Voltammetric Techniques in Assessing the Food Quality

Subjects: Biochemical Research Methods

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Generally, the detection of analytes or molecules present in food materials interferes with the chromophore moieties in the food. Some of the common problems with the use of conventional methods in determining food quality are low sensitivity to redox changes, turbidity, low spectrum resolution, and scattering issues related to the sample. Moreover, the miniaturization and portability of detectors are the biggest disadvantages of conventional methods. Therefore, there is a huge demand for quick, robust, selective, and easy methods, such as voltammetric methods, for determining the food's quality. They exhibit a higher level of selectivity for the redox reactions, and a faster response. They are very simple, economical, and their portability with unlimited miniaturization has made them an ideal and popular choice for assessing the food quality compared with other analytical methods.

Keywords: cyclic voltammetry ; differential pulse voltammetry ; electrochemical sensors ; food safety

1. Carbon Nanotube (CNT)-Based Nanocomposite for Assessing the Food Quality

S. Ijima, a Japanese scientist, discovered carbon nanotubes (CNTs) in 1991 ^[1]. Carbon nanotubes are cylindrical tube molecules rolled into a single sheet of graphene. The CNTs can be found as single-walled (SWCNT) with an approximate diameter of 1 nm and sometimes as multiwalled (MWCNT), whose diameter ranges more than 100 nm, whereas their length measures several micrometers to millimeters. They exhibit excellent porosity, a high surface area, and easy surface modification, and some of them act as conductors (armchair-shaped nanotubes) and semiconductors (zigzag-shaped nanotubes) as well.

MWCNTs exhibit good conductivity all the time, and their conductivity can be compared to that of metals ^[2]. On the other hand, the conductivity of SWCNTs mainly depends on their chiral vector; due to this, they can show electrical conductivity similar to that of metals, and sometimes behave similar to semiconductors or nonconductors. For instance, a small change in the pitch of the helicity can transform the conductivity of CNT from a conductor to a large-gap semiconductor. These unique electrical properties have made the CNTs one of the best, and the most popular choices for fabricating electrodes to assess food quality [3]. The key factors of electrodes, such as their efficiency, repeatability, and sensitivity, depend on multiple features, such as surface modification, the diameter and length of the electrode, and the number of layers present in CNTs. Due to the wide range of excellent properties demonstrated by CNTs; they are the future of electrochemical sensors. They also exhibit significant electrochemical reactivity for a large number of biomolecules and possess the ability to increase electron transfer processes [4]. Therefore, CNTs are excellent carbon materials and can be potentially used either completely as electrodes or in small amounts as modifiers in graphite powder to determine food chemicals ^[5]. The CNT-based electrochemical sensors are highly sensitive, robust, and show remarkable surface conductivity. The CNTs are sometimes added to various metal nanoparticles that help in the formation of additional sites for the electrocatalytic process, increase sensitivity, and lower the detection limits of the electrodes. Recent electrochemical research is progressively marching towards integrating the conventional biological concepts with digitalization by using instruments (electrochemical sensors) to establish simple, handheld systems that completely depend on specific electrochemical reactions of bioactive compounds and result in electrical, thermal, or optical signals to detect them easily.

Rahemi et al. fabricated MWCNT glassy carbon electrode (GCE) modified by β -cyclodextrin and a polyaniline film to detect the chlorophenoxy herbicide MCPA ^[6]. This herbicide readily gets absorbed by the leaves and roots of plants; consuming it results in irritation of the skin, serious damage to the eyes, and it also causes drooling, twitching, low blood pressure, unconsciousness, jerking, and spasms. They reported that the fabricated electrode showed excellent electrocatalytic oxidation of the herbicide MCPA with significant sensitivity, repeatability, and stability. They also reported

the possible use of the fabricated electrode to detect the presence of MCPA herbicide in real samples, such as natural waters. The use of CV for the present investigation is more advantageous than the sophistically established high-performance liquid chromatography technique. The CV method does not require the previous step's extraction, cleaning, or derivatization in the range of 10–100 μ mol L⁻¹. They also reported the obtained detection limit of 0.99 μ mol L⁻¹ in water [<u>fi</u>].

