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MINISTÉRIO DA EDUCAÇÃO
UNIVERSIDADE FEDERAL DE UBERLÂNDIA
Faculdade de Engenharia Química
Programa de Pós-graduação em Engenharia de Alimentos



DANIELLE GONÇALVES FILHO

**NEW INSIGHTS INTO THE DETECTION OF FOOD
ADDITIVES IN FOODSTUFFS: THE USE OF PULSE
VOLTAMMETRY TECHNIQUES**

Patos de Minas – MG

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Orientadora: Djenaine De Souza

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RESUMO

Aditivos alimentares são compostos químicos adicionados intencionalmente durante a produção de alimentos para controlar funções tecnológicas, como pH, viscosidade, estabilidade (cor, sabor, sabor e odor), homogeneidade e perda de valor nutricional. Esses compostos são fundamentais para inibir o processo de degradação e prolongar a vida útil dos alimentos. No entanto, o consumo inadequadamente empregado ou exagerado pode resultar em efeitos adversos à saúde do consumidor devido a alguns aditivos alimentares tóxicos, e sua presença no corpo humano em alta concentração tem sido relacionada ao desenvolvimento de algumas doenças, como alergias, hematológicas, autoimunes, e distúrbios reprodutivos, bem como o desenvolvimento de alguns tipos de câncer. Assim, o desenvolvimento e a aplicação de métodos analíticos simples, rápidos, de baixo custo, sensíveis e seletivos para identificação e quantificação de aditivos alimentares em diferentes gêneros alimentícios são fundamentais para o controle de qualidade e garantia da segurança alimentar. Esta revisão apresenta novos insights sobre a detecção de aditivos alimentares em alimentos usando as principais técnicas de voltametria de pulso, pulso diferencial e voltametria de onda quadrada, indicando as principais vantagens, desvantagens e a importância na escolha adequada dos materiais dos eletrodos de trabalho para melhorar os resultados analíticos. Também são mostrados alguns mecanismos redox importantes de aditivos alimentares, o que permite o desenvolvimento de métodos voltamétricos confiáveis, precisos e baratos para quantificar esses compostos em amostras de alimentos.

Palavras-chave: Aditivos Alimentares; Controle Alimentar; Voltametria de Pulso Diferencial, Voltametria de Onda Quadrada; Mecanismos Redox

ABSTRACT

Food additives are intentionally chemical compounds added during foodstuff production to control technological functions, such as pH, viscosity, stability (color, flavor, taste, and odor), homogeneity, and loss of nutritional value. These compounds are fundamental to inhibition the degradation process and prolonging the shelf life of foodstuffs. However, inadequately employed or exceeded consumption can result in adverse effects on consumers' health due to some toxic food additives, and their presence in the human body in high concentration has been related to the development of some diseases, such as allergies, hematological, autoimmune, and reproductive disorders, as well as the development of some types of cancer. Thus, the development and application of simple, fast, low-cost, sensitive, and selectivity analytical methods for identifying and quantifying food additives in different foodstuffs are fundamental to quality control and ensuring food safety. This review presents new insights into the detection of food additives in foodstuffs using the mains techniques of pulse voltammetry, differential pulse, and square wave voltammetry, indicating the main advantages, drawbacks, and the importance in the adequate choices of the working electrode materials to improve the analytical results. Also are shown some important redox mechanisms of food additives, which allows the development of reliable, accurate, and inexpensive voltammetric methods for quantifying these compounds in foodstuffs samples.

Keywords: Food Additives; Foodstuffs; Food Control; Differential Pulse Voltammetry, Square Wave Voltammetry; Mechanisms Redox

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1 GENERAL CONSIDERATIONS

The technological advances achieved in the last decades have directly impacted the habits and lifestyle of the entire world population, promoted greater comfort and quality of life, and significantly increased their life expectancy. This directly affected the food industry, resulting in the development of new methods and technologies to serve this growing population and offer nutritious, tasty, and safe foods from microbiological, nutritional, and physical-chemical points of view. One of the strategies used by the food industries was the development and employment of food additives, which are necessary to increase industrialized foods production, improve the microbiological properties by inhibiting spoilage of foods and microbial growth, enhance rheological properties (texture and homogeneity), and control physicochemical (such as pH) and sensorial (color, flavor, sweetness, and crunchiness) properties of foodstuffs, extending their shelf life and ensuring food safety for consumers (CAROCHO; MORALES; FERREIRA, 2015; GONÇALVES-FILHO; DE SOUZA, 2022; XU *et al.*, 2021).

According to the *Codex Alimentarius* (2019), about 25 food additives classes are used in industrialized food production. However, the type and quantities of food additives require a strict food safety policy since some compounds may pose a risk to consumers' health. According to research carried out over the years, the chronic consumption of high doses of some of these food additives has led to the onset of various types of diseases in different populations around the world (DAMODARAN; PARKIN; FENNEMA, 2017; MARTINS; SENTANIN; DE SOUZA, 2019; XU *et al.*, 2021). Among these, the high consumption of foodstuffs containing synthetic dyes, a crucial class of food additives used mainly to make foods more attractive for children and adults, has been related to behavioral disorders, such as attention deficit hyperactivity disorder (ADHD), allergies, and even cancer (AHMED *et al.*, 2023).

Frequently consuming foodstuffs containing preservatives also concerns legislators, scientists, and doctors worldwide. Bayram & Ozturkcan (2022) showed that the high consumption of foodstuffs that contain preservatives such as nitrate and nitrite results in potential sources of nitrosamines which may be associated with various cancers of the digestive and intestinal tract, in addition to tumors in the central nervous system, and lymphoid system (BARRY *et al.*, 2019; XIE *et al.*, 2016). Besides, some

research has shown that sulfites, another type of preservative, can irritate the respiratory tract mucosa and promote digestive problems (MALAKOOTIAN; HAMZEH; MAHMOUDI-MOGHADDAM, 2022) and asthma in susceptible populations (ZHANG *et al.*, 2014a).

The consumption of synthetic antioxidants in quantities higher than those established by legislation has been related to adverse health effects, such as allergic and idiosyncratic reactions and some genotoxic effects. These types of food additives are the most used by the industry due to their efficiency in delaying the oxidation of lipid compounds that promote color change and loss of nutritional value of foods and are also responsible for increasing the shelf life of products (GONÇALVES-FILHO; DE SOUZA, 2022; MIKOVÁ, 2001; OUSJI; SLENO, 2020; WANG *et al.*, 2021).

So, the type and the quantities of food additives used in the industrial process, the foodstuffs type that contain specific food additives, and the maximum level for daily consumption are established by specific legislation of each country. However, all countries need to encourage adequate quality control in industrialized foods, with the identification and quantification of food additives used or produced during the industrial process. The necessary quality control can be performed using analytical techniques that are simple, fast, low cost, low residues levels, and with easy operationalization to ensure a suitable quality in the foodstuffs.

2 SCOPE AND APPROACH

Analytical techniques are essential in food analysis to identify and quantify chemical components in raw materials, independent ingredients, processed products, final products, and all intermediate compounds formed during manufacturing (MARINS-GONÇALVES *et al.*, 2023). Among chemical compounds present in foodstuffs, the food additives require a rigorous analysis due to the addition of inadequate quantities, and excessive consumption can result in health problems for consumers.

Food additives can be detected in foodstuffs using instrumental techniques, mainly chromatographic and electroanalytical. Chromatographic techniques are based on the mobility of each food additive driven or pushed by a mobile phase (gaseous, liquid, or supercritical fluid) across a stationary liquid or solid phase contained in a column. The food additives that are volatile and thermally stable can be separated and

detected by gas chromatography (GC), already high-performance liquid chromatography (HPLC) can be adapted for the separation and detection of any food additive since the previous choice of the organic solvent considered the polarity of the food additive (HASSOUN *et al.*, 2023; MARTINS; SENTANIN; DE SOUZA, 2019).

However, the presence of lipids, proteins, carbohydrates, salts, surfactants, pigments, emulsions, and suspension in foodstuffs samples can interfere with the analysis of food additives by chromatographic techniques, requiring preliminary steps of separation and sample preparation, which can produce toxic residues, are expensive and require more time for analysis. The high cost of acquiring and maintaining the equipment and the need for highly specialized labor is the main drawback of the detection of food additives by these techniques (MARTINS; SENTANIN; DE SOUZA, 2019).

The electroanalytical techniques can also be used in the detection of food additives, mainly the voltammetric techniques because food additives present in their chemical structures, some chemical groups that present the capacity to receive and/or donate electrons or interact with other components of the environment when a potential difference is applied across the electrode/solution interface. The transfer of electrons produces a voltammogram in which it is possible to identify the food additive by the peak potential and quantify it by the electrical signal, as this is directly proportional to the concentration of the food additive in the solution.

The most common voltammetric techniques employed in the determination of food additives are differential pulse voltammetry (DPV) and square wave voltammetry (SWV), which have good advantages such as sensitivity and selectivity, require a simplified sample pretreatment reducing the time and cost of analysis, the instrumentation had low-cost and can be easily operated by less skilled labor (SCHOLZ, 2010; WANG, 2006).

So, this review shows the applicability of the voltammetric techniques in the identification and quantification of food additives from different classes in various foodstuffs samples. The advantages and drawbacks of using voltammetric techniques, highlighting redox mechanisms, and the importance of adequate choices of material for working electrode preparation are also discussed, indicating the perspectives of voltammetric analysis in the food industry.

3 FOOD ADDITIVES

According to the United States Food and Drug Administration (2004), food additives are “any substance the intended use of which results or can reasonably be expected to result – directly or indirectly – in its becoming a component of or affecting the characteristics of any food.” The European Food Safety Authority (2009) defines them as “substances intentionally added to foodstuffs to perform certain technological functions, such as color, sweeten or help preserve food.”

In recent years, the food industries have used around 230 types of food additives, whose technological functions range from physical and chemical stability, inhibition of oxidative processes, and improvement of organoleptic characteristics, in addition to extending the shelf life of foodstuffs. (FAO/WHO, 2019; MARTINS; SENTANIN; DE SOUZA, 2019; WHO FAO, 2018). So, the list of food additives is extensive, but the main additives used are preservatives, flavorings, sweeteners, stabilizers, and dyes (AHMED *et al.*, 2023; BLEKAS, 2016; XIE *et al.*, 2016).

3.1. Toxicological of food additives

The safety of food additives is a public concern since recent research has indicated that some compounds can promote several metabolic consequences for humans, which makes it necessary to evaluate consumers' potential adverse health effects of ingesting food additives (SAHU, 2017). According to Lerner and Matthias (2015), a significant increase in the incidence of autoimmune diseases has been noted in conjunction with the expansion of industrial food processing and addictive consumption, such as soft drinks, candies, and chocolates.

The consumption of foodstuffs containing food additives has caused changes in intestinal functions, allowing several autoimmune diseases. The main suspicion is that the additives nullify the human epithelial barrier function, thus increasing intestinal permeability and resulting in the entry of foreign immunogenic antigens. However, more research is still needed for a better understanding and evaluation of exposure to food additives and the relationships with intestinal permeability and autoimmunity (FÖRSTI *et al.*, 2014; ROSSER; MAURI, 2016; VALESINI *et al.*, 2015).

