

FULL PAPER

An overview of modified sensors with focus on electrochemical sensing of sulfite in food samples

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Sulfite is used as preservatives to slow browning and discoloration in foods and beverages during preparation, storage and distribution. In various pharmaceutical and food industries, sulfite is utilized for inhibition of nonenzymatic and enzymatic browning. Also, in brewing industries, it acts as an antioxidizing and antibacterial agent. Several toxic and adverse reactions, including vitamin deficiency, hypersensitivity, and allergic diseases have been attributed to sulfites ingestion that may cause dysbiotic oral and gut microbiota events. Thus, the content of sulfite in foods must be controlled and monitored, and it is essential to find a specific, reproducible, and sensitive method to detect sulfite. Some analytical solutions are being tested to quantify sulfite. However, due to their advantage over traditional techniques, electroanalytical techniques are attracting much attention, because they are simple, fast, affordable, and sensitive to implement. In addition, modifying the electrodes controls the morphology and size that results in the miniaturization to be used in portable electro-chemical devices. Therefore, the present review addressed some articles on the electro oxidation of sulfite from the real samples with the use of various kinds of electro-chemical sensors.

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Introduction

Experts in the field have largely considered the sulfite contribution and the respective alter ego, that is, sulfur dioxide, into the agri-food products due to its impact on some medical conditions. In fact, sulfite produces different technical impacts on various kinds of food products like inhibiting the enzymatic browning in the fresh products and shrimp, inhibiting the non-enzymatic browning in the dried or dehydrated vegetables and fruits,

anti-microbial actions in wine-making and wet-corn milling, anti-oxidant impact, bleaching in the maraschino cherries and hominy and dough conditioning [1-4]. Additionally, sulfite is utilized for preserving effectiveness and stability of specific medicines. Put differently, the increased concentration of sulfite causes allergic reaction in the sensitive consumers. In general, human serum shows a sulfite concentration in ranges between 0 and 10 μM .

Moreover, the U.S. Food and Drug Administration (FDA) suggested the existence of warning labels on all food products consisting of more than 10 mg/kg sulfite or beverage consisting of >10 mg/L sulfite. For this reason, sulfite content must be cautiously controlled in foodstuff [5-8].

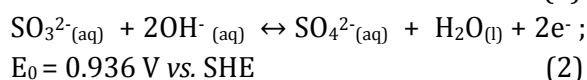
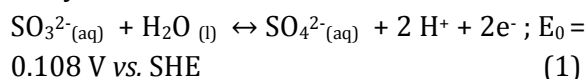
Researchers have presented various methods to determine sulfite in the beverages and food like titrimetry, flow injection analysis (FIA), iodimetry, ion chromatography, spectro-photometry, chemiluminescence, fluorometry, high-performance liquid chromatography (HPLC) as well as gas chromatography. Nonetheless, a number of them do not have accuracy and sensitivity (for example, spectrophotometry & iodimetry) and other methods need laborious sample preparation, costly instrument and professional experts [9-14].

Experts in the field have utilized electro-chemical systems as one of the solutions to the decentralised testing for numerous specimens because they have required cheap equipment and inexpensive operations, could be miniaturized, and have been accessible by simple-dipstick sampling [15-35]. In addition, electro-chemical methods have been considered to be very selective, quantitative, less time-consuming, and strongly sensitive, and enjoy broad dynamic ranges and fast responses [36-62]. Moreover, redox features of sulfite have been defined in a way that it is possible to reduce or oxidize the analyte in order to easily modify sulfite for electro-chemical determination.

Nevertheless, electro-chemical analysis of sulfite has been restricted by the problem of electrode surface fouling, issue of time, and reagent consuming process for the electrode surface regeneration. It is notable that while developing electro-analytical sensing systems to detect sulfite, sensing electrodes would be frequently modified with proper substances for achieving favorable outcomes in selectivity and sensitivity [63-100].

As nano-science and nano-technology have been developed, researchers have remarkably considered nano-materials and thus they contribute importantly to cases like catalysis, micro-electronics as well as electro-chemical sensors because of the respective thermal, optical, catalytic and electrical feature [101-130]. Therefore, nano materials would be highly feasible in enhancing the sensors' performance due to increased ratio of surface to volume, higher electrical conductivity, suitable biocompatibility, very good catalytic abilities as well as surface reaction activities [131-150].

Furthermore, electro-analytical chemistry has been largely developed in presenting screen-printed electrodes (SPE), the modified carbon-based electrodes and other electrodes. Therefore, our review is a summary of current achievements of distinct chemically-modified electrodes for electro-chemical sensing of sulfite. Designing the mentioned sensor has enabled researchers to improve the analytical performances of the available electro-analytical sensing systems with regard to selectivity, sensitivity, multi-plexed detection capability as well as field portability. Sulfite is an electroactive compound in both acidic and alkaline media (Eq. 1 and 2). Therefore, electrochemical sensors are ideal choice for analysis of sulfite in aqueous solution. This review paper focused on introducing modified sensors and application of them for sulfite analysis.



