



Original scientific paper

## A voltammetric sensor based on $\text{CuCo}_2\text{O}_4$ nanorods and ionic liquid for determination of sunset yellow

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### Abstract

In this study, we report the electrochemical detection of sunset yellow (SY) at the surface of a carbon paste electrode (CPE) modified with  $\text{CuCo}_2\text{O}_4$  nanorods (NRs) and an ionic liquid (IL). The electrochemical behavior of sunset yellow at the modified electrode was investigated using linear sweep voltammetry, differential pulse voltammetry and chronoamperometry techniques. The  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE sensor exhibited good performance and favorable electrochemical response for the electrochemical reaction of SY in a 0.1 M phosphate buffer solution at pH 7.0. The results showed a linear electrochemical response to SY within the concentration range of 0.2 to 160.0  $\mu\text{M}$ , with a detection limit of 0.06  $\mu\text{M}$ . In addition, The  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE sensor demonstrated good ability for SY determination in real samples.

### Keywords

Electrochemical sensors; carbon paste electrode; linear sweep voltammetry; differential pulse voltammetry; synthetic food dye

### Introduction

In recent years, the food and beverage industry has utilized various additives in its products. Nowadays, additives are commonly used in a variety of foods, including dairy products, beverages, cereals, snack foods, and ice creams, to make them more attractive and appealing to consumers. Among the various types of food additives, food colorants play an important role in food products

due to their impact on physical appearance and attracting the attention of consumers. Food colorants are categorized into natural and synthetic dyes. However, synthetic food dyes are more widely used in the food industry compared to natural colorants. This is because synthetic dyes are generally less expensive, more stable during food processing, and offer consistent color uniformity, resistance to light, oxygen, and pH changes, as well as better microbial stability and stronger tinting ability. The food industry's reliance on synthetic dyes is driven by their practical and economic benefits, which allow manufacturers to meet consumer demand for visually appealing food and beverage products [1-3]. Sunset yellow (SY) is a synthetic food dye containing an azo group. It is commonly used to color various food products, such as beverages, fruit juice powders, gum, jams, and sweets. Consumption of foods containing excessive amounts of SY, particularly by children, is not recommended as it can lead to hyperactive behavioral disorders. Additionally, SY has been associated with symptoms of intolerance, allergic reactions, and asthma in some individuals. Consequently, the analysis of synthetic colorants like SY in foods is of great importance. To ensure food safety and reduce human exposure to hazardous chemicals, sensitive, selective, and accurate analytical methods are required for the determination of SY in food products to which it has been added. These methods are necessary to monitor and control the levels of this synthetic dye in the food supply to protect consumer health [4,5]. The commonly proposed methods for the detection of SY dye in various food samples include High-performance liquid chromatography-mass spectrometry (HPLC-MS), Liquid chromatography-mass spectrometry (LC/MS), spectrophotometric techniques, capillary zone electrophoresis, and electrochemistry [6-12]. These analytical techniques have been extensively reported in the literature for the identification and quantification of SY in different food matrices.

Electroanalytical techniques, particularly voltammetry, offer several advantages for the determination of target species. These advantages include speed, high sensitivity, low detection limits, simplicity, and suitability for target species determination [13-18]. However, the efficiency of voltammetric techniques for the analysis of electroactive compounds depends on the choice of a suitable working electrode. The selection of suitable materials for the modification of the working electrode is crucial, as it can influence the sensitivity, selectivity, and overall performance of the voltammetric method for the determination of compounds in various matrices [19-23]. Recent studies and investigations have shown that nanoscience has received considerable attention from researchers due to its applicability in various fields [24-27]. Interestingly, nanostructures have been widely applied in the electroanalysis of various compounds due to their high surface area and improving the speed of electron transfer in electrochemical reactions [28-32]. Transition metal-based compounds have shown promise in catalyzing various electron transport pathways. Among them, cobalt oxide ( $\text{Co}_3\text{O}_4$ ) is an available material with good properties, making it a commonly used electrode material in electrochemical processes. Substituting the cobalt ions within the lattice structure with other cations, such as manganese, zinc, copper, or nickel, can further enhance the versatility and properties of the cobalt-based materials. One such material is  $\text{CuCo}_2\text{O}_4$ , which belongs to the spinel oxide family.  $\text{CuCo}_2\text{O}_4$  exhibits outstanding electrochemical performance, low cost, and desirable electrical properties, making it a valuable electrode material for various applications, including supercapacitors, lithium-ion batteries, and electrocatalysts. The incorporation of copper into the cobalt oxide structure can modify the material's electronic, structural, and catalytic properties, leading to improved electrochemical performance and expanding its potential for various applications. The good properties of  $\text{CuCo}_2\text{O}_4$  have attracted significant research interest in the scientific community for its utilization as an effective electrode material in various electrochemical applications [33,34].