Antioxidants are food additives widely associated with the development of some types of cancer, infertility, and other diseases and present a risk to several different

ecosystems. These food additives class are used mainly in foodstuffs rich in oil and fats lipids due to their efficiency in the inhibition of oxidative reactions that result in alterations in color, flavor, smell, and loss of nutritional value (GONÇALVES-FILHO; DE SOUZA, 2022). Propyl gallate, 3-tert-butyl-4-hydroxyanisole (BHA), 2,6-di-tert-butyl-4-methylphenol or butylated hydroxytoluene (BHT), and tert-butyl hydroquinone (TBHQ) are phenolic antioxidants considered as moderately hazardous. They are related to female and male infertility due to having an estrogenic effect, present a strong cytotoxic effect on the human, and exhibit carcinogenic and mutagenic effects (FELTER; ZHANG; THOMPSON, 2021; HAM *et al.*, 2019; LIU; MABURY, 2018; SARMAH *et al.*, 2020; YANG *et al.*, 2018).

Dyes are intensively used in food industries due to an influence directly on consumers' perception of flavor and sweetener. Synthetic dyes are chemically stable, easier to handle during foodstuff processing, and have bigger power colorants, promoting, this way, low cost. Among them, tartrazine and sunset yellow are the ones that stand out the most in foodstuff production. Studies to evaluate the genotoxicity of tartrazine demonstrated a toxic potential triggered by the ingestion of high dosages of the color or by cumulative effect due to the ability of tartrazine to damage human lymphocytes and bind to DNA (SASAKI *et al.*, 2002).

In another study, tartrazine was administered in mice and rats at dosages ranging from 125 and 500 mg/kg for 30 days, and it was observed that the animals showed neurotoxicity and deficits in learning and memory, both in a dose-dependent manner (MONTENEGRO, 2015). Based on histopathological examination, the neurotoxicity of tartrazine can be attributed to increased lipid peroxidation, production of reactive oxygen species, and inhibition of antioxidant enzymes (GAO *et al.*, 2011). However, it is necessary to note that the study employed a very high dose that people will likely ingest. Therefore, the need to obey the regulations established by each organization to avoid acceptable daily intake is not exceeded (AMCHOVA; KOTLOVA; RUDA-KUCEROVA, 2015).

Preservatives are food additives that prevent spoilage, improve food safety, and increase foodstuffs' shelf life due to their ability to avoid degradation by non-enzymatic and enzymatic reactions and reversible cleavage of proteins. Among them, sodium and potassium salts of nitrite and nitrate are used as curing salts in meat and the preservation of meat cured and sausages. However, these compounds can undergo chemical reactions to transform into nitroso amines, which are highly carcinogenic

compounds. Several studies point out the risks of ingesting these compounds due to be associated with the appearance of tumors in the stomach, gastrointestinal system, malignant thyroid, and bones (CROSS *et al.*, 2011; PICETTI *et al.*, 2022; SAMARDZIC *et al.*, 2021; XIE *et al.*, 2016).

The examples cited above are typical of the food additives most harmful to the consumer's health. So, due to all risks associated with the ingestion of large amounts of the most varied types of food additives, in addition to the contamination of various ecosystems by these compounds, it is necessary to regulate and frequently evaluate the concentration of these substances in foodstuffs and their residues in natural water, so that safety levels are maintained, and this is only possible through reliable, accurate, and inexpensive analytical techniques.

3.2. Chemical analysis of food additives

GC and HPLC are the techniques officially adopted by regulatory agencies worldwide to identify and quantify food additives. Analytical methodologies based on HPLC can be applied in the determination of a wide range of food additives, and it is enough that for the separation, the food additive is soluble in the liquid phase, which is possible by modifying the polarity of the organic solvent mixture used as mobile phase. Separation depends on the interaction of the food additive with the mobile and stationary phase, which results in similar retention times, requiring specific extraction steps to improve the selectivity of the analysis. The detection can be done at room temperature, avoiding destroying thermally sensitive additives. However, HPLC analysis requires a large number of organic solvents and is generally complex and time-consuming, resulting in band broadening and, therefore, lower resolution than GC. In addition, their main disadvantage is the high cost since large amounts of high-purity solvents are required.

Already, GC is used in the detection of any volatile and semi-volatile component in food, such as the food additives antioxidants, dyes, emulsifiers, preservatives, synthetic flavors, sweeteners, and taste enhancers, in foodstuffs and beverages. Some additives are directly analyzed, already others need previously derivative reactions to transform the nonvolatile to volatile compounds. Furthermore, selective, and sensitive detectors such as mass spectrometry, have broadened the applicability of CG in food

additives detection. However, the GC analysis requires a highly skilled operator due to the experimental complexity and high cost of the instrumentation.

Food additives analysis is performed in very complex matrices, which require several extraction steps so that it is possible to eliminate substances interfering agents and pre-concentration of analytes, which further increases cost and waste generation (DEMIRHAN *et al.*, 2017; SAURA-CAYUELA *et al.*, 2023). Further, better extraction procedures in combination with innovative detection systems, by HPLC or GC are improving more and more the sensitivity for detecting compounds, even at trace level.

4 ELECTROANALYTICAL TECHNIQUES

Electroanalytical techniques are based on measurements of electrical properties (conductivity, current, impedance, potential, and resistance, among others) in a system containing two or more electrodes that are placed in contact with a solution containing the dissolved species of interest and a suitable electrolyte. This way, the electrochemical process occurs in the interface electrode-solution allowing in electrical signals that can be used for analytical purposes and or evaluation of the redox process type that occurs in a predetermined medium.

In environmental, biological, and industrial analysis, such as food quality control, the electroanalytical techniques based on the variation of the potential with time, in a predetermined manner, or voltammetric techniques, are most attractive due to sensitivity and experimental simplicity. In these techniques, a potential difference is applied that has enough energy to promote the electron-transfer reaction, allowing current signals and potential peak values to be used to quantify and identify the compound under study, respectively (BOCKRIS; REDDY; GAMBOA-ALDECO, 2002; WANG, 2006).

Voltammetric techniques are classified according to the potential application mode, which is related to the application of potential steps, potential sweeps, and pulse potential. Amperometry is a very important technique involving steps of potential application, in which a potential is constantly applied, and the resulting current is measured over time. Among potential sweep techniques, in linear and cyclic voltammetry a potential difference is applied and the current is measured over time (WANG, 2006).

4.1 Pulse voltammetry

The emergence of pulse voltammetry, which is nothing more than a combination of potential pulse application and potential sweep techniques, culminated in a significant improvement in analytical sensitivity and selectivity due to a substantial increase in the relationship between the faradaic current (of electron transfers) and capacitive current (electric double layer) (ZOSKI, 2007). They are often much more sensitive than spectroscopic techniques or most approaches performed by chromatographic techniques. In addition, the use of pulse voltammetry can provide information about the chemical form in which a compound of interest appears, making it possible to define oxidation states, detect complexation reactions, and characterize acid-base chemistry, information often neglected by other analyzes (BOCKRIS; REDDY; GAMBOA-ALDECO, 2002; GARRY, 2004; WANG, 2006).

The basic instrumentation for using pulse voltammetry is simple, requiring an electrochemical cell, where the supporting electrolyte plus an inert electrolyte solution is added, which is composed of inorganic salts, buffer solutions, and mineral acids, which can be pure and must not be easily reduced or oxidized in the analysis potential range. The main function of the supporting electrolyte is to reduce the resistance of the solution, minimize the effects of electromigration and control the ionic forces of the medium. The electrochemical cell is generally composed of three electrodes, the working electrode, where the potential difference will be applied, and the chemical reaction will occur; the reference electrode, which has a constant potential (that is, independent of the properties of the solution) and, finally, the auxiliary electrode consisting of an inert material (generally platinum), used for current transport (WANG, 2006).

The material of the working electrode is one of the main factors to be evaluated, as it is where the redox reactions will occur, which will directly interfere with the values of the measured currents, since the target compounds change their charges on the electrode surface, exchanging one or more electrons with the conductor. The proper choice of working electrode material depends on the redox behavior of the target compound and the background current over the potential region used in the analysis. Electrical conductivity, surface reproducibility, potential range, mechanical properties, cost, availability, and toxicity are important considerations in implementing this technique (SCHOLZ, 2010; WANG, 2006).

5 PULSE VOLTAMMETRY IN FOOD ANALYSIS

In the last decades, the improvements in instrumentation and the research about materials for working electrode preparation have broadened the applicability of pulse voltammetry in different areas (MARINA FONSECA ALMEIDA; DE SOUZA, 2023; MEGALE; DE SOUZA, 2023), including food analysis (AYERDURAI; CIEPLAK; KUTNER, 2023; BASAK; VENKATRAM; SINGHAL, 2022; CHI; LIU, 2023; HOYOS-ARBELÁEZ; VÁZQUEZ; CONTRERAS-CALDERÓN, 2017; IBRAHEEM SHELASH AL-HAWARY *et al.*, 2023; MENG *et al.*, 2023; VILLALONGA *et al.*, 2022). So, the determination of chemical components in raw materials and foodstuffs has been more frequently performed using the pulse voltammetry techniques differential pulse voltammetry (DPV) and square wave voltammetry (SWV). In these techniques, the sensitivity presents similar values to that obtained by chromatographic detection, but its main advantage is simply because complex and expensive extraction steps are not necessary (CETÓ; PÉREZ; PRIETO-SIMÓN, 2022; GONÇALVES-FILHO; DE SOUZA, 2022; LIPSIKH *et al.*, 2018)