Electrochemical detection of sulfite

According to the research, sensing electrodes should be used for electro chemical sensing of sulfite in order to pass current to an aqueous solution and create a number of measurable and beneficial electrical signals relative to the electro-chemical reactions in the solution

because of the presence of sulfite ions. Since the electrodes can be miniaturized and easily modified, electro-chemical setup would be usually shorter, simplified and portable, resulting in their effectiveness and usefulness for sulfite determination. Electro-chemical procedures commonly have a setup with the working electrode (WE), reference electrode (RE) as well as count electrode (CE). Therefore, it is possible to modify WE with various substances for specific detection of sulfite. Such interface materials contribute vitally to the selective and sensitive detection of sulfite [151-155].

Since they are inexpensive, have higher electron transfer kinetic, acceptable chemical stability, diverse carbon materials, from the traditional large-scale carbon-based materials (e.g., glassy carbon electrodes (GCEs), CPEs & graphite) to the well-known nano sized carbon materials (e.g., carbon nanotubes (CNTs)), the screen printed electrodes (SPEs), gold (Au) electrodes and Pt electrodes, they have a widespread utilization in the electro-chemical sensing systems.

GCEs for sulfite detection

It is widely accepted that glassy carbon (GC) artefacts have been created by a robust controlled heating plan of a pre-modelled polymeric (phenol-formaldehyde) resin body in an inert atmosphere so that the carbonization procedure initiates at a temperature >300 °C. Hence, it is recommended that polymeric material does not pass into a tarry or liquid state directly before the carbonization because it is possible to obtain a product that fully differs from the glassy carbon. Therefore, the carbonization process, wherein nitrogen, oxygen and so forth are removed (300 °C to 500 °C) should be run very slowly for ensuring diffusion of the gaseous products to the surface. Moreover, rapid heating would result in the increased pressure into the material, which produces cracks, distortion and blister. Thus, thickness

of the artefacts walls should be confined ~ 5 mm. It is notable that at a temperature range from 500 to 1200 °C, hydrogen would be slowly removed and just carbon would remain. Analyses have shown that the carbonization process would be followed by the volume shrinkage of $\sim 50\%$ and annealing at >1200 °C gradually eliminate the local defects. In addition, the fully uniform ribbons may stack above each other and create microfibrils that twist, bend and or intertwine to yield a GC end-product. GC has been extensively employed in voltammetric electrodes during the last twenty years. Since they exhibit lower residual current in the ranges of nearly $+1$ V in aqueous media as well as more extended ranges in organic solvents and aqueous micellar solution, they are usually utilized as one of the indicator electrodes. Moreover, numerous papers made up of the GCEs exhibited higher sensitivity of the apparent rates of redox reactions toward the electrode surface state [156-171].

A lot of modified GCEs have been made to detect sulfite from the real samples (Table 1).

In their study, Manusha and Senthilkumar introduced a new redox mediator-based ionic liquid in order selectively and sensitively detect sulfite. Therefore, they devised phenothiazine imidazolium ionic liquid with hexa-fluorophosphate counter anion (PTZ-IL), synthesized it as a molten salt, and employed as a redox mediator in preparing the modified electrode. Then, PTZ-IL has been immobilized on the multi-walled carbon nanotubes (MWCNTs) deposited GCE for fabricating the PTZ IL/MWCNT/GCE modified electrode. Figures 1A and 1B present the synthetic process of PTZ-IL formation and a schema of the electrode modification. In addition, researchers applied cyclic voltammetry (CV) for examining the electro-chemical functions of the PTZ-IL/MWCNT/GCE. It has been found that this new sensor has a redox couple with the cathodic and anodic peak potential at $+0.594$ and $+0.707$ V, attributed to the phenothiazine/phenothiazine radical cation

redox couple. Moreover, PTZ-IL/MWCNT/GCE exhibited very good electro-catalytic activities for sulfite oxidation, according to which, an amperometric sensor has been presented to detect sulfite. Furthermore, this non-

enzymatic sensor displayed a wider linear range between 30 and 1177 μM with $282.2 \mu\text{A}\text{mM}^{-1} \text{cm}^{-2}$ sensitivity and a limit of detection (LOD) equal to $9.3 \mu\text{M}$ for detecting sulfite [172].