In recent years, ionic liquids (ILs) have gained significant attention from electrochemists due to their remarkable properties. Some of the key advantages of ILs that have made them attractive for electrochemical applications include non-volatility, high viscosity, high conductivity, high polarity, high thermal stability, and wide electrochemical window. These unique physicochemical properties of ILs have made them attractive candidates for incorporation into electrochemical systems, such as energy storage devices, sensor applications, *etc.* [35,36]. The ability to tailor the properties of ILs through the selection of cations and anions further expands their versatility in electrochemical applications.

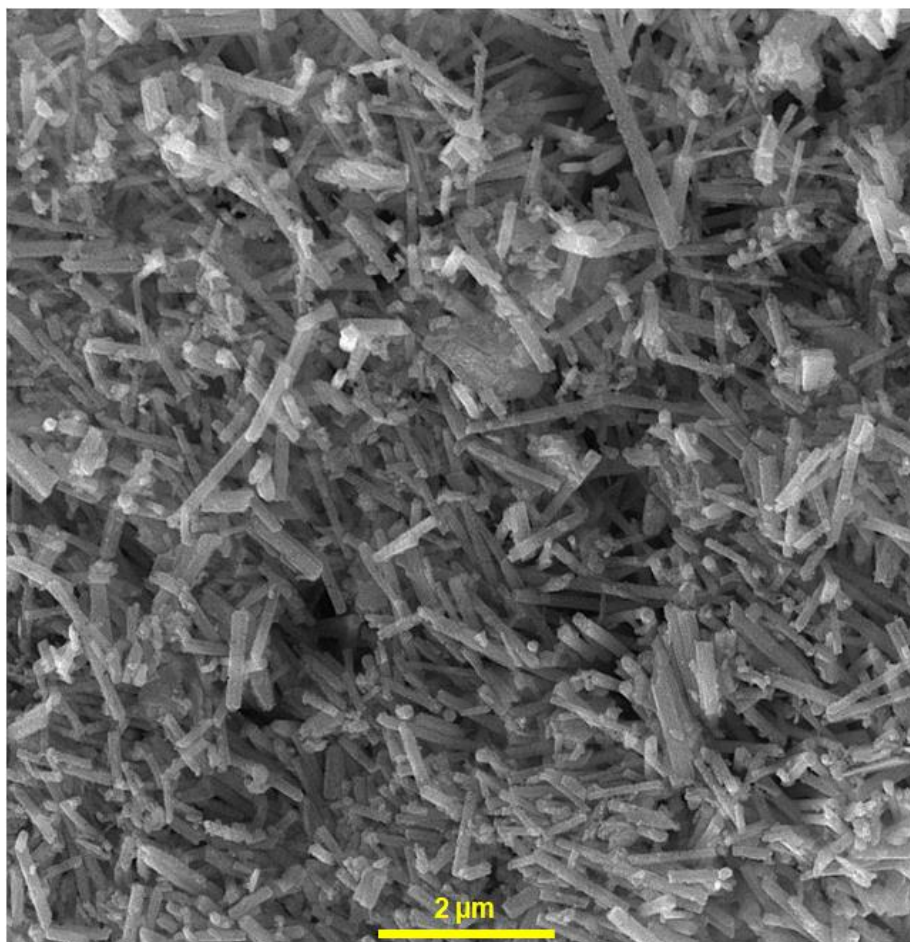
In this study, we developed a CPE modified with  $\text{CuCo}_2\text{O}_4$  NRs and an IL for the electrochemical detection of SY in real sample.