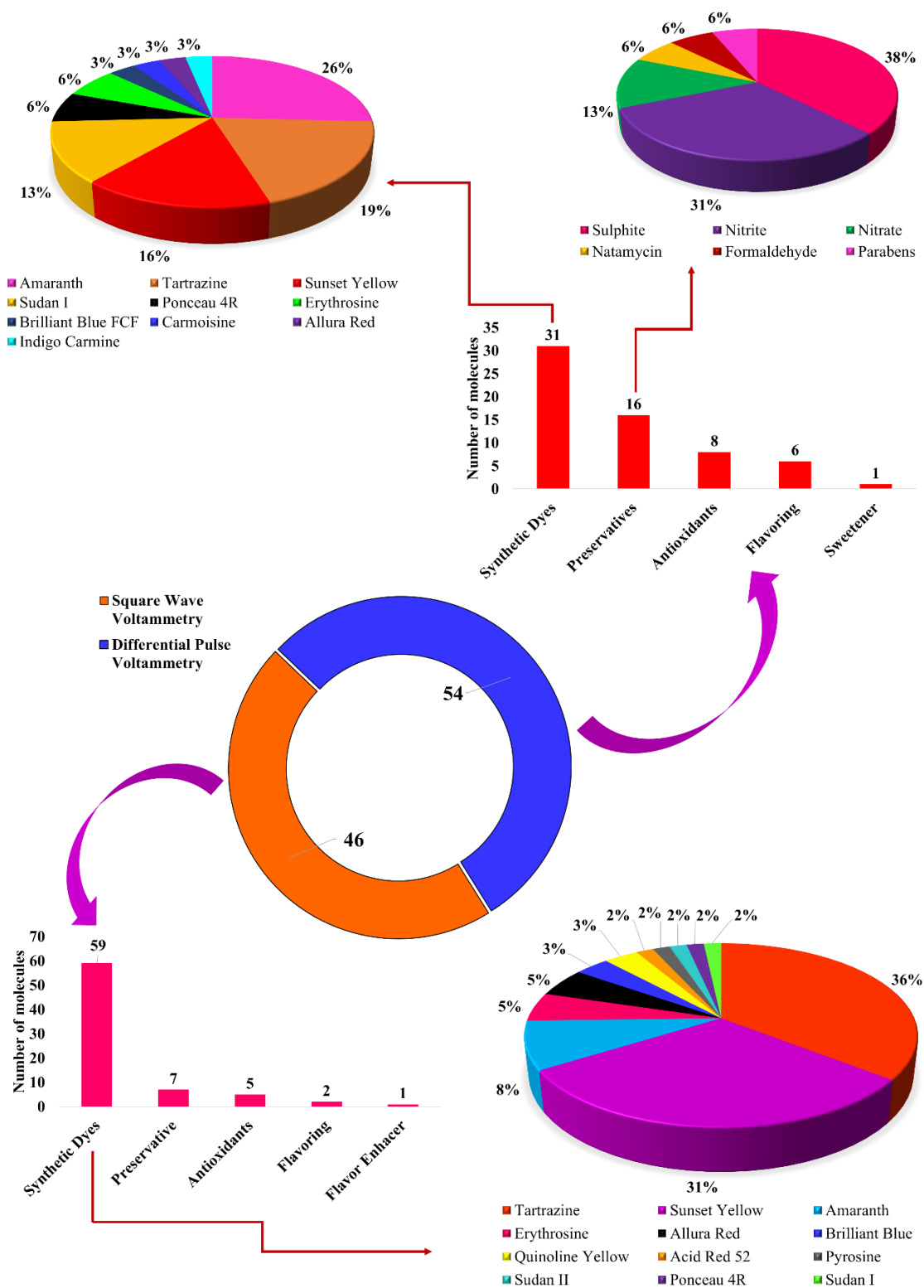
Furthermore, DPV and SWV present sensitivity over a wide range of linear concentrations, the possibility of using different electrolytes and solvents, and short analysis times. The case of evaluating and studying the kinetic parameters of the redox reactions that the analyte undergoes through the application of an electric potential pulse (SCHOLZ, 2010) also presents applicability in food analysis, mainly in antioxidant activity evaluation and the stability studies of the foodstuffs after the addition of food additives.

For food additives and other food components, the analytical signals obtained by pulse voltammetry are related to the redox reaction that occurs at the electrode/solution interface, and the reactants and/or products of the reaction can be adsorbed on the surface of the working electrode, resulting in a reduction in the intensity of the analytical signals and difficulties in its reproducibility. Therefore, the adequate choice of material for the preparation of the working electrode is the limiting factor in the success of detection. For this, various types of working electrodes have been reported, including surfaces chemically modified by nanoparticles, polymers, and carbon forms (CETÓ; PÉREZ; PRIETO-SIMÓN, 2022; LIPSIKH *et al.*, 2018), as will be presented afterward.

A systematic search was carried out in the ScienceDirect® and Scopus® databases to obtain information about the applicability of pulse voltammetry in the detection of food additives, from various classes, in foodstuffs samples. For this, the keywords "*determination of food additives by pulse voltammetric techniques*," "*dyes*" plus "*voltammetry*", "*preservatives*" plus "*voltammetry*", "*antioxidants*" plus "*voltammetry*", among other types of food additives plus "*voltammetric techniques*" were used considering all works published between January of 2012 and May of 2023.

Erro! Fonte de referência não encontrada. shows the perceptual values of published manuscripts using voltammetric techniques in food additives detection, which correspond to about 116 articles. A balance between SWV and DPV can be observed. In addition, dyes were the class with the most published studies, aiming at their determination in foods due to their toxic potential and mainly because they are widely used to make foods more appealing to children, making it very important to control their food levels.

Figure 1: Use of the square wave and differential pulse voltammetry in the detection of food additives from different classes, indicating the main compounds analyzed. Data were obtained from the scientific databases ScienceDirect® and Scopus® from 2012 to 2023.



5.1 Detection of food additives by differential pulse voltammetry

DPV is based on small amplitude pulses fixedly applied and superimposed on a linear potential ramp. The adequate pulse promotes a redox reaction ($\text{Ox} + n\text{e}^- \rightarrow \text{Red}$), allowing a current signal that is measured before and after the application of the pulse. A difference between forward and backward pulse amplitude results in a current signal with height directly proportional to the concentration of the target compound, which is plotted against the applied potential and a peak potential that can be used for its identity.

This mode of pulse potential application results in peak-shaped voltammograms with peak current values higher than that obtained by cyclic or normal pulse voltammetry due to decreased capacitive/background currents. Furthermore, the peak currents (I_p), peaks potential (E_p), and peak width at half-height ($w_{1/2}$) present relationships with the concentration of redox chemical species, diffusion coefficients, and time and intensities of pulse potential applied, and other electrochemical parameters, according to:

$$I_p = \frac{nFAD_{\text{Ox}}^{\frac{1}{2}}C_{\text{Ox}}^*}{(\pi t_p)^{\frac{1}{2}}} \left(\frac{1 - \sigma}{1 + \sigma} \right) \quad \text{Equation 1}$$

$$E_p = E^0 + \frac{RT}{nF} \ln \left(\frac{C_{\text{Red}}}{C_{\text{Ox}}} \right)^{1/2} - \frac{\Delta E_p}{2} \quad \text{Equation 2}$$

$$w_{1/2} = 3.52 \frac{RT}{nF} \quad \text{Equation 3}$$

where n is the number of electrons involved in the half-reaction given in equivalents per mole, F is the Faraday constant and presents the 96.487 C eq^{-1} , A is the electrode area shown in cm^2 , D_{Ox} is the diffusion coefficient for the species Ox given in $\text{cm}^2 \text{ s}^{-1}$, $\sigma = \exp(nFa/2RT)$ and a is the pulse amplitude, R is the gas constant and present of value $8.3143 \text{ J C K}^{-1} \text{ mol}^{-1}$, T is the absolute temperature in Kelvin, t_p is pulse potential time, E^0 is the standard potential given in volts, and C_{Red} and C_{Ox} are the concentration of chemical species in the reduced and oxidized forms provided in mol L^{-1} .

A suitable optimization of the supporting electrolyte and pH of the solution can result in alterations in the diffusion coefficient and, consequently, in the I_p values related to the sensitivity of the analysis. Besides, the proper choices of pulse amplitude

and time of the pulse duration can result in improvements in the E_p and $w_{1/2}$ related to selectivity in the analysis. For this, a previous optimization of the experimental and voltammetric parameters is necessary for each electroactive compound, considering the use of different working electrode materials. So, DPV has been frequently used in food additives detection in foodstuffs and beverages.

Among these, vanillin flavor detection has been frequently performed by DPV using varied types of working electrode materials. Nixon et al. (2023) used a chemically modified electrode and a previous choice of supporting electrolyte and pH of the medium, which has a significant impact both on the electrochemical behavior of the vanillin molecule during electrochemical oxidation and on the electrochemical performance of the sensor, composed by glassy carbon electrode (GCE) modified by lanthanum nickelate spheres embedded acid functionalized carbon nanofiber composite. DPV resulted in an anodic response associated with the oxidation of the vanillin due to the loss of two electrons and one proton ($- 2e^- / - H^+$) from the aromatic ring. So, in phosphate buffer solution pH 7.0, calibration curves were constructed allowing two linear ranges, from 5 to 235 $\mu\text{mol L}^{-1}$ and from 235 to 3035 $\mu\text{mol L}^{-1}$ with a calculated LOD of 6.12 nmol L^{-1} . The procedure was applied to chocolate and ice cream samples, and the results obtained were compared with HPLC analysis, where no variations were observed in the results obtained through any of the approaches, demonstrating the applicability of the DPV methodology.

Vanillin also was determined by Kumari et al. (2023) using DPV and a nanocomposite prepared with zinc and manganese. The effect of the electrolyte on the system was investigated, and it was found that the peak current decreases as the pH becomes more basic. In this study, was used a 0.1 mol L^{-1} phosphate buffer solution at pH 6.0, with an increase linear potential in a step of 5 mV s^{-1} . After experimental and voltammetric optimization, analytical curves presented a linear range of 20 - 120 $\mu\text{mol L}^{-1}$, allowing a low detection limit of 120 $\mu\text{mol L}^{-1}$. DPV methodology was applied in ice cream and presented an excellent performance in the determination of flavor.

Nehru, Chen and Dong (2023) evaluated the electrochemical performance of a voltammetric sensor based on graphite carbon nitride nanotubes decorated with molybdenum disulfide composite for vanillin determination. The methodology was optimized at 0.05 mol L^{-1} phosphate buffer solution and 50 mV s^{-1} of pulse potential variation, allowing in a linear range from 0.005 to 458.9 $\mu\text{mol L}^{-1}$ and LOD 4 $\mu\text{mol L}^{-1}$. In addition, the practical applicability of the DPV and the proposed sensor was also

successfully evaluated in the determination of vanillin in ice cream, obtaining good recoveries of vanillin and demonstrating that the quantitative determination of the proposed electrochemical sensor shows real-time applicability for the detection of vanillin.

Nitrite was determined by Faisal et al. (2023) using DPV allied to electrode modified by gold nanoparticles and polymeric films, which exhibited linear electrochemical responses to nitrite detection over a range from 1.5 to 22.5 $\mu\text{mol L}^{-1}$ in pH 7.0, the limit of detection (LOD) 1.11 $\mu\text{mol L}^{-1}$ that is appreciable. Other parameters, reproducibility, and response time were properly investigated and found to be reliable. In addition, the sensor revealed its consistent performance in the analysis of water samples.

Li et al. (2023) created a non-enzymatic electrochemical sensor based on methyl red electrodes and modified carbon paste combined with DPV that can measure nitrite in food samples. They obtained a linear range from 0.0 to 7000 $\mu\text{mol L}^{-1}$ from analytical curves constructed from the standard addition method, allowing a LOD of 0.011 $\mu\text{mol L}^{-1}$. In the present study, the accuracy and applicability of the proposed electrochemical nitrite sensor for the determination of nitrite in prepared meat specimens were examined. The results showed good recovery values (greater than 96.00%) and adequate precision (relative standard deviation less than 5.10%), as well as good agreement between amperometric measurements and the Test Kit for Nitrate assays. These findings demonstrated that SWV and the proposed sensor can be used as reliable nitrite sensors to track nitrite levels in food samples.

Nitrites and sulfites were detected by Ferlazzo et al. (2023) using DPV and an electrochemical sensor based on nano biochar, which was obtained from hydrochar derived from orange peel residue synthesized by hydrothermal carbonization at 180 °C. The hydro char starting material was deposited on a commercial screen-printed carbon electrode (SPCE) and its electrochemical characteristics were investigated the sensor has better electrochemical properties than the pure SPCE sensor for the detection of sulfites in wines and nitrites in water, showing two well-defined and separated anodic peaks at + 0.45 and + 0.67 V, respectively. Linear correlations between anodic peaks and concentration of sulfites and nitrites were found up to 3200 $\mu\text{mol L}^{-1}$ and 1400 $\mu\text{mol L}^{-1}$, respectively. This study demonstrated, for the first time, the promising capacity of nano biochar derived from orange peel residues for the simultaneous electrochemical oxidation of sulfites and nitrites in complex samples.