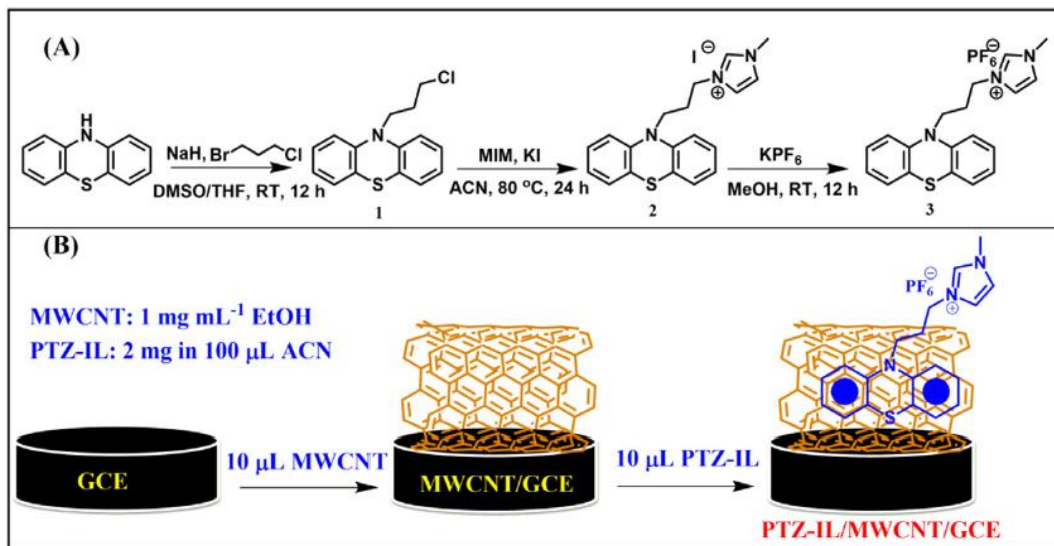


FIGURE 1 (A) The synthetic process of PTZ-IL (3) and (B) Schema of the construction process of PTZ-IL/MWCNT/GCE [172]

In this regard, Zhu *et al.* addressed the synthesis of LaFeO₃ using the sol-gel method and thus procured the LaFeO₃/graphene composite materials by ultra-sonic dispersion. Then, they utilized the composite modified electrode as the electro-chemical sensor for detecting the sulfite content. According to the electro-chemical test outputs, LaFeO₃/graphene composite modified electrode had a suitable response signal for sulfite and the response current was proportional to the sulfite concentration in ranges between 1 and 200 μM . Moreover, linear relationship equaled $I(\mu\text{A})=0.119 C(\mu\text{M})+0.135$, correlation coefficient (R^2) equaled 0.994, and LOD ($S/N=3$) equaled 0.21 μM . Finally, this new technique has been substantially utilized to the white wine samples and recovery rate has been ranged between 97.63 and 103.02% ($n=5$) [173].

Moreover, Yang *et al.* constructed a sulfite electro-chemical sensor by Nafion and molybdenum disulfide (MoS₂). According to DPV and CV, MoS₂ showed very good catalytic

activities for the redox of SO₃²⁻. Upon the optimization of acidity of the scan rate, supporting electrolyte, and other variables for electro-chemical response of SO₃²⁻, this new sensor exhibited a broad dynamic linear range between 5.0×10^{-3} and 0.5 mM ($r=0.997, n=15$) to detect SO₃²⁻ with a LOD equal to 3.3×10^{-3} mM that may be employed for SO₃²⁻ content detection in water, which enjoys benefits like acceptable reproducibility, prolonged stability, reasonable recovery and anti-interference [174].

In addition, Adeosun *et al.* showed the electro-chemical synthesis of the conductive poly-pyrrole chitosan (PPYCHI) thin film for sensitively detecting sulfite in the real specimens. Figure 2 is a schema of this synthetic process. As seen, this co-polymeric PPY-CHI film has acceptable electro-catalytic behaviours for oxidizing sulfite. Therefore, it has been employed to detect sulfite with DPV and a LOD, linearity and sensitivity equal to 0.21 μM ($S/N = 3$), 50-1100 μM and $15.28 \mu\text{A}\mu\text{Mcm}^{-2}$, respectively [175].

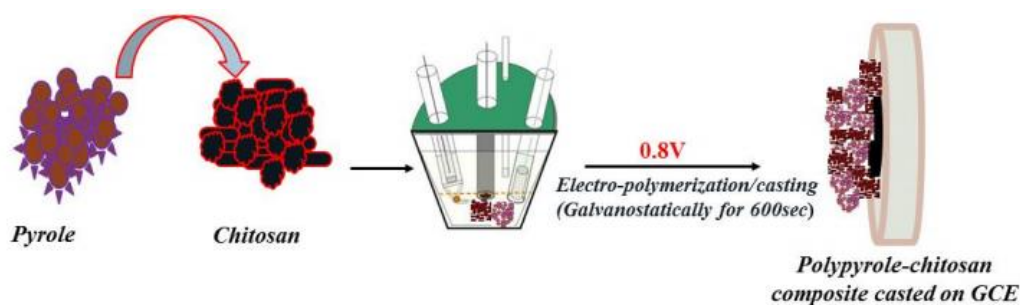


FIGURE 2 Schema of the synthetic process for electro-polymerization of poly-pyrrolechitosan [175]

Furthermore, Pandi *et al.* electro-deposited the completely organized lutetium (III) hexacyanoferrate micro-particles on the poly(aurine) modified GCE, that is, (LuHCF/poly(aurine)-GCE). It has been found that this LuHCF/poly(aurine)-GCE enjoys very good electro-chemical activity as well as higher sensitivity for sulfite sensing. They also examined electro-chemical oxidation of sulfite on the LuHCF/poly(aurine)-GCE with the use of DPV and CV. Results have shown that this modified electrode had higher sensitivity equal to $448 \mu\text{A mM}^{-1} \text{cm}^{-2}$ and 1.33 Mm LOD for electro-chemical oxidation of sulfite with more acceptable functionality and selectivity [176].