Tyrosine produces tyramine during the decarboxylation reaction, which is generally present in chocolate, wine, banana, fish, beer and so on ^[Z]. Excess consumption of foods containing tyramine can lead to tachycardia, vomiting, strong migraine headaches, rash, hypertonia, palpitation, flushing, rash, and so on ^[B]. There are reports on the electrochemical determination of tyramine using a MWCNT-modified graphite electrode by cyclic voltammetry ^[B].

The fabricated sensor electrode demonstrated greater sensitivity and repeatability. Kochana et al. ^[9] claimed that their fabricated electrode displayed high sensitivity, specificity, and selectivity for the determination of tyramine in the samples of food products.

2. Graphene and Related Nanocomposites to Determine the Food Quality

Canadian theoretical physicist P. R. Wallace first discovered graphene in the year 1947, and A. Geim and K. Novoselov were further investigated [10][11][12]. Graphene is considered a super material because of its dynamic mechanical rigidity and thermal stability, but apart from that, it also exhibits unique electrical properties. This extremely unique electrical property of graphene has attracted most electrochemists and researchers to carry out their research on it. The electrical properties depicted by graphene are significantly different from the electrical properties exhibited by other carbon materials, such as CNTs, fullerenes, graphite and so on [13][14]. The unique physical, chemical, mechanical, and thermal properties of graphene, combined with its excellent light transparency of 97.7%, make it a potential material for the construction of electrodes. Generally, the charge carrier mobility of graphene is found to be approximately 15,000–20,000 cm² V s⁻¹ at room temperature. All these properties have made graphene a potential electrode candidate for assessing food quality. Bolotin has reported that the electron's mobility in graphene layers is at least 100 times greater than that of silicon ^[15].

Nowadays, graphene is one of the most applied sensor materials in food safety assessment because of its high reactive sites, specific surface area, and charge carrier mobility ^[16]. The two important types of graphene, reduced graphene oxides (rGO) and graphene oxides (GO), are well reported by Plachá et al. ^[17] and exhibit slightly different properties. GO is more economical than graphene and requires a very short time for synthesis. Hummer's process is one of the commonly used methods of preparing GO which uses potassium permanganate and concentrated H_2SO_4 as oxidizing agents ^[18]. GO is acidic because of its hydrophilic nature. The GO sheets can be reused even after dissolving in water. One of the important characteristics of GO is the possibility of converting GO to graphene by different thermal reduction processes ^[19]. On the other hand, rGO can be synthesized by reducing the GO by laser radiation, electrochemical, or thermal reduction techniques. Preparation of single-layered graphene in solvents is always a tough job and needs extra precautions and experience. Various properties of graphene materials were well reported by Qureshi et al. ^[20].

The surface of graphene oxide can be modified with carboxyl and carbonyl groups by linking the carbon atoms present in GO with either hydroxy or epoxy groups $\frac{[21]}{2}$. Thus, the electrical conductivity increases, followed by the enhanced surface area of the GO. Therefore, GO can be employed as a modifier in the electrode or as a whole electrode to detect different toxic chemicals and food pathogens in foods $\frac{[21]}{2}$.

The trace of phoxim's presence in food was detected using an electrode developed by Wu et al. using poly(3-methyl thiophene)/nitrogen-doped graphene material for the construction of the electrode ^[22]. Phoxim is a type of pesticide that comes under organophosphate pesticides. As it is discussed earlier, these types of synthetic pesticides are used to kill insects and mammals and seriously affect the acetylcholinesterase neurotransmitter enzyme. The authors have optimized the cyclic voltammetric conditions and reported that the current varied linearly over two linear ranges (0.02–0.2 μ M and 0.2–2.0 μ M) concerning the concentration of phoxim. They obtained a low detection limit of 6.4 nM ^[22].

Yun et al. fabricated electrochemically rGO [ERGO] grafted with 5-amino-1,3,4-thiadiazole-2-thiol-Pt [ATDT-Pt] nanoparticles for GCE to detect orange II ^[23] dye. Orange II is a type of azo dye mostly used in organic LEDs, inks, textiles, hair dyes, shoe polishes, and foodstuffs ^[23].