## Experimental

### Materials

All the chemicals used in this study were purchased from Sigma-Aldrich and Merck companies and used as received without any further purification. A PBS (0.1 M) with a pH of 7.0 was used in all the analysis experiments. All the aqueous solutions were prepared using deionized water.

The  $\text{CuCo}_2\text{O}_4$  NRs were successfully synthesized and characterized, as reported in our previous work [37]. The FE-SEM image of  $\text{CuCo}_2\text{O}_4$  NRs is shown in Figure 1.



**Figure 1.** FE-SEM image of  $\text{CuCo}_2\text{O}_4$  NRs

### Fabrication of $\text{CuCo}_2\text{O}_4$ NRs/IL/CPE

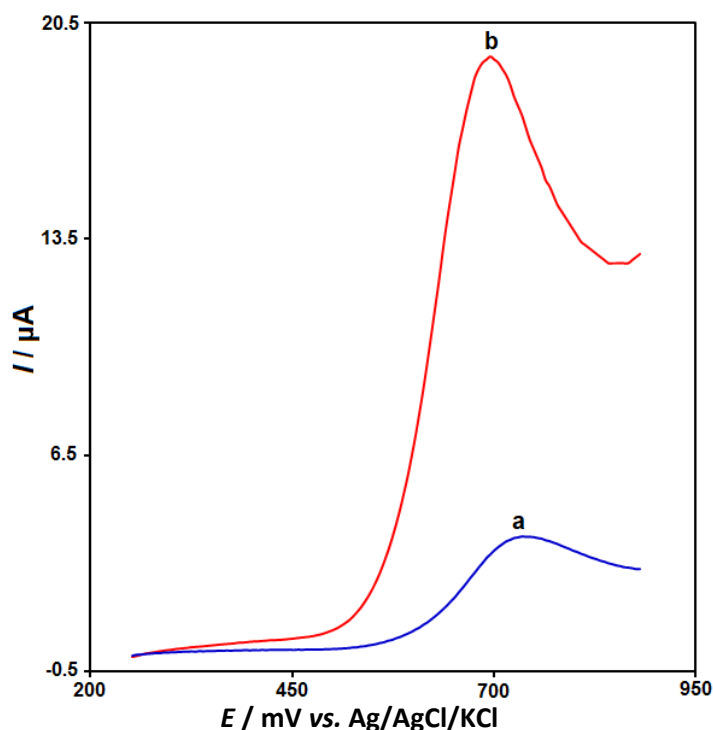
The  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE was prepared using the following procedure:

In a mortar, 0.196 g of graphite powder and 0.004 g of  $\text{CuCo}_2\text{O}_4$  NRs were mixed using a pestle. 20  $\mu\text{L}$  of an IL (1-butyl-3-methylimidazolium hexafluorophosphate) and 65  $\mu\text{L}$  of paraffin oil were added to the graphite-  $\text{CuCo}_2\text{O}_4$  NRs mixture as binders. The resulting paste was thoroughly homogenized by continuous grinding with the pestle. The paste was then packed into the end of a glass tube and a copper wire was utilized for establishing an electrical contact.

