF/GCE	0.02 μM	0.04–1.10 μM	102
36.	EDCs	BPSIP	g-C ₃ N ₄ @GN/SPCE	0.02 \pm 0.01 μM	1–100 μM and 100–1000 μM	103
37.	EDCs	OPP	E-Mn ₂ O ₃ @GO/GCE	0.63 nM	0.002-20 μM	104
		BP		0.88 nM	0.003-24 μM	
38.	EDCs	EE2	CB/Pd NPs/GCE	81 nM	0.5-119 μM	106
39.	EDCs	17 β -E2	wMC _{0.67} /GCE	8.3 nM	0.05-10 and 10- 80 μM	108
40.	EDCs	DES	Fe ₃ O ₄ -NC/GCE	4.6 nM	0.01-12 μM	109
		17 β -E2		4.9 nM	0.01-20 μM	
41.	NRPs	IDP	Ag@Meso-C/Hematite Ore/GCE	0.257 μM	10.80–195.50 μM	114

42.	NRPs	IDP	f-MWCNT/EDTA/SPCE	3.1×10^{-3} pM	0.001–0.05 nM, 0.001–0.04 μ M 0.001 nM–0.04 mM	115
43.	NRPs	IDP	WS ₂ /GCE	0.28 μ M	10–90 μ M	116
44.	NRPs	IDP	Nickel foam/Fe-rich FeCoNi-MOF	0.04 pM	1–1.2 $\times 10^8$ pM	117
45.	NRPs	TMX	Co ₃ O ₄ @g-C ₃ N ₄ /SPCE	0.0049 μ M	0.1–420 μ M	119
46.	NRPs	TMX	Fe ₂ O ₃ @g-C ₃ N ₄ /MSB/GCE	0.137 μ M	0.01–200 μ M	120
47.	NRPs	IDP	N/Cu–HPC/GCE	0.026 μ M	0.5–60 μ M	121
		TMX		0.062 μ M	1–60 μ M	
48.	NRPs	DNF	N/NiCu@C/GCE	0.01 μ M	0.5–60 μ M	122
		IDP		0.017 μ M	0.5–60 μ M	
		TMX		0.007 μ M	1–60 μ M	
49.	DBPs	ClO ₂ ⁻	Fe ₃ O ₄ /CPE	8.6 nM	1–10 μ M, 20–	127
					100 μ M	
50.	DBPs	ClO ₂ ⁻	CB-SPCE	0.01 ppm	0.05–20 ppm	128
51.	DBPs	BrO ₃ ⁻	PANOA/ERGO/Pd/GCE	1 μ M	4–840 μ M	130
52.	DBPs	BrO ₃ ⁻	Ti ₃ C ₂ T _x /GCE	41 nM	50 nM – 5 μ M	131
53.	DBPs	TCAA	PcFe@ZIF-8/GCE	1.89 nM	0.02 - 1 μ M	133
54.	DBPs	TCAM	AgNPR@MoS ₂ /GCE	0.17 μ M	0.5- 10 μ M and 10- 80 μ M.	135

5. Conclusions and Future Perspectives

CECs are so toxic that presence of very minute quantities can have adverse effects on the health of living beings. Since the wastewater treatment fails to remove CECs, they continue to exist in drinking water, enter the body of living organisms, bioaccumulate and is hazardous. Electrochemical sensors help in monitoring these CECs in real samples both qualitatively and quantitatively and thus ensure safety in food and drinking water. Therefore, a number of electrochemical sensors have been developed for CEC detection in real samples. Electrochemical sensors are developed for detection of pharmaceuticals, PCPs, illicit drugs, EDCs, pesticides, and DBPs.

Already known CECs should be detected and if found above the regulatory limits of international agencies, like WHO and EPA, proper action must be taken to prevent their noxious effects. More efficient electrochemical sensors must be developed for the simultaneous detection of CECs which can be present together because combinations of some CECs can be more toxic than their individual presence. Such combinations must be monitored with a single electrochemical sensor. Also, there is a number of CECs for which there are no reports of electrochemical sensors available, which needs to be properly identified. Sensors should be developed for the sensitive and selective detection of those CECs. Although the number of chemicals classified under CEC is increasing, there are a number of chemicals which are still left unidentified as CECs as their harmful effects are unknown. Such toxic chemicals need to be screened and their use needs to be regulated.

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