Moreover, Yu *et al.*'s study dealt with the synthesis of Au NPs-reduced graphene oxide composite (AuNPs-rGO) using the chemical coreduction method with ethylene glycol (EG) as the reducing agent. They modified the GCE with an AuNPs-rGO composite through dropping method. In addition, researchers employed CV and electrochemical impedance spectroscopy (EIS) for investigating electro-chemical behaviors as well as electro-catalytic activities of the AuNPs-rGO/GCE electrode for sulfite oxidation. With regard to the outputs, AuNPs-rGO/GCE has higher electro-chemical activities to oxidize sulfite through synergistic effect of the rGO and AuNPs. Based on the results in $0.10 \text{ M H}_2\text{SO}_4$, linear range for sulfite detection via amperometry at 0.40 V versus SCE equaled $2.0 \times 10^{-7} - 2.3 \times 10^{-3} \text{ M}$ ($r^2 = 0.9956$) with the LOD equal to $4.5 \times 10^{-8} \text{ M}$ ($S/N = 3$) [177].

In their study, Devadas *et al.* reported a shape-controlled preparation of Praseodymium hexacyanoferrate (PrHCF) with a simple electro-chemical procedure. Therefore, the electro-chemically fabricated PrHCF modified GCE showed a very good electro-catalytic activities for sulfite oxidation. Electro-chemical oxidation of sulfite on the PrHCF modified GCE has been examined by the linear sweep voltammetry (LSV) and CV. Results have shown this sulfite sensor sensitivity equal to $0.036 \text{ mA mM}^{-1} \text{cm}^{-2}$ and lower LOD as 2.15 mM . Finally, researchers detected sulfite from the specimens obtained from the tap water and red wine for confirming the real-time utilization of the PrHCF modified GCE [178].

Wang and Xu presented one of the new methods for selectively extracting the free (pH 8.4) and total sulfite (pH of 11.0) from the muscle foods and the following detection by a voltammetric sensor. Their technique has been on the basis of eletro-catalytic oxidation of sulfite at the modified GCE constructed by immobilization of $9 \mu\text{g}$ of acetyl-ferrocene (AFC) over the GCE surface along with $35 \mu\text{g}$ of carbon black (CB) for improving the electron transfer into the poly (vinyl butyral) (PVB) membrane matrix. Results indicated linearity of the external standard calibration curve in ranges between 0.03 and 4.0 mmol L^{-1} with $15 \mu\text{mol L}^{-1}$ LOD. Ultimately, this new technique could be utilized for determining free and total sulfite in the shrimp muscle fortified specimens [179].

TABLE 1 Modified GCEs to detect sulfite

GCE modified with	Method	LOD	Linear ranges	Real samples	Ref
PTZ IL/MWCNT	amperometric	9.3 μ M	30–1177 μ M	Vinegar and pickle	172
LaFeO ₃ /graphene	DPV	0.21 μ M	1–200 μ M	White wine	173
MoS ₂ /NF	DPV	3.3 \times 10 ⁻³ mM	5.0 \times 10 ⁻³ –0.5 mM	Water	174
PPY-CHI	DPV	0.21 μ M	50.0–1100.0 μ M	Orange Juice, malt drink and human serum	175
LuHCF/poly(taurine)	DPV	1.33 μ M	-	Water	176
AuNPs-rGO	amperometric	4.5 \times 10 ⁻⁸ M	2.0 \times 10 ⁻⁷ –2.3 \times 10 ⁻³ M	Red wine	177
PrHCF	LSV	2.15 μ M	600 μ M–8 mM	Red wine and water	178
AFc/CB/PVB	CV	15 μ M	0.03–4.0 mM	Shrimp muscle	179

CPEs for sulfite detection

It is widely accepted that carbon paste that has been proposed as one of the mixtures of the graphite powder and an appropriate liquid binder, is a laboratory-made electrode material with the widespread utilization [180–190]. It is also the most flexible substrate for biological and chemical modifications [191–200]. It is notable that heterogeneous feature of the CPEs, specific effects of the liquid binder, variability in the employed carbon materials as well as diverse proportional compositions need extensive and reliable characterization procedures [201–209]. Carbon paste electrodes amplified with conductive mediators are a good choices with powerful ability in electrochemical sensing systems [210–219]. For example, Ralph Norman Adams (2008) from Kansas University published a one-page report, wherein the above electrode has been presented that has been basically developed as one of the alternatives to the dropping mercury electrode [220–235]. Because of multiple beneficial features, CPEs have a widespread utilization mostly for voltammetric measurements. Nevertheless, carbon paste-based sensors could be employed in coulometry, potentiometry and amperometry. As opposed to the complex modification of the solid substrates, it is possible to easily modify the carbon pastes for the quantitative new sensors with the favorable and usually pre-defined features.