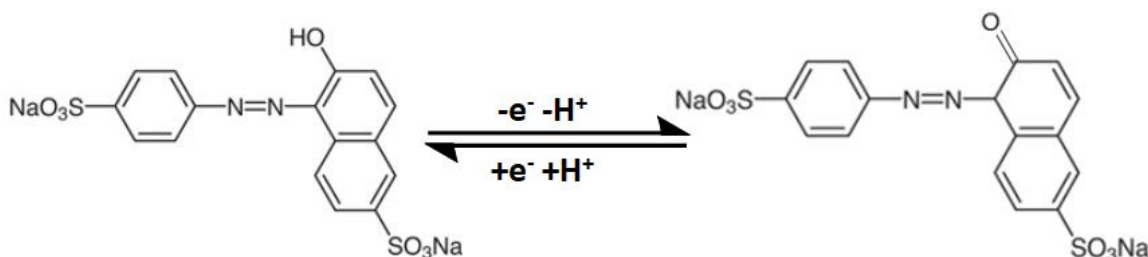
## Results and discussion

### Electrochemical behavior of sunset yellow

Figure 2 presents the LSV results for the electrochemical oxidation of SY (100.0  $\mu\text{M}$ ) at the bare CPE and the  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE. With the bare CPE (linear sweep voltammogram a), the oxidation of SY produced a broad peak with an anodic peak potential ( $E_{\text{pa}}$ ) of 740 mV and an anodic peak current ( $I_{\text{pa}}$ ) of 3.85  $\mu\text{A}$ . In contrast, at the  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE, the linear sweep voltammogram showed a more defined oxidation peak with an  $E_{\text{pa}}$  of 700 mV and an  $I_{\text{pa}}$  of 19.3  $\mu\text{A}$  (linear sweep voltammogram b) for sunset yellow. The  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE exhibited a significantly lower peak potential (40 mV negative shift compared to the bare CPE) and about a 5-fold higher oxidation current compared to the bare electrode. The good properties of the  $\text{CuCo}_2\text{O}_4$  NRs and the role of IL as a conductive binder improve the electrochemical reaction of SY at the surface of modified CPE. The electrochemical reaction of SY is shown in Scheme 1.

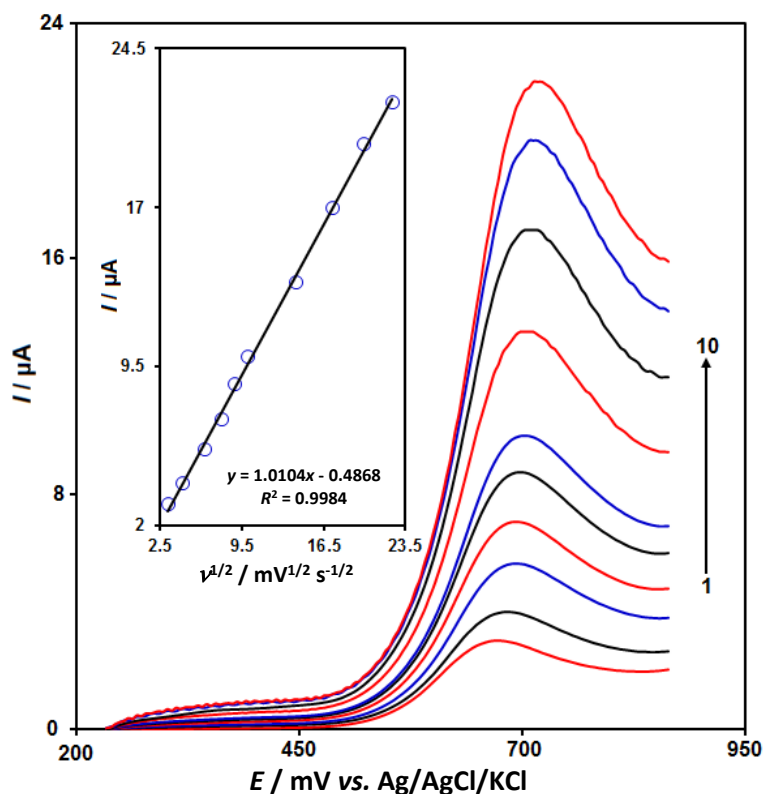


**Figure 2.** LSVs of (a) bare CPE; and (b)  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE in the presence of 100.0  $\mu\text{M}$  SY (PBS, 0.1 M, pH 7.0) at scan rate 50 mV/s



**Scheme 1.** The mechanism for electrochemical reaction of SY

The LSVs for 30.0  $\mu\text{M}$  SY were recorded at the  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE with a range of scan rates from 10 to 500 mV/s, and the obtained data are presented in Figure 3. The inset in Figure 3 shows a linear relationship between the peak current ( $I$ ) and the square root of the scan rate ( $v^{1/2}$ ), with the equation  $I_{pa} = 1.0104 v^{1/2} - 0.4868$  and a coefficient of determination ( $R^2$ ) of 0.9984. This linear relationship between  $I$  and  $v^{1/2}$  confirms that the electrochemical reaction of SY at the surface of the  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE is a diffusion-controlled process.