Singh & Singh (2023) used DPV and an electrochemical sensor prepared from electro polymerization of a thin film of ortho-phenylenediamine in the determination of acesulfame-K, artificial sweeteners. Analysis was carried out to validate the methodology, in which the electrode showed a dynamic linear range from 0.1 to 17.0 $\mu\text{mol L}^{-1}$, LOD, and limit of quantification (LOQ) of 0.35 $\mu\text{mol L}^{-1}$ and 1.2 $\mu\text{mol L}^{-1}$, respectively, with good selectivity, repeatability, and stability. Commercial potential for acesulfame-K detection was evaluated by use of the proposed methodology for the determination of acesulfame-K in samples of cola drinks, sweets, and tabletop sweeteners that were fortified with aliquots of acesulfame-K by the standard addition method. The obtained results demonstrated that the methodology is sensitive and selective for the determination of acesulfame-K in food samples, in addition, the sensor was highly selective and specific and unaffected by the matrix effect, and the samples do not require any pre-treatment, demonstrating the robustness of the proposed methodology.

The sensitivity in DPV detection of food additives can be improved using previous steps of preconcentration, or stripping voltammetry, in which a suitable potential and time, the food additive can be preconcentrate from the solution into or onto the working electrode before DPV analysis. So, azo dyes, toxic food additives, have been evaluated using stripping steps due to the azo groups can be easily preconcentrate in different working electrodes.

Among this, Nguyen et al. (2023) developed a methodology for the determination of amaranth in samples of soft drinks and sweets using DPV allied to preconcentration steps in a glassy carbon electrode modified with reduced graphene oxide. The optimum conditions used to determine this dye were Britton-Robinson buffer solution with pH 3.0, adsorption potential at 0.0 V, adsorption time of 30 seconds at a sweep rate of 20 mV s^{-1} , agitation of the solution followed by equilibrium time of 5 s and DVP analysis. So, it was observed a linear range of the peak current and concentration from 0.1 $\mu\text{mol L}^{-1}$ to 10 $\mu\text{mol L}^{-1}$ and LOD from 0.028 $\mu\text{mol L}^{-1}$. The proposed method can be applied to determine amaranth in samples of soft drinks and sweets using the standard addition method. The accuracy was evaluated using a recovery test, with recoveries efficiencies from 95% to 105%, proving an effective and sensitive analytical procedure.

Dmukhailo, Tvorynska, and Dubenska (2023) investigated the increase in the electrochemical reduction of the synthetic azo dye tartrazine using preconcentration

steps allied to DPV under three different types of solid silver amalgam electrodes (AgSAEs), in particular, the polished one (p-AgSAE), covered by mercury film (f-AgSAE) or by the meniscus of mercury (m-AgSAE), and the comparison with a hanging mercury drop electrode (HMDE). The highest peak reduction of tartrazine in AgSEAs was obtained in the Britton-Robinson buffer (pH 3.4). It has been shown that the accumulation step can considerably increase the height of the tartrazine reduction peak, allowing a simple, fast, and sensitive method for the determination of tartrazine based on its reduction in the working AgSAEs. The results showed that all three electrodes can be applied for tartrazine electroanalysis, however, the best detection limit ($0.078 \mu\text{mol L}^{-1}$) and quantification limit ($0.24 \mu\text{mol L}^{-1}$) were obtained under m-AgSAE. The developed procedures were successfully applied for the determination of tartrazine in soda, succade, and veterinary medicine using a standard addition method, resulting in a recovery from 90 to 104%. Therefore, AgSAEs are beneficial electrochemical tools for monitoring the quality and safety of food products.

Alves et al. (2022) described a simple method for the simultaneous electrochemical determination of tartrazine and sunset yellow using DPV as a voltammetric technique and an electrochemical paper-based analytical device based on graphite powder and nail polish, supported on cardboard as a voltammetric sensor. The electrochemical method was optimized and showed excellent analytical performance with low detection limits of 2.38 and $2.09 \mu\text{mol L}^{-1}$ for tartrazine and sunset yellow, respectively, with a wide linear range from 4.0 to $60.0 \mu\text{mol L}^{-1}$. In addition, the proposed method did not require a complex sample treatment step (simple dilution only) and was applied in the determination of both dyes in samples of isotonic, juice, juice powder, freeze pop, and gelatin, with satisfactory selectivity about to other food ingredients found in the analyzed samples. Thus, the disposable device proved to be an excellent alternative tool for the analysis of both dyes in food samples.

Table 1 shows some DPV detections of food additives from different classes, indicating the type of working electrode, experimental conditions, foodstuffs samples, the LOD, linear range (LR) of the DPV responses, and recovery efficiencies. These data were obtained using the methodology indicated early in Scope and Approach, considering research published from 2012 to 2023.

Table 1: Published research about the identification and quantification of food additives by differential pulse voltammetry, indicating the class and the food additive, the type of working electrode used, experimental and voltammetric conditions, type of sample in which the analysis was performed, limits of detection (LOD), the linear range of the voltammetric responses (LR) and recovery values (Rec.). This information was obtained from scientific databases in the years 2012 to 2023.

Classes	Analytes	Electrode	Experimental conditions	Sample	LOD ($\mu\text{mol L}^{-1}$)	LR ($\mu\text{mol L}^{-1}$)	Rec. (%)	Ref
Antioxidant	BHA	Polymer/GCE	pH 6.0 Phosphate Buffer $\alpha=60 \text{ mV}$ $\nu=50 \text{ mV s}^{-1}$	Edible oil	0.21	0.5 - 70	96 - 117	(XIAO <i>et al.</i> , 2022)
	TBHQ	NPs/GCE	pH 2.0 BR Buffer $\nu=50 \text{ mV s}^{-1}$	Edible Oil			99 - 108	(YUE <i>et al.</i> , 2019)
	BHA	PC/CPE	pH 7.0 Phosphate Buffer $\nu=50 \text{ mV s}^{-1}$	Potato Chips	0.11	0.33 - 110	98 - 104	(MANORANJITH AM; NARAYANAN, 2021)
	TBHQ	NPs/SPCE	pH 7.0 Phosphate Buffer	Canola Oil Flaxseed Oil Sesame Oil	0.001	0.031 - 12,5	98 - 102	(SEBASTIAN <i>et al.</i> , 2022)

Flavoring	TBHQ	GCE	pH 5.0 BR Buffer	Edible oil		91 - 110	(FARAJMAND <i>et al.</i> , 2020)
	Vanillin		pH 7.0 Phosphate Buffer	Vanilla Bean Vanilla Tea Biscuit	0.2	0.1 - 7.0 10 - 40	84 - 106 (SHANG; ZHAO; ZENG, 2014)
	Vanillin		pH 6.0 Phosphate Buffer	Ice Cream	120	20 - 120	(KUMARI <i>et al.</i> , 2023)
Preservatives	4- hexylresorcinol Preservative	MIP/CFP	pH 7.0 Phosphate Buffer	Shrimps	6.03	30 - 300	(GEORGE <i>et al.</i> , 2023a)
	Nitrite	NPs/GCE	Phosphate Buffer	Pickled vegetable Pickled beef Pickled chicken	0.082	0.4 - 20	96 - 104 (ZHANG <i>et al.</i> , 2015a)
	Nitrite	CPE	pH 4.0 Phosphate Buffer	Salami Sausage Bologna	0.09	0.5 - 10	98 - 103 (YILDIZ <i>et al.</i> , 2014)

Synthetic Dyes	Nitrite	NPs/GCE	pH 8.0 BR Buffer $\alpha=60$ mV $\nu=40$ mV s ⁻¹	Bacon Ham Salami Sausage	0.09	4 - 148	97 - 104	(WONG <i>et al.</i> , 2022)
	Sulfite	NPs/SPE	pH 7.0 Phosphate Buffer $\nu=50$ mV s ⁻¹	Orange Juice Nonalcoholic malt drink	0.08	0.6 - 400	97 - 104	(MALAKOOTIAN; HAMZEH; MAHMOUDI- MOGHADDAM, 2022)
	Nitrite	NPs/SPCE	pH 5.0 Acetate Buffer	Tap Water	0.2	1 - 500	94 - 101	(XU <i>et al.</i> , 2020)
	Nitrite		pH 4.0 Phosphate Buffer	Sausage	1.0	3.0 - 100	94 - 103	(SABER- TEHRANI <i>et al.</i> , 2013)
	Acid Red 52	MWCNT/CPE	pH 7.0 Phosphate Buffer $\alpha=60$ mV $\nu=50$ mV s ⁻¹	Water	0.03		93 - 105	(GOLESTANEH, 2020)

Sunset Yellow Tartrazine	NPs/SPCE	pH 8.0 Phosphate Buffer $\alpha=60$ mV $\nu=50$ mV s ⁻¹	Soft Drink	0.025 0.05	0.05 - 5.0 0.075 - 5.0	94 - 104	(WU; LEE, 2020)
Sunset Yellow Tartrazine		pH 2.0 BR Buffer	Isotonic Juice Powdered juice Freeze pop Gelatin	2.38 2.09	4.0 - 60	95 - 103	(FIGUEIRA ALVES <i>et al.</i> , 2022)
Amaranth	MPs/CPE	pH 7.0 Phosphate Buffer $\nu=50$ mV s ⁻¹	Grape Juice Peach Juice Apple Juice	10.2	0.05 - 240	97 - 103	(AKBARI JAVAR <i>et al.</i> , 2022)
Amaranth	NPs/GCE	pH 3.0 BR Buffer $\nu=20$ mV s ⁻¹	Strawberry Juice Tic tac candy Raspberry Juice	0.028	0.1 - 1.0	95 - 105	(KIM THUONG NGUYEN <i>et al.</i> , 2023)
Sunset Yellow	MIP/GCE	pH 7.0 Phosphate Buffer $\nu=50$ mV s ⁻¹	Fruit Juice Mirinda Drink Orange Juice	0.004	0.01 - 1.4 1.4 - 16	95 - 107	(ZHAO; ZHAO; ZENG, 2014)

Sunset Yellow Tartrazine	Graphene/GCE	pH 5.0 Phosphate Buffer $\nu=100 \text{ mV s}^{-1}$	Orange powderer drink	0.0014 0.0012	0.05 - 20	97 - 101	(YE <i>et al.</i> , 2013)
Sunset Yellow	NPs/GCE	pH 4.0 Phosphate Buffer	Soft drink	0.002	0.002 - 109	93 - 98	(WANG <i>et al.</i> , 2015a)
Amaranth		pH 6.0 Acetate Buffer	Fruit drinks	0.05	0.05 - 50	93 - 101	(HAN <i>et al.</i> , 2014)
Tartrazine	MIP/GCE	pH 7.0 Phosphate Buffer $\nu=50 \text{ mV s}^{-1}$	Fanta drink Mirinda drink Orange powder	0.008	0.03 - 5.0 5.0 - 20	96 - 108	(ZHAO; ZENG; ZHAO, 2014)
Amaranth	Pyrolytic/SPE	pH 7.0 Phosphate Buffer	Orange juice Apple juice	0.01	0.03 - 290	97.5 - 103	(BEITOLLAHI <i>et al.</i> , 2022)
Tartrazine Sunset Yellow Brilliant blue	BDDE	pH 2.0 BR Buffer $a=60 \text{ mV}$ $\nu=20 \text{ mV s}^{-1}$	Powdered Juice Gelatin	0.006 0.0013 0.02	0.005 - 0.1	90 - 111	(MEDEIROS <i>et al.</i> , 2012)