CPEs have been proposed to be very well known due to their inexpensive accessibility that make them particularly appropriate to prepare the electrode materials with the intended compositions as well as pre-defined features. These advantages stem from the optimum constellation of the physico- and electro-chemical features of this carbon-like substrate, which have been highly known amongst the practical as well as theoretical electro-chemists or even electro-chemical science. Finally, such electrodes are utilized as the strongly selective sensors for organic and inorganic electro-chemistry [236–251].

In this regard, Zabihpour *et al.* dealt with the incorporation of the MgO/SWCNTs nano-composite and 1-Butyl-3-methyl-imidazolium bis(trifluoromethylsulfonyl)imide [Bmim][Tf2N] into the CP matrix for obtaining a modified CPE (MgO/SWCNTs-[Bmim][Tf2N]-CPE) as the electro-analytical instrument. According to the results, MgO/SWCNTs-[Bmim][Tf2N]-CPE created a better analytical oxidation signal for FA that has been 3.17 times higher than the unmodified sensors. Moreover, this electrode produced distinctive electro-oxidation signals for FA $\Delta E_p=300$ mV in the presence of sulfite, verifying its utility for FA detection in the presence of sulfite ions that are usually found in the food samples such as red wine. Additionally, MgO/SWCNTs-[Bmim][Tf2N]-CPE has been shown as one of the robust tools for FA and sulfite ions detection with the recovery data of 97.84 to 103.1% in the

concentration range between 0.009 and 450 μM as well as 0.1 and 450 μM with a LOD equal to 3.0 nM and 50 nM [252].

In addition, Norouzi and Parsa created a modified electrode in the course of the electro-polymerization of 4-Aminobenzoic acid in the presence of sodium dodecylsulfate (SDS). Therefore, they incorporated the Ni(II) ions into the polymer via immersing the modified electrode into a 0.1 M Ni(II) ions solution. Then, researchers employed CV for investigating the electro-chemical behaviors of the Ni/poly(4 aminobenzoic acid)/sodium dodecylsulfate/CPE (Ni/poly(4-AB)/SDS/CPE). Results have shown the acceptable activities of the polymeric modified electrode for sulfite electro-oxidation in a phosphate buffer solution (PBS) at a pH of 11 and finally, LOD equaled 0.063 mM. Based on the optimum experimental condition, peak current response linearly enhanced as the sulfite concentration elevated in ranges between 0.1–1 and 1–10 mM [253].

Furthermore, Miraki *et al.* presented one of the electro-chemical platforms based on the CPE modified with the NiO NPs and acetylferrocene (AF) (CPE/NiO-NPs/AF). According to the findings, CPE/NiO-NPs/AF had acceptable electro-catalytic activities to detect sulfite in the concentration range of 0.005 to 500 μM with the LOD of 0.001 μM . It has been found that the electro-catalytic interactions of sulfite with AF at the CPE/NiO-NPs/AF surface may resolve the overlapping single of nitrite and sulfite for their simultaneous detection. Finally, researchers found higher performance of CPE/NiO-NPs/AF to detect nitrite and sulfite in the samples of wastewater [254].

In another study by Winiarski *et al.*, water-soluble form of 3-npropyl(4-methylpyridinium) silsesquioxane chloride (Si4Pic+Cl-) has been synthesized and characterized. Therefore, researchers demonstrated its potency to be utilized as one

of the stabilizing agents for AuNP. In fact, CPEs modified with the Si4Pic+Cl- and AuNP have been fabricated and employed in detecting and quantifying sulfite in the acidic medium. With regard to the voltammetric signal for sulfite, the electrode modified with the AuNPs (CPE/Au-Si4Pic+Cl-) exhibited an electro-catalytic impact of ca. -0.20 V in comparison with the bare electrode. According to the square wave voltammetry analysis (SWVA) and based on the optimum experimental condition, reduction peak enhanced as the sulfite concentration elevated in ranges between 2.54 and 48.6 mg L^{-1} . Finally, LOQ and LOD equaled 2.68 and 0.88 mg L^{-1} for sulfite [255].

Another study conducted by Sroysee *et al.* described an amperometric sulfite bio-sensor, which contained a CPE ($\text{Fe}_3\text{O}_4@Au$ -Cys-FA/CPE) modified with the immobilized sulfite oxidase (SOx) on an Au-coated magnetite NP core encased within a conjugated folic acid (FA) cysteine (Cys) shell. Therefore, a poly-dimethylsiloxane (PDMS) as well as mineral oil mixture as the binder has been used to manufacture the bio-sensor electrode, which enhanced sensitivity and physical stability of the electrode. Figure 3 is an illustration of the preparation process of the sulfite bio-sensor. As seen, $\text{Fe}_3\text{O}_4@Au$ -Cys-FA electrode exhibited acceptable electro-catalytic activities with reasonable retention of the chemisorbed SOx on the electrode due to the respective larger surface areas. Then, researchers employed amperometric measurements from the $\text{Fe}_3\text{O}_4@Au$ Cys-FA/CPE biosensor to quantify sulfite, and used an in-house assembled flow cell at +0.35 V (versus Ag/AgCl) with the PB carrier of 0.10M at a pH of 7.0 and the flow rate equal to 0.8 mL min^{-1} . Ultimately, this system detected sulfite in ranges between 0.1 and 200 mg L^{-1} with 10 mg L^{-1} LOD (3 s of blank) [256].