Yun et al. ^[23] have detected orange II in 0.1 M acetate buffer solution of pH 4.5 with significant reversible redox peaks. The authors have reported a wide linear range of 1×10^{-8} - 6×10^{-7} M and a low detection limit of 3.4×10^{-10} M (s/n = 3)

for orange II dye detection. The fabricated electrochemical sensor exhibited good sensitivity, robustness, and selectivity for the real samples with significant recovery.

Manjunath et al. reported the catechol determination present in the food by fabricating the voltammetry sensor using poly (adenine) modified graphene ^[24]. Catechol is one of the toxic organic compounds that can be produced synthetically and used as a precursor to pesticides, flavors, and fragrances ^[24]. Excessive usage of catechol causes depression in the central nervous system and can raise blood pressure, sometimes it absorbs through the skin, and causes an illness resembling that induced by phenol, except the convulsions are more pronounced. During the catechol determination with the above voltammetry sensor, there was a linear increase in the oxidation peak current of catechol with its concentration in the range of 2×10^{-6} –8 $\times 10^{-6}$ M and 1×10^{-5} –1.5 $\times 10^{-4}$ M with a 2.4 $\times 10^{-7}$ M detection limit. The authors used a real water sample to determine the catechol using the developed sensor.

Shi et al. developed a graphene and thionine nanocomposite electrode to detect fumonisin B1 ^[25]. Fumonisin B1 is a cardiotoxic chemical generally found in Fusarium verticillioides cultures and naturally contaminated foods ^[26]. The intake of fumonisin B1 can cause equine leukoencephalomalacia and pulmonary edema in pigs. The surface of the graphene sheet was loaded with a huge number of thionine molecules to enhance its electrical conductivity, surface area, and thus increase the electrochemical signal of the electrode. They reported that the resultant decreased current is proportional to the concentration of fumonisin. They claimed that the fabricated electrochemical sensor exhibits greater sensitivity and selectivity.

Dalkıran et al. constructed a Xanthine electrochemical sensor modified with graphene/cobalt oxide nanoparticles/chitosan composite to determine the freshness of fish ^[27].

Ma and Chen ^[28] fabricated a diethylstilboestrol (DES) sensor using a graphene doped gold nanoparticle-modified electrode. The DES is a veterinary medicine used for the treatment of estrogen-deficiency disorders and is also used as a growth stimulant in animals ^[29]. The consumption of even the smallest residual traces of DES in meat can cause carcinogenic disorders in humans ^[28]. The authors reported that the fabricated electrode displayed high sensitivity and specificity in determining the DES even in the presence of interfering ions, such as estradiol, estriol, estrone, and folic acid.

Kartika et al. constructed silver nanoparticle/graphene nanoplatelets on modified screen-printed carbon electrodes for determining rhodamine B (RhB) dye in food products ^[30].

3. Carbon Dots (CDs) and Their Nanocomposites as Sensors for Assessing the Food Quality

Carbon dots (CDs) are the new grades of the carbon family and have attracted many researchers in the past few years. They are considered quasi-zero-dimensional carbon materials consisting of small carbon atoms with a size of less than 10 nm. They exhibit low toxicity, excellent quantum yield, good photoluminescence, extremely refined size, eco-friendliness, are less expensive, and are easily synthesized. One of the remarkable features of CDs is that their physicochemical properties can be easily regulated using surface passivation and functionalization ^[31]; thereby allowing the application of CDs in the fabrication of electrocatalysis, chemical, biosensing, and optoelectronic devices. Xu et al. in 2004, accidentally obtained carbon nanoparticles with fluorescence during the purification of SWCNTs ^[32]. There are mainly three types of CDs available and they are carbonized polymer dots, graphene quantum dots, and carbon quantum dots. This classification is based on their formation mechanisms, micro-/nanostructures, and properties ^[32].