**Figure 3.** LSVs of  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE at different scan rates: 10 (1), 20 (2), 40 (3), 60 (4), 80 (5), 100 (6), 200 (7), 300 (8), 400 (9), and 500 (10) mV/s, in the presence of 30.0  $\mu\text{M}$  SY (in PBS, 0.1 M, pH 7.0). Inset:  $I$  vs.  $v^{1/2}$  plot for maximum current of 30.0  $\mu\text{M}$  SY electrooxidation

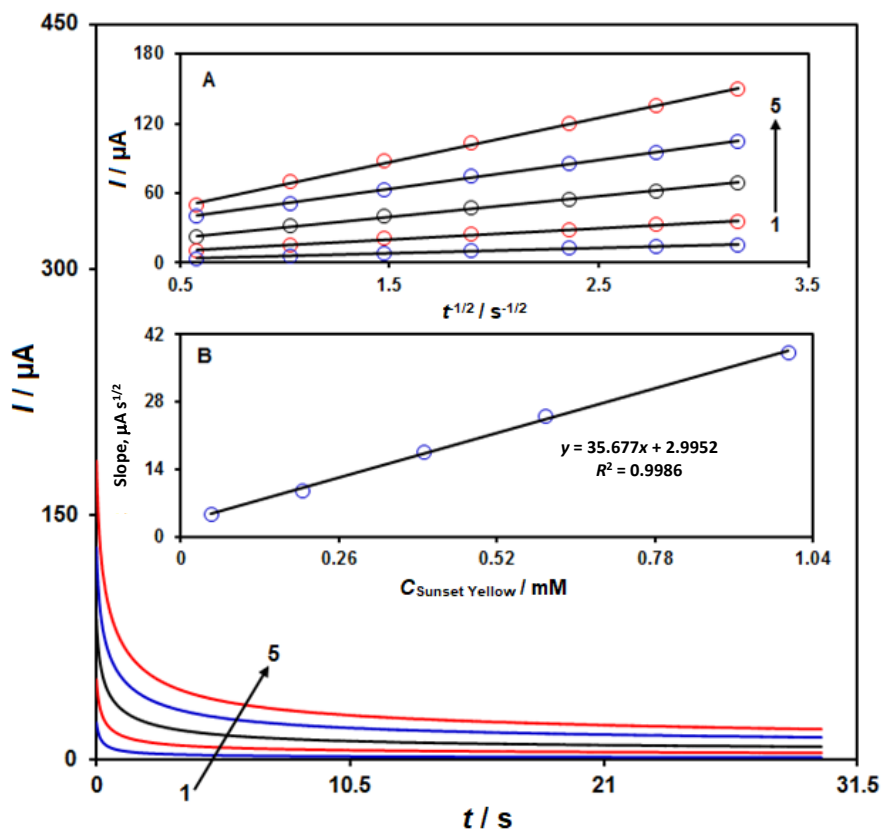
### Chronoamperometry

The diffusion coefficient ( $D$ ) of SY at the surface of the  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE was determined by recording chronoamperograms at an applied potential of 700 mV for SY concentrations ranging from 0.05 to 1.0 mM (Figure 4). The Cottrell plots were drawn by plotting the current ( $I$ ) against the inverse square root of time ( $t^{-1/2}$ ) for the different SY concentrations (Figure 4A). The slopes of these Cottrell plots were then drawn against SY concentrations (Figure 4B), and the slope obtained from this plot was utilized to calculate the diffusion coefficient of SY at the modified electrode surface. Based on the Cottrell equation and the obtained slope from Figure 4B, the value of the diffusion coefficient was determined to be  $D = 5.3 \times 10^{-5} \text{ cm}^2/\text{s}$ .