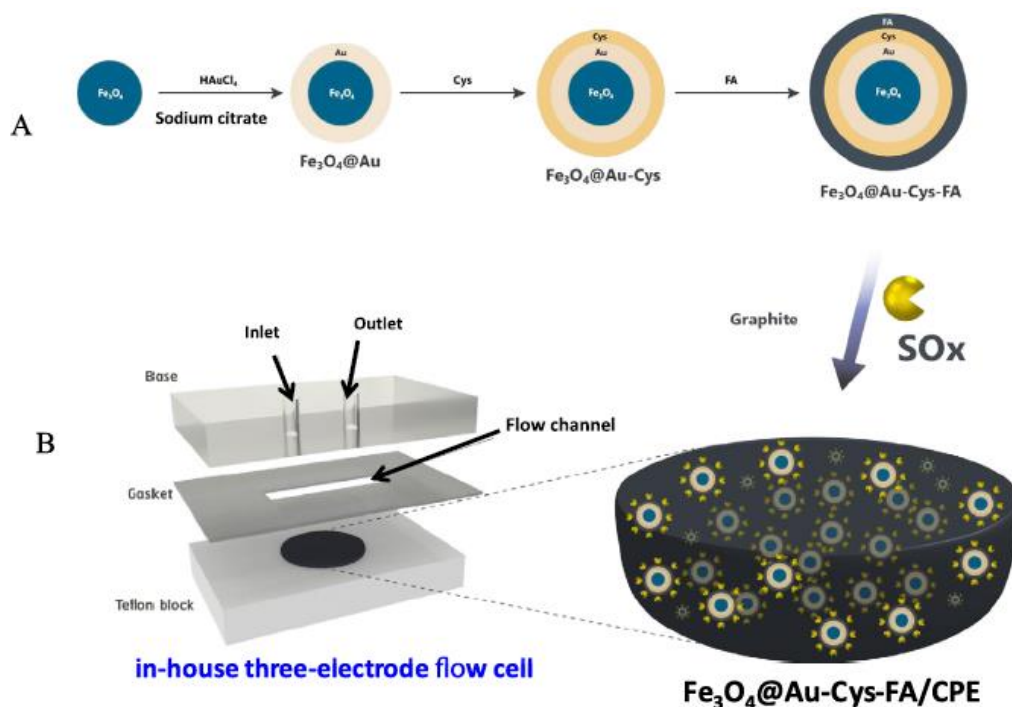


FIGURE 3 The preparation process of the sulfite bio-sensors (A) $\text{Fe}_3\text{O}_4@Au\text{-Cys-FA}$ nano-composites with the immobilized SO_x . (B) $\text{Fe}_3\text{O}_4@Au\text{-Cys-FA}$ modified CPE [256]

It is notable that Silva *et al.* also devised a square-wave voltammetric technique with regard to the sulfite electro-chemical reduction to quantify this preservative in the commercial beverages. Therefore, they used CPE chemically modified with the MWCNTs as the working electrode. It has been found that at the optimal experimental condition, the linear response to the sulfite concentration ranges between 1.6 and 32 mg $\text{SO}_2 \text{ L}^{-1}$ (25 to 500 $\mu\text{mol L}^{-1}$ of sulfite) and LOD equals 1.0 mg $\text{SO}_2 \text{ L}^{-1}$ (16 $\mu\text{mol L}^{-1}$ of sulfite) [257].

SPEs for sulfite detection

Researchers have used the screen-printing methods in the electronics industry to make the printed circuit boards since 1950. In fact, screen-printed technology involves the layer-by-layer deposition of ink on a solid substrate by a screen or mesh, which defines the sensor geometry. Moreover, such a new technology enjoys design flexibility, acceptable reproducibility, process automation as well as selection of diverse substances. However, SPEs do not have the common problems of the classical solid electrodes like laborious

cleaning procedure and memory effects. Finally, analyses have provided a new window due to the broad ranges of form of the SPEs modification [258-293].

There is enough information of the SPEs fabrication to design disposable sensors, which entails some fundamental phases like choosing screen, choosing and preparing ink, choosing substrate as well as the drying, curing and printing phases. Therefore, it is possible to make numerous configurations of the SPEs on the basis of various substances. In comparison with other technologies for making the electrodes like thick- and thin-film electrodes are affordable and can be easily fabricated so that can be used for mass production [294-322].