Hou et al. constructed a graphene quantum dots/gold NPs modified GCE for the effective determination of malachite green dyes ^[33]. These dyes are mainly used as colorants in industry and as antimicrobial agents due to their low cost and availability. However, malachite green is a highly toxic dye, and its excessive intake can cause serious health issues, such as carcinogenesis, mutagenesis, chromosomal fractures, teratogenicity, and respiratory toxicity. Using the fabricated graphene quantum dot electrode, the authors have obtained a pair of quasi-reversible adsorption-controlled redox peaks, respectively, at 0.502 V (Epa) and 0.446 V (Epc) using a 0.05 mol L⁻¹ H₂SO₄ solution ^[33]. The fabricated electrode was used for the detection of malachite green in fish samples, and the authors reported that the obtained results are satisfactory, reproducible, and stable with a 96.25–98.00% recovery rate. Costas-Mora et al. ^[34] electrochemically determined the methylmercury using carbon dot nanoprobe. Methylmercury is a very toxic neurotoxin discharged into the human body after consuming fish and affects the brain and nervous systems of particularly pregnant women and infants ^[35]. Authors claimed to fabricate a highly sensitive and robust methylmercury electrochemical sensor, which can detect the analytes in less than 1 minute even at very low concentrations with the detection limit of 5.9 nM.

Dong et al. ^[36] developed the amantadine electrochemical sensor using carbon dots. Amantadine is basically not a veterinary drug but was used largely in the poultry industry before it was recently banned in many countries. This antiviral drug can cause nausea and dizziness in humans if there is an excess amount of intake. The fabricated electrode was successful in detecting even the traces of amantadine residues in chicken, with a limit of detection of 0.02 ng mL⁻¹. Xiang et al. fabricated graphene quantum dots electrodes to detect hepatitis B virus (HBV) DNA ^[37]. HBV is a very harmful human pathogen that can infect easily and cause liver inflammation, cirrhosis, and liver cancer. According to the authors, the fabricated electrochemical sensor exhibits high sensitivity with a detection limit of 1 nM, and a linear detection range from 10 to 500 nM. However, they reported that the developed sensor could be a potential candidate for detecting other probe DNA, due to the strong interaction between single-stranded DNA and graphene quantum dots ^[37].

Song et al. developed carbon dots that reduced gold NPs for the determination of ractopamine in pork meat ^[38]. It is an animal feed additive used to increase leanness and food conversion efficiency in farmed animals, but it can cause down syndrome and severe cardiovascular stress in humans after consuming meat containing ractopamine.

4. Ordered Mesoporous Carbon (OMC)-Based Nanocomposites for Food Assessment

The porous carbons are the new addition to the family of carbon materials, showing greater porosity with maximum surface area and energy ^[11]. Due to their excellent chemical inertness, electrical conductivity, high mechanical strength, and ordered and regular structure, this new grade of carbon material is quite famous among researchers and therefore uses electrode materials ^[39]. The classification, characteristics, and applications of OMC were reported in detail by Shashanka in their previous publication ^[40]. Libbrecht et al. ^[41] reported the 2D hexagonal OMC structure in their previous article.

Kochana et al. fabricated mesoporous carbon electrochemical sensors to detect tyramine in food products ^[9]. Their consumption can cause strong migraine headaches, vomiting, tachycardia, rash, hypertonia, palpitation, flushing, rash and so on. It has been reported that the developed mesoporous carbon electrochemical sensor electrocatalytically oxidized tyramine with a linear range from 6 to 130 μ M, and a detection limit of 1.5 μ M, respectively. They also reported the high biological affinity of the fabricated sensor against tyramine due to the low Michaelis–Menten constant ^[42] (66 μ M). The efficiency of the sensor was evaluated in food products as well and showed excellent sensitivity, repeatability, and limits in detection. Yang et al. ^[43] fabricated a ractopamine sensor using an OMC-modified electrode by the cyclic voltammetric method. The ractopamine is a toxic veterinary drug used as heart tonics, bronchodilators, and tocolytics. Ractopamine accumulates in animal tissue, and its consumption can cause frequent vomiting, muscular tremors, and cardiac palpitations ^[43]. Authors reported the oxidation mechanism of ractopamine along with its electro-oxidation behavior in the presence of an OMC-modified electrode ^[43]. Finally, the authors concluded that the present electrode can detect the ractopamine in pork samples with high accuracy, sensitivity, and selectivity. Nanomaterials of rare earth metals are also used for the construction of electrical and electronic devices due to the noticeable changes in their structure, morphology, and reactivity ^{[44][45]}.

Guo et al. developed an OMC-modified GCE for the detection of melamine ^[46] in milk products. Melamine is mainly used to produce melamine-formaldehyde resins and is also used as a filler for protein-rich diets in milk powder. Excessive intake of melamine causes kidney stones, traces of blood in urine, high blood pressure, and little to no urine production. The fabricated electrode, in the presence of 0.1 M copper ions converts, non-electroactive melamine into an electroactive Cu-melamine complex ^[46].