Sunset Yellow Tartrazine	NPs/SPCE	pH 6.0 Phosphate Buffer $\alpha=60$ mV $\nu=50$ mV s ⁻¹	Beverages	0.0005 0.045	0.01 - 20 0.02 - 20	80 - 117	(JAMPASA <i>et al.</i> , 2016)
Erythrosine	NPs/CPE	pH 5.9 Phosphate Buffer $\alpha=25$ mV $\nu=100$ mV s ⁻¹	Gelatine Candy Smarties	0.002	0.1 - 1.0 1.0 - 100		(ARVAND; POURHABIB; ASADI, 2018)
Pyrosine	NPs/GCE	0.1M HCl		5.98	21 - 598		(WANG <i>et al.</i> , 2013)
Amaranth Tartrazine Quinoline yellow	GCE	pH 4.0 Phosphate Buffer $\alpha=25$ mV $\nu=50$ mV s ⁻¹	Red Grapes Orange	0.5 1.0 0.25		101 - 104	(GHANBARI <i>et al.</i> , 2019)
Sudan II	NPs/CPE	pH 4.0 Phosphate Buffer $\nu=106$ mV s ⁻¹	Chili Sauce Ketchup Sauce	0.0017	0.1 - 1.0 1.0 - 2.0	95 - 102	(HEYDARI; GHOREISHI; KHOABI, 2019)

Sunset Yellow	NPs/CBE	$\nu=100 \text{ mV s}^{-1}$	Jelly Soft Drink Ice cream Candy	0.9	0.12 - 108	96 - 102	(BALRAM <i>et al.</i> , 2022a)
Sunset Yellow Tartrazine	NPs/CPE	Phosphate Buffer $a=60 \text{ mV}$ $\nu=60 \text{ mV s}^{-1}$	Orange juice Fruit Juice	0.03 0.002	0.05 - 1.6	96 - 104	(GHOREISHI; BEHPOUR; GOLESTANEH, 2012)
Ponceau 4R Tartrazine	Metal/CPE	pH 4.0 Acetate Buffer $a=40 \text{ mV}$ $\nu=50 \text{ mV s}^{-1}$	Drinks	0.08 2.0	0.005 - .014		(ZHANG <i>et al.</i> , 2015b)
Sunset Yellow	CPE	pH 4.0 Acetate Buffer $a=50 \text{ mV}$ $\nu=40 \text{ mV s}^{-1}$	Drink	0.0007	0.002 - 0.2		(SONGYANG <i>et al.</i> , 2015)
Sunset Yellow Tartrazine	NPs/GCE	pH 5.0 Phosphate Buffer $\nu=50 \text{ mV s}^{-1}$	Orange Juice Fruit Juice Peach Gelatin	0.03 0.01	0.1 - 3.0 0.07 - 1.86	95 - 101	(DORRAJI; JALALI, 2017)

Tartrazine	MIP/CFE	pH 4.0 Phosphate Buffer $\alpha=50$ mV $\nu=30$ mV s ⁻¹	Saffron Powder Juice	0.00002	0.0006 - 0.16	98 - 102	(GEORGE <i>et al.</i> , 2023b)
Erythrosine	HMDE	pH 7.5 BR Buffer	Sprite Marinda Lemon Shani Cade	0.18	0.003 - 0.07	86 - 101	(AHMAD <i>et al.</i> , 2023)
Tartrazine	Graphite/GCE	pH 4.6 Acetate Buffer $\alpha=50$ mV $\nu=40$ mV s ⁻¹	Soft Drinks	0.0028			(SONG <i>et al.</i> , 2014)
Allura red		pH 3.0 Phosphate Buffer	Strawberry	0.008	0.05 - 10	95 - 97	(YU <i>et al.</i> , 2016)
Erythrosine	Glucose/CPE	pH 11.2 Phosphate Buffer	Fruit Juice	0.021	0.1 - 10	82 - 98	(NAYAK; SHETTI, 2016)

Sunset Yellow Tartrazine	Metal/CPE	pH 8.0 Phosphate Buffer $\alpha=50$ mV $\nu=40$ mV s ⁻¹	Soft Drink	0.00005 0.00014	0.0003 - 50 0.001 - 0.01		(Jl <i>et al.</i> , 2016)
Sunset Yellow	MIP/GCE	pH 4.5 Acetate Buffer	Candy Peach Juice Beverage	0.005	0.05 - 100		(ARVAND; ZAMANI; SAYYAR ARDAKI, 2017)
Tartrazine	Polymer/GCE	pH 7.0 Phosphate Buffer	Chips Candy Jelly Soft Drink	0.032	0.1 - 206	98 - 101	(SAKTHIVEL <i>et al.</i> , 2018)
Sunset Yellow Tartrazine		pH 5.0 Acetate Buffer	Orange Sweetly Green Sweetly Cocktail Margarita	0.002	0.005 - 0.25	94 - 109	(KOLOZOF <i>et al.</i> , 2020)
Sunset Yellow Quinoline Yellow	NPs/GCE	pH 5.0 Acetate Buffer $\alpha=15$ mV $\nu=30$ mV s ⁻¹		0.086 3.4	0.3 - 3.0 12 - 20	93 - 106	(BLANCO <i>et al.</i> , 2020)

Sunset Yellow Tartrazine	MWNTfilm/GCE	pH 8.0 Phosphate Buffer $\alpha=50$ mV $\nu=40$ mV s ⁻¹	Orange Juice Fruit Juice	0.188 0.022		91 - 96	(ZHANG <i>et al.</i> , 2009)
Sunset Yellow Sudan I	Graphene/GCE	pH 3.0 pH 7.0 Citrate buffer	Soft Drink Chili Powder	0.1	0.1 - 800 0.1 - 1000	93 - 109	(SUN <i>et al.</i> , 2023)
Tartrazine	NPs/CPE	pH 3.0 Phosphate Buffer	Orange Powder Cool Drink	0.08	0.35 - 5.44	99 - 103	(KARIM-NEZHAD <i>et al.</i> , 2017)
Sunset Yellow Tartrazine	NPs/GCE	pH 6.0 Phosphate Buffer	Orange Juice Jelly	0.0024 0.0086	0.01 - 3.0 0.03 - 6.0	94 - 108	(DENG <i>et al.</i> , 2016)
Tartrazine	Polymer/CPE	pH 7.0 Phosphate Buffer		0.28	1 - 27 35 - 87	94 - 98	(MANJUNATHA, 2018)
Allura Red Tartrazine		pH 4.0 BR Buffer	Soft Drink Gelatin Candy Isotonic Drink	0.3	1.0 - 5.0		(RODRÍGUEZ <i>et al.</i> , 2015)

	Allura Red Brilliant Blue	MWCNT/GCE	pH 7.0 Phosphate Buffer	Isotonic Drink	91 - 130	(SIERRA- ROSALES <i>et al.</i> , 2019)
	Sunset Yellow Tartrazine	Polymer/GCE	pH 7.0 Citrate buffer	Beverage Juice Powder Sugar tablets	95 - 101	(CHAO; MA, 2015)
Flavor	Glutamate	Glutamate dehydrogenase/GC E	pH 7.0 Phosphate Buffer	57	0.1 - 500	(GHOLIZADEH <i>et al.</i> , 2012)

Legend: a: Pulse Amplitude; u: scan increment; MWCNT: multiwalled carbon nanotube composite; GCE: glassy carbon electrode. MIP: Molecularly Imprinted Polymers; CPE: carbon paste electrode; SPCE: screen-printed carbon electrode; ePAD: electrochemical paper-based analytical device; /CFP: carbon fibre paper electrode; MIES: magnetic molecularly imprinted electrochemical sensor; HMDE: Hanging mercury drop electrode.

5.2 Detection of food additives by square wave voltammetry

SWV involves the application of a series of pulses of potential with equal amplitude superimposed on a staircase of potential. During the forward pulse, the species of interest diffuses to the electrode surface and are immediately reduced or oxidized. During the backward pulse, it analyzes whether it was just oxidized or reduced and returns to the initial condition in a reversible reaction or no reaction occurs if the system is irreversible (MIRCESKI; KOMORSKY-LOVRIC; LOVRIC, 2007). Current values are measured just before, called forward current (I_f), and at the end of each pulse, called backward current (I_b). The net or resultant currents (ΔI) are plotted against the corresponding potential of the staircase waveform, allowing a voltammogram with a Gaussian profile and intensities defined by:

$$\Delta I = I_f - I_b \quad \text{Equation 4}$$

The mode of pulse potential in SWV results in a suitable reduction in the contribution of residual capacitive current and, consequently, intense improvements in the sensitivity for detection of redox compounds (DE SOUZA *et al.*, 2004; DE SOUZA; MACHADO; AVACA, 2003). Like DPV, the maximum point of the signal on the "x" axis is called I_p , from this point, drawing a line parallel to the x-axis, the E_p on the "y" axis is obtained. E_p and I_p are used to identify and quantify the redox compounds. In practice, resultant currents are more significant than the forward or backward currents, and the analytical signals are generally higher, giving SWV very low detection limits.

For this, various SWV methods were developed and applied to detect food additives, allowing procedures with low cost, simplicity, selectivity, high sensitivity, and compactness of the equipment. Table 2 shows some specific applications of the SWV procedure in the detection of food additives from various classes and different foodstuffs samples, indicating the type of working electrode used and the experimental and voltammetric parameters previously optimized.

Table 2: Published research about the identification and quantification of food additives by square wave voltammetry, indicating the class and the food additive, the type of working electrode used, experimental and voltammetric conditions, type of sample in which the analysis was performed, limits of detection (LOD), the linear range of the voltammetric responses (LR) and recovery values (Rec.). This information was obtained from scientific databases in the years 2012 to 2023.