Aflatoonian *et al.* reported synthesizing the MOWS_2 nano-composite and employed DPV, CV as well as chrono-amperometry (CHA) for examining the electro-chemical behaviours of sulfite on the MOWS_2 nano-composite modified the SPEs. According to the electro-chemical specification, researchers observed acceptable electro-catalytic activity and surface area impact of the MOWS_2 nano-

composite. They also found the considerable enhancement of oxidation signals of sulfite on the MOWS₂/SPE as compared with the bare SPEs. Ultimately, with regard to the optimal condition, sulfite quantification ranged from 0.08-700.0 μM with lower LOD of 0.02 μM (S/N=3) [323].

Furthermore, Hemmati *et al.* dealt with synthesizing the Fe₃O₄ NPs via solvo-thermal approach with the use of the ethylene glycol (EG). Then, the dropping procedure has been used to modify the GSE with the Fe₃O₄ NPs. Researchers have also employed DPV, CHA and CV for investigating electro-catalytic activities as well as electro-chemical behavior of Fe₃O₄/SPE electrode for sulfite oxidation. According to the DPV analysis and based on the optimal experimental condition, oxidation peak enhanced as the sulfite concentration elevates in ranges between 0.5 and 100.0 μM. Finally, LOD equaled 0.1 μM for sulfite [324].

Moreover, Maaref *et al.* designed their own electro-chemical technique to detect sulfite with the use of Schiff-base modified graphite SPEs as a disposable chemo-sensor. Therefore, they used differential pulse voltammograms of the modified SPE in the presence of sulfite and observed a typical peak current at 350 mV. Moreover, they found the sensor linear responses in the concentration ranges between 0.5 and 300.0 μM of the analyte. Finally, LOD equaled 0.3 μM for sulfite and thus this method could be used to sulfite detection in the natural water samples [325].

In their study, Molinero-Abad *et al.* modified the SPCEs with tetra-thiafulvalene (TTF) and sulfite oxidase enzyme in order to selectively and sensitively detect sulfite. Therefore, they optimized the amperometric experimental condition with regard to the significance of the sulfite quantification in the wine samples as well as innate complicatedness of the samples, specifically red wine. Researchers observed the bio-sensor response to sulfite with a cathodic current (at +200 mV versus the SP Ag/AgCl electrode & pH 6) in the broad concentration

ranges with 6 μM LOD at 60 °C. Therefore, this method could be employed for sulfite detection in red and white samples, with the average recovery equal to 101.8 and 101.5% [326].

Consequently, Molinero-Abad *et al.* reported an amperometric detection of sulfite with the use of the SPCEs modified with silver and Au NPs. It has been found that the electrode is relatively selective and shows responses to sulfite with the oxidation current at 300 mV and pH of 6 in the concentration ranges between 9.80 and 83.33 μM with the recoveries in ranges between 96 and 104 %. Hence, this method could be employed for sulfite detection in pickle juice, vinegar and drinking [327].

Other kinds of sensors for sulfite detection

A composite has been prepared by Do Carmo *et al.* from titanium (IV) silsesquioxane and phosphoric acid (TTiP) and then occluded into HFAU zeolite (ZTTiP). In the next step, the material has been modified chemically with nickel and potassium hexa-cyanoferrate (III) (ZTTiPNiH). Afterwards, a modified GPE has been used to obtain voltammetric behavior of ZTTiPNiH, which indicated a completely organized redox couple with KCl (3M) (20% w/w; v = 20 mV s⁻¹; KCl; 1.00 mol L⁻¹) and a formal potential of E⁰=0.51V (versus Ag/AgCl (sat)) that corresponded to the Ni^{II}Fe^{III}(CN)₆/Ni^{III}Fe^{III}(CN)₆ redox process. Upon the precise voltammetric examinations, the GPE modified with ZTTiPNiH has been employed for easy and fast determination of sulfite. Then, according to the analytical curve, a linear response has been achieved in the concentration range between 0.05 and 0.80 mmol L⁻¹, the LOD (3σ) equal to 0.05 mmol L⁻¹, amperometric sensitivity equal to 14.42 mA L mol⁻¹ and the relative standard deviation (RSD) equal to 4.21% (n=3) for sulfite [328].

In addition, Preecharueangrit *et al.* made their own amperometric sulfite sensor on the basis of the nickel hexa-cyanoferrate (NiHCF)

electro-deposited on a layer of the ordered mesoporous carbon, covered on an Au electrode surface (NiHCF/OMC/Au). This NiHCF/OMC/Au had acceptable electro-catalytic activities for sulfite oxidation and at the optimized condition, their sensor linearly responded in the concentration ranges between 2.5 μM to 50 mM and a 2.5 μM LOD (with the S/N ratio equal to 3). Finally, electrode showed higher operational stability so that it could be applied up to 104 times (with a RSD equal to 5.9%) and acceptable electrode-to-electrode repeatability (with an RSD of less than 7.0%, for $n=6$) [329].

Likewise, Devaramani *et al.* presented one of the simplified strategies for covalent anchoring of the cobalt hexa-cyanoferrate (CoHCF) particles on the graphitic carbon substrate with the use of p-phenylenediamine as the linker molecule. Therefore, they made a pellet electrode by the CoHCF particles modified graphitic carbon and employed it in the electro-catalytic oxidation of sulfite. Their sensor exhibited linear range of concentration of 4 to 128 μM of sulfite with a limit of quantification (LOQ) of 5.8 μM and 1.7 μM LOD [330].