5. Boron Doped Diamond (BDD)-Based Nanocomposites for Food Quality Assessment

Most carbon materials are highly conductive ^{[47][48][49][50]}. However, a natural or pure diamond cannot be used as the material for the construction of an electrode due to its excellent electrical insulation properties. However, the conductivity of diamond materials can be enhanced by p-doping approximately 1018 and 1021 atom cm⁻³ with boron. Boron-doped diamond is proven to have one of the largest potential windows of all electrode materials, with significant conductivity, stability, robustness, low noise, chemical inertness, biocompatibility, resistance to passivation, and comparatively less fouling and background current. Due to a wide range of properties, BDD is considered one of the strongest candidates for electrode materials to determine toxic food chemicals.

Švorc et al. used a miniaturized boron-doped diamond electrode to detect theobromine in chocolate products ^[51]. The main source of theobromine (a dimethylxanthine alkaloid) is cocoa, chocolate, and other related products. It can cause

reduced yields of cattle milk, thymus atrophy in rats, retarded growth, and lethargy in pigs. Under optimized experimental conditions, the linear calibration curve for theobromine was observed in the concentration range of 0.99-54.5 µM with a sensitivity of 0.07 µA/µM. Micheletti et al. [52] carried out voltammetric determination of one of the toxic azo dyes, carmoisine E-122, in food using a cathodically pretreated BDD electrode. Commonly, the synthetic carmoisine E-122 is used in many foods, such as chewing gum, candies, sauces, and beverages, to provide a red to maroon color to the food [52]. However, excessive consumption of carmoisine E-122 has an adverse effect on the renal and hepatic functions and results in hyperactivity in children ^[53]. The authors have reported that the analyst is linear over the concentration range of 0.059–1.31 µmol L⁻¹ with limits of detection of 7.0 for an anodic process. The authors reported that the prepared electrode successfully determined the carmoisine E-122 dye present in both surface water and food samples with maximum sensitivity [52]. Medeiros et al. [54] simultaneously determined different types of synthetic dyes present in food using a single electrode (a cathodically pretreated BDD electrode). Most of the synthetic dyes contain toxic chemicals and can cause cancer, asthma, and many other diseases. Usually, these dyes are added to sweets, sugar candies, beverages, jellybeans and so on, to make them more visually appealing to customers ^{[54][55]}. Chuanuwatanakul et al. ^[56] fabricated a BDD-based electrode to determine chloramphenicol (a veterinary drug) using cyclic voltammetry. This antibiotic drug was very popular in treating and preventing bacterial infections in animals due to its low cost and is mostly used in aquaculture farming. However, it is banned all over the world because it can cause aplastic anemia, agranulocytosis, and many more diseases in humans [57].

6. Fullerenes and Their Nanocomposites for Food Quality Assessment

Fullerene is also called a buckyball and it is an allotrope of carbon wherein single and double bonds involved in carbon atoms connect to form a fused ring having 5–7 carbon atoms. Fullerenes may be hollow spheres, ellipsoids, or many other sizes and shapes. Generally, there are different types of fullerenes available based on the number of carbon atoms present in the molecule. It is possible to prepare C_{70} , C_{76} , C_{78} , C_{84} , C_{100} , and C_{240} fullerenes. These new compounds of carbon materials exhibit unique and unexpected properties. The extraordinary electrical conductivity of fullerenes makes it a potential candidate for fabricating electrochemical sensors for determining food quality.

Tajeu et al. developed fullerene/MWCNT/Nafion-modified GCE for the detection of caffeine ^[58]. Most of the pharmaceutical and food industries use caffeine in tea, coffee, and soft drinks. Excessive use of caffeine causes increased gastric acid secretion and diuresis and can stimulate the central nervous and cardiovascular systems. The fabricated sensor provides an irreversible oxidation peak of caffeine at approximately +1.33 V in HClO₄. The authors reported that the electron transfer process is diffusion-controlled ^[58]. The fabricated electrode shows excellent stability even in the presence of interfering compounds. Therefore, the authors claimed that the fabricated sensor is a potential candidate for the detection of caffeine in various foods and drugs.

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