Classes	Analytes	Electrode	Experimental conditions	Sample	LR ($\mu\text{mol L}^{-1}$)	LOD ($\mu\text{mol L}^{-1}$)	Rec (%)	Ref
Antioxidant	TBHQ BHA	MIP-MWCNT	0.15 mol L ⁻¹ H ₂ SO ₄ $a=100$ mV $\Delta E_s=25$ mV $f=10$ s ⁻¹	Soybean oil Mayonnaise Margarine	2.84-150 1.66-150	0.85 0.50		(DOS SANTOS MORETTI <i>et al.</i> , 2016)
	BHA BHT PG	Pt-UME	$a=20$ mV, $\Delta E_s=5$ mV $f=20$ s ⁻¹	Edible Oil			95-109 93-109 92-106	(BLANDON-NARANJO <i>et al.</i> , 2022)
	TBHQ BHA	NPs/GCE	pH 3.0 Phosphate buffer $a=25$ mV $\Delta E_s=40$ mV $f=10$ s ⁻¹	Edible Oil		0.1 0.05		(GAN <i>et al.</i> , 2016)
	BHT	Pt-UME	0.1 M H ₂ SO ₄	Corn		5.7 7.8	92-102	(ROBLEDO <i>et al.</i> , 2014)

Sweetener	Aspartame	NPs/GCE		Beverage	15.0 - 27.0	12.3		(BALGOBIND <i>et al.</i> , 2016)
	Vanillin	BDDE	pH 2.5 Phosphate buffer $a=40$ mV $\Delta E_s=12$ mV $f=100$ s ⁻¹	Turkish milk Pudding Almond flour	3.3 - 98	0.16	91.3-86.1	(YARDIM; GULCAN; ŞENTURK, 2013)
Flavoring	Vanillin	Polymer/GCE	pH 4.0 Acetate buffer $a=100$ mV $\Delta E_s=8$ mV $f=10$ s ⁻¹	Vanilla Sugar	10 - 450	0.06	98.5 - 99.8	(FILIK; AVAN; MÜMIN, 2017)
	Vanillin	BDDE	pH 2.5 Phosphate buffer $a=70$ mV $\Delta E_s=14$ mV $f=50$ s ⁻¹	Vanilla Sugar		1.54		(ALI <i>et al.</i> , 2017)

Preservatives	Vanillin	Metal/CPE	pH 6.0	Chocolate Biscuit Coffee	0.03 - 800	0.009	(CHERAGHI; TAHER; KARIMI-MALEH, 2017)
	Vanillin	Pt electrode	pH 7 Phosphate buffer $a=50\text{ mV}$, $\Delta E_s=10\text{ mV}$ $f=10\text{ s}^{-1}$	Vanilla sugar	0.05 - 0.43	0.019	(FORT; COBZAC; TURDEAN, 2022)
	Vanillin	NPs/GCE		Biscuit	2.0 - 100.0		(HUANG <i>et al.</i> , 2014)
	Sulfite	NPs/CPE	pH 5.0 Acetate buffer	White Wine	2.0 - 70	0.98	(TAGUEU MASSAH <i>et al.</i> , 2021)
	Sulfite	SPCE	pH 4.0 Phosphate buffer $a=75\text{ mV}$, $\Delta E_s=7\text{ mV}$ $f=14\text{ s}^{-1}$	Wine	5.0 - 80	16.5	(LOZER <i>et al.</i> , 2023)

Natamycin	BDDE	0.05 M H ₂ SO ₄ $a=25$ mV, $\Delta E_s=10$ mV $f=50$ s ⁻¹	Wine	0.98 - 1.16	0.031	96-99	(MIELECH- ŁUKASIEWICZ; LEONIUK, 2020)
Sulfite	Cooper electrode	0.1 M NaOH $a=50$ mV, $\Delta E_s=6.0$ mV $f=50$ s ⁻¹	Tap Water Grape Juice Coconut Water Cashew Juice	25.0 - 100	4.0	113-90	(ARAÚJO <i>et al.</i> , 2021)
Nitrite	NPs/GCE	pH 2.0 BR buffer $a=70$ mV, $\Delta E_s=10$ mV $f=10$ s ⁻¹	Beef Sausages Chicken Sausages Lunchmeat Smoked meat	6.2-125	1.89	95.4	(BERISHA <i>et al.</i> , 2020)
Nitrate Nitrite	Polymer/CPE	pH 3.0 BR buffer $a=100$ mV, $\Delta E_s=6.0$ mV $f=50$ s ⁻¹	Mineral Water Sausage Hot-dog Sausage Salami Cheese	6.0 - 110	0.62 87.0		(AFKHAMI <i>et al.</i> , 2012)

Formaldehyde	GCE	pH 5.5 Acetate buffer $a=30$ mV, $\Delta E_s=5.0$ mV $f=50$ s ⁻¹	Mushrooms		4.32	87-108	(PINTO <i>et al.</i> , 2019)
Sulphite	CPE	pH 1.0 BR buffer	White wine Coconut water Fruit juice	25 - 500	16.0	95-104	(SILVA; TAKEUCHI; SANTOS, 2015)
Nitrate Nitrite	CPE	pH 7.0 Phosphate buffer $a=50$ mV, $\Delta E_s=5.0$ mV $f=8.0$ s ⁻¹	Salami Sausage	1 - 10	8.0 0.95		(TURDEAN; SZABO, 2015)
Nitrite	NPs/CPE	pH 4.0 BR buffer $a=10$ mV, $\Delta E_s=5.0$ mV $f=30$ s ⁻¹	Water	0.05 - 250.0	11.2		(AFKHAMI <i>et al.</i> , 2014)
Sulfites	CBE	pH 7.4 Phosphate buffer	Rice Wine	0.008 - 1.0	6.0	88-99.5	(XU <i>et al.</i> , 2016)

Synthetic Dyes	Parabens	GCE	pH 4.5 Phosphate buffer $a=30$ mV, $\Delta E_s=8$ mV $f=35$ s ⁻¹	Sweetener	0.78 - 4.48	0.29 0.38 0.36 0.39	82.6- 121.8	(DANTAS <i>et al.</i> , 2021)
	Nitrite	NPs/CPE	pH 4.0 Phosphate buffer $a=12$ mV, $\Delta E_s=2.0$ mV $f=25$ s ⁻¹	Sausage	0.5–50 mg_L ⁻¹	0.12		(ÜZER <i>et al.</i> , 2016)
	Amaranth	NPs/CPE	pH 7.0 Phosphate buffer	Beverages	0.008 - 550	0.003		(SHEIKHSHOAIE <i>et al.</i> , 2017)
	Brilliant Blue Tartrazine	Graphite/CPE	pH 2.0 Phosphate buffer $a=25$ mV, $\Delta E_s=4$ mV $f=15$ s ⁻¹	Soft drink	0.005 - 4.0 0.01 - 1.0	0.002 0.003	99.3 - 101.9	(WANG <i>et al.</i> , 2015e)
	Sunset Yellow Tartrazine	NPs/GCE	pH 4.4 Acetate buffer	Soft Drink Candy Royal Jelly	0.0062 - 6.2	0.001	98 - 101	(GAN <i>et al.</i> , 2013b)

Ice Cream							
Amaranth Tartrazine	SPCE	pH 7.0 Phosphate buffer	Orange juice Soft drink		30.0 60.0		(NUÑEZ-DALLOS <i>et al.</i> , 2018)
Sunset Yellow Tartrazine	NPs/GCE	pH 7.0 BR buffer	Carbonated beverage	0.004 - 1.0 0.007 - 1.5	0.0005 0.0008	97 - 106	(WANG; ZHAO, 2015)
Carmoisine	BDDE	pH 2.0 Phosphate buffer $a=70$ mV, $\Delta E_s=10$ mV $f=10$ s ⁻¹	Candy	0.059 - 1.31 0.01 - 0.079	7.0 3.0	105	(MICHELETTI <i>et al.</i> , 2020)
Allura red	GCE	pH 4.0 Phosphate buffer $a=70$ mV, $\Delta E_s=10$ mV $f=10$ s ⁻¹	Soft Drink	0.039 - 0.91	0.0091	88 - 108	(SILVA; WONG; FATIBELLO-FILHO, 2020)
Amaranth Ponceau 4R	Polymer/CPE	pH 7.0 BR buffer $a=40$ mV, $\Delta E_s=5$ mV $f=10$ s ⁻¹	Fruit drink	0.005 - 0.5 0.008 - 0.01	0.0005 0.001		(WANG <i>et al.</i> , 2015c)

Erythrosine	CPE	pH 11.2		100 - 0.1	0.022		(NAYAK; SHETTI, 2016)
Sudan I	NPs/GCE	$a=25\text{ mV}$, $\Delta E_s=4.0\text{ mV}$ $f=15\text{ s}^{-1}$	Chili powder Ketch up	40 - 5000 20 - 0.7	0.01		(THOMAS <i>et al.</i> , 2015)
Sudan I	NPs/GCE	pH 7.0 BR buffer	Red Chili Tomato sauce Apple juice Grape juice	0.001 - 1.0	0.00005		(WANG <i>et al.</i> , 2015b)
Sunset Yellow	Si-CPE	pH4.2 BR buffer		0.7 - 50	0.001		(REDDY <i>et al.</i> , 2019)
Amaranth	GCE	pH 7.0 BR buffer	Soft drink	0.0005 - 4.0	0.0001	105-95	(WANG <i>et al.</i> , 2015d)
Sudan I	HMDE	pH 12.3 Phosphate buffer	Orange energy drinks	0.002 - 1.1	0.006		(GÓMEZ <i>et al.</i> , 2016)
Sunset Yellow Tartrazine	TiO ₂ -CPE	0.1 M H ₂ SO ₄	Candy Royal Jelly Ice cream Juice Powder Soft Drink	0.02 - 2.05 0.02 - 1.18	0.006 0.008	96-102	(GAN <i>et al.</i> , 2013a)

Sudan I	NPs/CPE	pH 7.0 Phosphate buffer	Chilli Sauce Chilli Powder Tomato Sauce Strawberry Sauce	0.008 - 600	0.003		(ELYASI; KHALILZADEH; KARIMI-MALEH, 2013)
Ponceau 4R Amaranth	EGPE/GCE	pH 7.0 BR buffer $a=25$ mV, $\Delta E_s=4.0$ mV $f=10$ s ⁻¹	Grape Juice	0.06 - 4.0 0.08 - 4.0	0.002 0.036	94.2- 107.2	(ZHANG <i>et al.</i> , 2014b)
Amaranth	NPs/GCE	pH 6.0 BR buffer	Fanta Drink Milinda Drink Minute Maid	0.008 - 1.2	0.023		(ZHANG <i>et al.</i> , 2022)
Sunset Yellow	Purpald®/GCE	$a=100$ mV $\Delta E_s=6$ mV $f=38$ s ⁻¹	Beverage	0.005 - 0.15	0.001	101.9- 104.7	(TABANLIGIL CALAM; TAŞKIN ÇAKICI, 2023)
Erythrosine	Glucose-CPE	pH 11.2 Phosphate buffer		0.1 - 100	0.02	91.6-98.0	(NAYAK; SHETTI, 2016)
Indigo Carmine	BDDE	0.3 M H ₂ SO ₄	Tap Water	0.5 - 84.1	0.058	86-100	(SILVA <i>et al.</i> , 2016)

Amaranth	GNM/GCE	pH 7.0 Phosphate buffer	Chocolate beans Fruit Drinks	0.005 - 1.0	0.0007	96.6- 100.4	(WANG <i>et al.</i> , 2018)
Amaranth Tartrazine	SPCE	pH 3.2 Phosphate buffer $a=10$ mV, $\Delta E_s=10$ mV $f=15$ s ⁻¹	Kola soft drink Orange jelly Soft drink powder	0.15 - 1.20 0.15 - 0.80	0.03 0.07	93-104	(PERDOMO <i>et al.</i> , 2017)

Legends: a : Pulse Amplitude; ΔE_s : scan potential increment; f : Frequency; MIP: Molecularly Imprinted Polymers; MWCNT: Multi-Walled Carbon Nanotubes; NP: Nanoparticles; GCE: Glassy Carbon Electrode; UME: Ultra Micro Electrode; CPE: Carbon paste electrode; BDDE: Boron-doped diamond electrode; CBE: Carbon-black Paste Electrode.