Moreover, Adeloju and Hussain addressed the modification of a platinum electrode surface with platinum NPs (PtNPs). Therefore, they entrapped the enzyme sulfite oxidase (SOx) on the respective surface in an ultra-thin poly-pyrrole (PPy) film and thus those PtNPs with 30 nm to 40 nm diameter were deposited on the Pt electrode via cycling the electrode potential 20 times from -200 to 200 mV at the sweep rate equal to 50 $\text{mV}\cdot\text{s}^{-1}$. Additionally, researchers have used CV, EIS, potentiometry and chrono-potentiometry for examining the electro-chemical behaviors of the PtNPs/PPy-SOx film. Based on the optimal condition, this biosensor showed 57.5 $\text{mV}\cdot\text{decade}^{-1}$ sensitivity, a linear response between 0.75 and 65 μM of sulfite, 12.4 nM LOD as well as a response time between 3 s to 5 s [331].

In another study, Rawal and Pundir immobilized the sulfite oxidase (SO_x) (EC

1.8.3.1) purified from *Syzygium cumini* leaves over the Prussian blue NPs/poly-pyrrole (PBNPs/PPY) nano-composite film that were electro-deposited on the Au electrode surface. Therefore, they built their own electro-chemical sulfite bio-sensor with the use of a SOX/PBNPs/PPY/Au electrode as the working electrode, Pt wire as the auxiliary electrode linked by a potentiostat as well as Ag/AgCl as the standard electrode. Their bio-sensor has shown optimal response within two seconds while working at 20 $\text{mV}\cdot\text{s}^{-1}$ in 0.1 M Tris-HCl buffer at a pH of 8.0 and a temperature of 30 $^{\circ}\text{C}$. Researchers have shown the minimum LOD and linear range to be 0.1 μM (S/N=3) and 0.5-1000 μM . This sensor has been assessed with 95.0% recovery of the added sulfite in the samples of red wine and 1.9% and 3.3% within and between the batch coefficients of variations [332].

Look to the future

Electrochemical sensors have created a new world for the analysis of electroactive compounds in recent years [333-360]. Many researchers consider sulfite as one of the most widely used compounds in food industry as well as wood industry [361-365]. Electrochemical sensors have created a new window to sensing of this compound. It is hoped that in the coming years, by combining different technologies with electrochemical techniques, high-speed sulfite analysis will be performed and sulfite rapid detection kits will be marketed. One of the emerging technologies that can help in this direction is nanotechnology with unique features [366-380]. New nanomaterials with unique and amazing properties have transformed the world of science and quickly entered various sciences [381-395]. The ability of nanomaterials to improve the efficiency of analytical, removal and catalytic systems has made these compounds an important tool for achieving unattainable dreams in various sciences [396-411]. Therefore, it is expected

that in the coming years and in line with research conducted in recent years, kits with the ability of fast and inexpensive analysis to determine the amount of sulfite in food and industrial samples will be developed. Sulfite is utilized for inhibition of nonenzymatic and enzymatic browning. In addition, in brewing industries, it acts as an antioxidizing and antibacterial agent. Therefore, fast and portable systems are necessary for analysis of sulfite. In the near future and due to wide range application of sulfite, companies will need sensitive decomposition sensors for fast sulfite analysis.

Conclusion

Researchers have provided diverse electrochemical sensors to detect sulfite in different specimens due to the necessity of the application of a rapid procedure for determining content of sulfite in the food samples. Moreover, modifying the electrochemical sensors referred to the enhanced current responses for sulfite electro-oxidation, anti-fouling impact, and higher selectivity, stability and sensitivity. In general, we overviewed three kinds of electrochemical sensors, including the modified GCEs, the modified CPEs, and the modified SPEs. Since GCEs have been proposed to be the most applied substrates for modifications, they have larger diameters whereas the SPEs and CPEs are the miniaturized instruments with the very few samples. In fact, CPEs are inexpensive and can be easily prepared. In addition, they exhibited rapid regeneration of the active surface areas with the ability of eliminating the fouling effects. While preparing the CPEs, an oily binder would be employed for creating the paste and this mineral oil has been considered to be one of the insulating materials and also a reason for lower current ranges while utilizing SPE or GCE. In this way, the problem has been resolved and the use of ionic liquids as the binder materials for CPE formation has shown

that ILs have higher viscosity and conductivity. Therefore, the kind of real specimens employed should influence choosing the electro-chemical sensor to detect sulfite. A number of utilizations require lower LOD, others demand wider concentration ranges, and a number of them should use the strongly stable and selective methods whereas others should entail the concentration ranges greater than micromolar levels of sulfite. Fabrication of electrochemical sensors will create a bright future for making portable kits for analyzing sulfite in food samples. This point helps in fast investigation on food quality.

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