The publications evaluated in this review indicated the predominance in the detection of dyes, mainly azo dyes, that are a synthetic and very toxic class of food additives. These compounds have the azo chemical group ($R-N=N-R'$) that connects two aromatic rings and other substituents, which can be easily reduced and/or oxidized, allowing in suitable voltammetric signals for identification and quantification, even in complex samples, such as foodstuffs. Dyes do not pose a problem when used in the daily intake amounts, however, they harm human health when consumed excessively. Among these, sunset yellow consumption results in a long-term accumulation in the body, it can even cause severe liver and kidney damage. So, a rigorous control of quality by food industries is required and some researchers have been developed indicating the applicability of SWV in dyes detection.

Tabanlıgil Calam & Taşkın Çakıcı (2023) developed a SWV procedure for detection of sunset yellow under a glassy carbon electrode modified by polymeric films. Optimal experimental ($HClO_4$ as supporting electrolyte), and voltammetric (frequency of 37.609 s^{-1} , potential step of 5.507 mV , and pulse amplitude of 99.602 mV) conditions were previously optimized. Three linear ranges were obtained for sunset yellow concentrations (4.97 to $1.18\text{ }\mu\text{mol L}^{-1}$, $1.18\text{ }\mu\text{mol L}^{-1}$ to $15.16\text{ }\mu\text{mol L}^{-1}$, and 15.16 to $0.153\text{ }\mu\text{mol L}^{-1}$). The LOD value found was 1.15 nmol L^{-1} and the sunset yellow concentration was determined with satisfactory results in samples of powdered instant drinks and orange-flavored carbonated drinks. Already Wu et al. (2023) used SWV allied to molecularly imprinted electrochemical sensors for the sensitive detection of sunset yellow. The methodology was developed using phosphate buffer solution pH 7.4 and a wide linear range from 10 nmol L^{-1} to $100\text{ }\mu\text{mol L}^{-1}$ and a low LOD of 1.60 nmol L^{-1} was obtained, and the application in fortified food samples allowed recoveries efficiencies from 94.0 to 97.0%, as well as relative errors from 5.4 to 8.3%, revealing its promising potential in detecting this color.

Flavor food additives are largely used in foodstuffs, beverages, candies, and ice creams. Among these, vanillin if used in excess by the consumer can results in allergic reactions, migraine headaches, and may result in food obsession. This is necessary for rigorous control of quality in foodstuffs production. In this context, Fort; Cobzac; Turdean (2022) developed a square-wave voltammetry procedure allied to mathematical treatment for data processing of the analytical responses of vanillin under a platinum electrode. It observed that the oxidation reaction is irreversible and controlled by adsorption, typical of the redox reaction that occurs in phenolic

derivatives. Using 0.1 mol L⁻¹ phosphate buffer pH 7.0 plus 0.1 mol L⁻¹ NaCl and 1% ethanol as supporting electrolyte, the experimental SWV parameters were optimized as pulse frequency of 10 s⁻¹, pulse amplitude of 0.05 V and scan potential increment of 0.01 V were constructed calibration curves, which presented linear responses in the range of 50 and 430 nmol L⁻¹ of vanillin with LOD 19 nmol L⁻¹. The methodology was applied in the detection of vanillin in samples of vanilla sugar and vanilla essence and the obtained results demonstrated that the methodology had a satisfactory analytical performance and can be used for the determination of vanillin in foods variety of products (sugar, essences, pudding).

Yardim et al. (2013) developed a methodology for the determination of food additive vanillin using SWV allied to boron-doped diamond electrode pre-treated anodically in aqueous solutions and preconcentration steps, allowing a well-defined voltammetric response in phosphate buffer, pH 2.5. Analytical curves were constructed obtaining a linear range from 33.0 to 980 µmol L⁻¹ with a LOD of 0.16 µmol L⁻¹. The practical applicability of the proposed method was tested for the determination of this flavoring agent in commercial pudding powder (a Turkish milk pudding with almond flour). Results presented analytical parameters like data obtained using other electrode surfaces previously reported in the literature.

Furthermore, the BDD electrode exhibited a more sensitive electrochemical response to vanillin oxidation than the others, except carbon paste prepared with diphenyl ether as the paste liquid (COOKEAS; EFSTATHIOU, 1992) and modified vitreous carbon electrodes (ZHENG *et al.*, 2010). However, the results obtained by this method from the literature showed that the linearity range of the calibration chart could only be expanded to a concentration about five times lower than that obtained by the proposed method. On the other hand, in the case of the modified vitreous carbon electrode, its sensitivity is approximately four times greater than the BDD electrode. It is important to point out that the preparation of modified electrodes is not convenient and can sometimes be complicated. Based on the above, it can be seen that a great advantage of the BDDE use, in addition to the adequate analytical sensitivity, was its simplicity, which enables its use without the need for a procedure to modify the electrode surface. The use of BDD also minimizes the requirement for organic solvents and therefore makes the proposed method more environmentally friendly than the more commonly reported electroanalytical methods.

Among other food additives, tert-butylhydroquinone (TBHQ) is a widely used preservative that inhibits the rancidity of foodstuffs and can preserve food without any alteration in its color, smell, texture, and aroma. However, higher concentrations of TBHQ can stimulate carcinogenicity, immune system damage, convulsions, medullary paralysis, vision disturbances, and dermatitis, thus requiring rigorous quality control in food industries. So, SWV permits the detection of low concentrations of TBHQ and other preservatives, with suitable sensitivity and accuracy. Among this, Balram (2022b), developed an electroanalytical procedure for TBHQ detection using SWV allied to a sensor based on a surface-functionalized carbon black into Co_3O_4 spinel nanorods. For this, it used buffer solution pH 7.0, where analytical curves were constructed with a linear range from 0.12 to 62.2 $\mu\text{mol L}^{-1}$ and LOD of 1.0 nmol L^{-1} . Furthermore, the methodology was applied to food samples such as bovine tallow, peanut oil, and lake water, resulting in recovery values between 95 to 104%, indicating a good accuracy of the SWV and the proposed sensor.

On the other hand, Sesbastin et al. (2022) employed SWV allied to a sensor prepared by carbon black modified by ternary metal oxide functionalized with β -cyclodextrin, which exhibited exceptional electrocatalytic activity for the detection of TBHQ, evident by the resulting very low detection limit of 1 nmol L^{-1} . Furthermore, the TBHQ sensor developed showed all the important characteristics of a good electrochemical sensor, including excellent selectivity, stability, reproducibility, and repeatability. Furthermore, to validate the practical feasibility of detecting TBHQ, we successfully determined this food additive in edible oils.

In another study, an excellent detection performance for TBHQ was observed using SWV allied to nanoparticle composites decorated with gold on tungsten carbide. Voltammetric experiments were performed in 0.05 mol L^{-1} of phosphate buffer solution, allowing low working potential and high peak current. In particular, the composite was able to detect TBHQ oxidation within a linear concentration range from 5 to 75 nmol L^{-1} , with an extremely low detection limit of 0.20 nmol L^{-1} . The sensor's practicality was also evaluated in the TBHQ analysis in samples of soybean oil, blended oil, and red wine, with satisfactory recovery rates obtained (EZHIL VILIAN *et al.*, 2021).

Sulfides are used as preservatives in wine production, in the prevention of oxidation, inhibition of bacterial growth, and control of enzymatic and nonenzymatic reactions during their preparation, storage, and distribution stages. However, quality control is needed due excessive use may cause nausea, allergic reactions, gastric

irritation, nettle rash, and asthmatic attacks. So, Lozer et al.(2023), developed a methodology for the determination of sulfites in wines using SWV and screen-printed carbon electrodes modified with carboxyl-functionalized multi-walled carbon nanotubes. Using phosphate buffer solution pH 4.00 as supporting electrolyte, SWV parameters were optimized using experiment design, and chosen values obtained for step potential are 7.00 mV, modulation amplitude 75.0 mV, and frequency 14.05 s⁻¹. The sulfites showed an oxidation peak around 0.700 V, which corresponds to the HSO⁻₃ species, and quantification and detection limit values of 49.9 and 16.5 µmol L⁻¹, respectively, in the range of 5.0 to 800.0 µmol L⁻¹. The method showed no interference between common molecules found in wine. Furthermore, wine samples were analyzed by the proposed technique and compared with the official Brazilian method (iodometric titration). There was no significant difference between the sulfite concentrations obtained in both procedures. These results show that the proposed methodology is promising for monitoring sulfite agents, using a small amount of sample per analysis, and the possibility of applying *in loco* determination of sulfites.

Xu et al (2016) determined sulfites in rice wine samples using SWV allied to an electrochemical sensor based on a carbon black paste electrode in phosphate buffer solution pH 7.4 as a supporting electrolyte, it was observed that the sulfite oxidation current in this sensor increased significantly compared to the bare glassy carbon electrode or graphite paste electrode. Through first-order derivative processing, peaks have become narrower and sharper, leading to improved sensitivity. The external standard calibration curve was in the range from 0.008 to 1.0 µmol L⁻¹. with a detection limit of 6.0 µmol L⁻¹. This method was applied in the determination of sulfites in rice wine samples available and the results were compared with a titration method.

Nitrites are used as conservators in the production of meat products. However, nitrates and nitrites salts can result in nitrosamines production when heated to a high temperature and these compounds can raise cancer risk. So, Qin et al. (2023) employed SWV and chemically modified graphene oxide electrodes as an electrochemical nitrite procedure of detection. Using phosphate buffer solution pH = 7.0 the sensor responded linearly with the increase of nitrite concentrations up to 1000 µmol L⁻¹ and a low LOD (2.0 µmol L⁻¹) was obtained. In addition, the selectivity, stability, and reproducibility qualities of the sensor are also evaluated. Furthermore, it successfully detected the analyte in samples of tap water, mineral water, and river water with relative standard deviations of less than 5%.

Wong et al. (2022) built a voltammetric sensor using a glassy carbon electrode based on hierarchically porous carbon with nanoparticles of silver sulfide, Nafion[®], and fullerene for the determination of nitrite in foods. The use of Nafion[®] and fullerene contributed to increasing the specific surface area and improvements the electrochemical performance of the sensor. Using optimal conditions, a linear response range from 4.0 to 148 $\mu\text{mol L}^{-1}$, with a LOD of 0.09 $\mu\text{mol L}^{-1}$. The results showed that the proposed method can selectively detect nitrite in the presence of other compounds without interference and with good stability, and for this, was successfully applied for the detection of nitrite in food samples, demonstrating a good degree of accuracy and satisfactory efficiency.

In the production of foodstuffs, food additives mixtures are usually employed to obtain the desired technological properties. Therefore, the development of analytical methodologies for the simultaneous detection of various food additives is desirable. Among this, Ascencio-Flores et al. (2023) used SWV in the simultaneous detection of sunset yellow and tartrazine in commercial drinks, allowing in an analytical procedure with a wide linear range from 0.0038 to 12.9 $\mu\text{mol L}^{-1}$ and LOD of 17.6 nmol L^{-1} and 30.9 nmol L^{-1} for sunset yellow and tartrazine, respectively. These results were obtained using a carbon paste electrode modified by carbon nanofibers superficially recovered with silver nanoparticles, allowing the simultaneous detection of both dyes with excellent selectivity, even in the presence of various interferents (sodium benzoate, NaNO_3 , NaHCO_3 , glucose, and citric acid), with good reproducibility, repeatability, and stability.

Food dyes are organic substances that have important effects on human health and food safety. Although these substances do not pose a problem when used in daily intake amounts (ADI), they harm human health when consumed in excess. Amaranth and carminic acid are ingredients of synthetic and natural food colors, respectively. The analysis of these substances in samples of food, pharmaceuticals, cosmetics, and textiles is extremely important due to their genotoxicity, cytostatic and cytotoxic effects. Electroanalytical methods, which have great advantages over traditional analytical methods, illuminate the scientific world. Electrochemical monitoring modules, which are fast, simple, accurate, reliable, and highly selective, hold promise for the determination of both substances. So far, food determinations of amaranth and carminic acid have been successfully performed with electrochemical monitoring techniques in many numbers in the literature. Voltammetric techniques are the most

used among these electroanalytical methods. In particular, SWV and DPV techniques, which have extraordinary properties, have been strongly preferred. LOD comparable to the standard analytical method has been achieved using these methods, which have very fast analysis durations, high precision and accuracy, do not require lengthy pre-processing, and have great selectivity. Furthermore, more sensitive and selective analyzes of amaranth and carminic acid in natural samples have been performed with numerous indicator electrodes (ALIZADEH *et al.*, 2022).

Blandon-Naranjo *et al.* (2022) developed a simple and reliable electroanalytical method to simultaneously determine the antioxidants butyl hydroxyanisole, butyl hydroxytoluene, and propyl gallate. It used SWV allied to platinum and carbon fiber disk ultramicroelectrodes (electronic tongue configuration) combined with chemometrics tools. This procedure enabled percent recovery assays in good agreement with those obtained by the chromatographic method, demonstrating the analytical accuracy of the proposed method. Furthermore, the results of the simultaneous determination of these antioxidants in edible oil samples were better than others found in the literature regarding repeatability and the percent recoveries.

Parabens are a class of food additives used as preservatives in sweeteners, however, are considered endocrine disruptors due to their negative effects on the endocrine system. So, these compounds were evaluated by Dantas *et al.* (2021) using SWV combined with a glassy carbon electrode for the simultaneous determination of methyl, ethyl, propyl, and butyl parabens in sweeteners. To overcome the strong overlapping of voltammetric signals caused by calibrated and uncalibrated constituents, split partial least squares with residual bilinearization were used as the calibration model. Satisfactory and unbiased results were obtained with a linear response in the range from 0.78 to 4.48 $\mu\text{mol L}^{-1}$, and recoveries from 82.64% to 121.77%. So, the voltammetric data demonstrated that the proposed methodology was capable of simultaneously determining four parabens in complex samples, such as sweeteners, without any previous pre-treatment.

The sensitivity of SWV allied to stripping voltammetry and the easier preparation of samples for electroanalytical measurements enabled the natural antioxidants detection in margarine and edible oils by Sýs *et al.* (2017). For this, they used previous extraction of tocopherols using silicone oil, which was subsequently used as the lipophilic binder of a glassy carbon paste electrode, with subsequent electrochemical detection by square wave anodic stripping voltammetry. Analytical parameters were

evaluated and the LOD was calculated as 3.30 nmol L^{-1} with only 15 seconds of accumulation in $0.1 \text{ mol L}^{-1} \text{ HNO}_3$ indicating that this procedure is a very sensitive method for the determination of tocopherols, being comparable to reversed-phase HPLC chosen as reference method.

5.3 The most commonly used working electrodes in food additives detection

The success in the voltammetric analysis is related to the good choices of the material for working electrode preparations since the redox reaction occurs in the interface electrode/solution, and any change in this can result in alterations of the I_p , E_p , and $w_{1/2}$. In practice, the working electrode material is chosen according to the chemical structure of the compounds of interest. The redox reaction occurs in specific chemical groups that can be easily oxidized and/or reduced in selected surfaces. In addition, the characteristics of signal-to-noise ratio and reproducibility in the signals and also results in voltammetric methods with adequate robustness, efficiency, sensitivity, low cost, and aim to meet the principles of green chemistry should also be considered in the working electrode choice (MARTINS; SENTANIN; DE SOUZA, 2021).

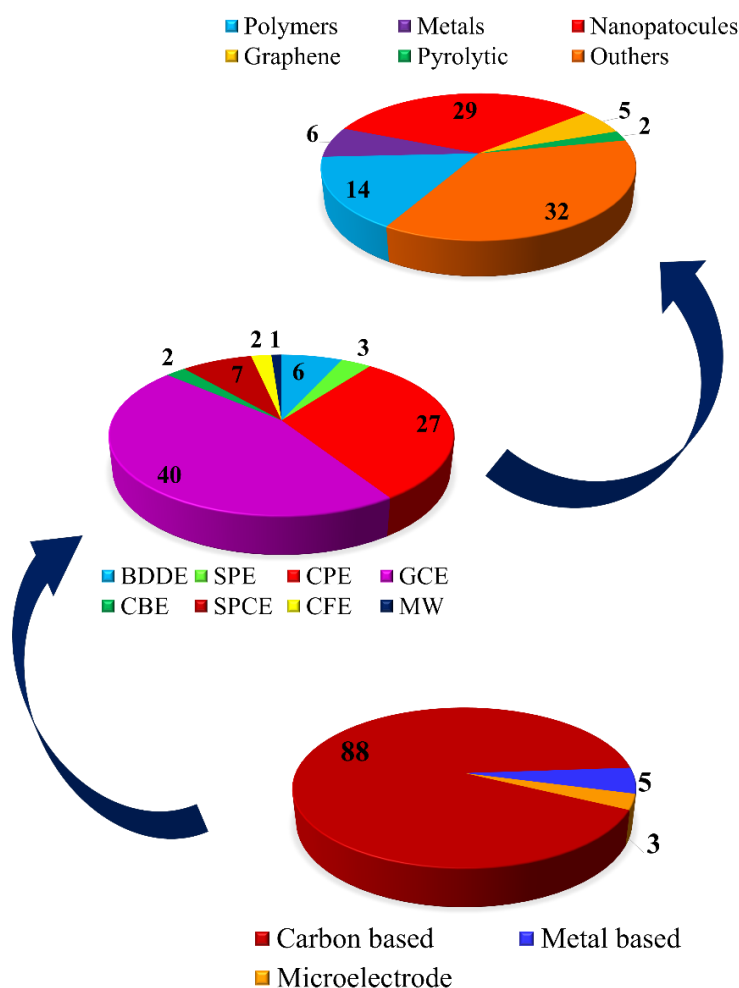
Erro! Fonte de referência não encontrada. shows the main materials used to prepare working electrodes for food additives detection, which were selected using the tools choices described in *Scopus and Approaches* item. Carbon-based electrodes are the most used for food additives analysis, as shown in Figure 2 because these materials have a wide range of anode potential, low cost, chemical inertness, and mechanical stability.

As observed in Figure 1, dyes are the most researched food additives due to their toxic effects on human health, and the maximum acceptable levels for the food additive are well established. The use of DPV and SWV has been frequently reported, as previously demonstrated. However, detection success depends on the materials used as working electrodes or voltammetric sensors. For this, recently, some reviews were made considering using various materials, mainly carbon-based and chemical modifications, to improve the analytical performance of the sensor.

Al-Hawary et al. (2023) summarized the recent progress in the development and application of working electrodes based on nanomaterials comprising carbon-based, silica-based, polymer, and metallic-based composites in the detection of food dyes.

This review showed the advantages and disadvantages of these materials, allowing improvements in the analytical parameters due to several features of nanomaterials, such as high capturing area, biocompatible, high conductive probes, and excellent electronic properties.

Figure 2: Main materials employed in working electrode preparation for food additives detection. Data were obtained from the scientific databases ScienceDirect® and Scopus® from 2012 to 2023.



Karimi-Maleh et al. (2022) reviewed the use of carbon nanomaterial in the preparation of voltammetric sensors for dye detection. They observed that carbon nanotubes (CNT) and graphene (Gr) based sensors allowed improvements in the azo dye detection range and reduced the limit of detection. However, these materials still need some adjustment to enhance the electrocatalytic active sites and improve the stability and sensitivity of electrodes, which can be strongly affected by the purity of the carbon nanomaterials, size, shape, and specific surface area.

Monisha et al. (2023) also reviewed the synthesis, advantages over other sensors, and applications of nanomaterials in the detection of various azo-toxic dyes.

They demonstrated that the employ of nanosensors results in analytical procedures with great sensitivity and specificity, as well as are perfect for high-throughput applications, including real-time monitoring.

Laborda et al, (2012) discussed the maximization of the voltammetric responses of DPV and SWV analysis using chemically modified electrodes with conductive porous layers. These modifications enable improvements in voltammetric response due to promoting an increase in the kinetics and the diffusion transport of the target species.

6 Trends and Perspectives

As observed in Tables 1 and 2, we have a wide variety of materials for working electrodes used in studies over the last 10 years. success in obtaining the voltammetric profile of food additives, as it is a class with various types of molecules. Therefore, the potential range, electrical conductivity, toxicity, surface reproducibility, mechanical properties, and manufacturing cost must be considered. However, no work electrode material does not have all the desirable characteristics, and mainly for the analysis of food additives, most carbon surfaces are used in the form of carbon paste, carbon fiber, vitreous carbon, carbon nanotubes, carbon nanotubes, carbon films, carbon composites, graphene, and diamond. Its main features are a wide range of anodic potential, low cost, chemical inertness, and mechanical stability (SCHOLZ, 2010).

Considering the use of electrochemical techniques and the development and use of working electrodes that can be easily prepared and miniaturized, disposable devices, of low cost and without toxic composition, are needed that allow fast in situ analyses with small volumes of samples in very short times. The possibility of automation in analytical procedures and field analysis with adequate reliability and repeatability is the main perspective. Therefore, the use of screen-printed electrodes, based on screen-printing technology, presents itself as an interesting possibility, expanding the applicability of voltammetric techniques in quality control and food safety assessment. Furthermore, these electrodes can be easily modified by chemical compounds, including biomolecules and metallic nanoparticles, improving their sensitivity and selectivity.

7 Conclusions

The use of electrochemical techniques allows analytical parameters like those obtained by chromatographic techniques; however, chemical analysis results in simplified sample preparation steps, using simple instrumentation, low cost, and rapid analysis. However, the use of electrochemical techniques to analyze food additives requires the use of chemically modified electrodes to improve sensitivity and allow reproducibility of voltammetric signals. Appropriate modification choices in the working electrodes result in an adequate analysis of food additives, with a simple, fast, and low-cost sample preparation procedure before the voltammetric analysis.

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