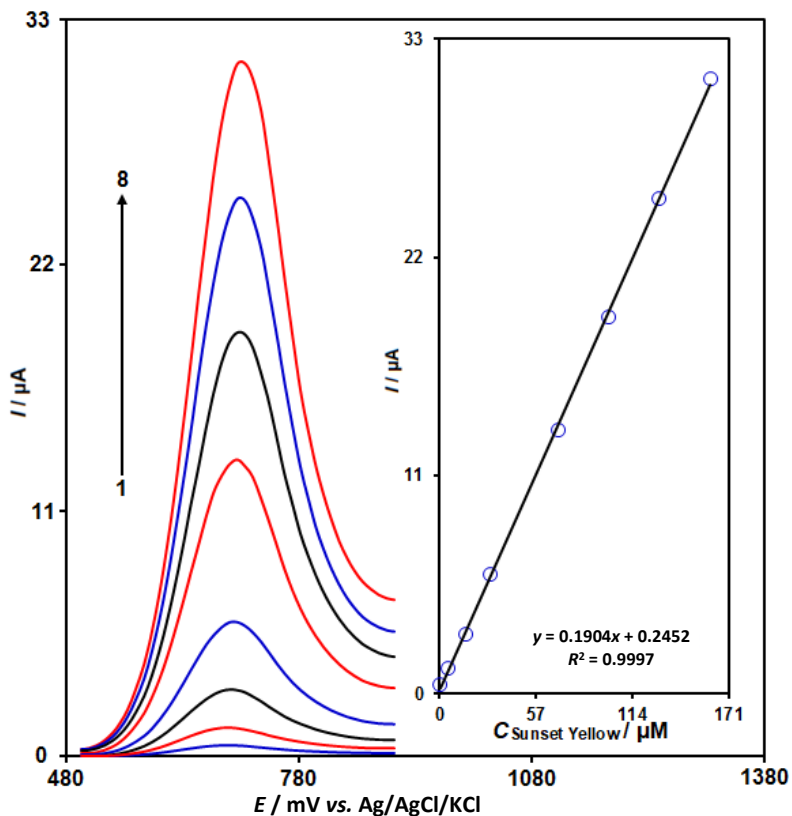
### Calibration curve

Figure 5 presents the DPVs obtained for various concentrations of SY ranging from 0.2 to 160.0  $\mu\text{M}$  at the  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE. According to the recorded voltammograms, the intensity of the oxidation peak current of SY increased with its concentration. The inset of Figure 5 shows the linear relationship between the peak current and the concentration of SY. From the obtained calibration curve, the oxidation peak current of SY changed linearly with the increase of its concentration in the concentration range of 0.2 to 160.0  $\mu\text{M}$ . The LOD was calculated as 0.06  $\mu\text{M}$ .





**Figure 4.** Chronoamperograms obtained at the  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE in the presence of 0.05 (1), 0.2 (2), 0.4 (3), 0.6 (4), and 1.0 (5) mM SY at pH 7.0. Inset A: Plots of  $I$  vs.  $t^{1/2}$  obtained from chronoamperograms. Inset B: the plot of the slope of the straight lines against the SY concentration



**Figure 5.** The DPVs of  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE in 0.1 M PBS (0.1 M, pH 7.0) containing different concentrations of SY: 0.2 (1), 5.0 (2), 15.0 (3), 30.0 (4), 70.0 (5), 100.0 (6), 130.0 (7) and 160.0 (8)  $\mu\text{M}$ . Inset: The plot of  $I$  vs. concentration of SY at the  $\text{CuCo}_2\text{O}_4$  NRs/IL/CPE

### Measurement of sunset yellow in real samples

Finally, the DPV method was used to verify the applicability of the CuCo<sub>2</sub>O<sub>4</sub> NRs/IL/CPE sensor for determination of SY in various samples, including orange jelly powder and orange juice. The amounts of SY in these samples were determined (see Table 1). Then, the standard addition method was used to determine SY in the samples spiked with SY at various concentrations. The recovery values in these samples ranged from 97.7 to 103.8 %. Therefore, the developed sensor in this study was suitable for the quantitative determination of SY in real samples.

**Table 1.** Determination of SY in orange jelly powder and orange juice samples (n=5)

Sample	SY concentration, $\mu\text{M}$		Recovery, %	RSD, %
	Spiked	Found		
Orange jelly powder	0	2.3	-	3.7
	2.0	4.2	97.7	2.8
	4.0	6.5	103.2	1.9
	6.0	8.2	98.8	2.5
	8.0	10.4	101.0	2.4
Orange juice	0	2.9	-	2.6
	3.0	6.0	101.7	3.4
	5.0	8.2	103.8	1.8
	7.0	9.7	98.0	2.9
	9.0	12.0	100.8	2.1

### Conclusion

In this study, we developed an electrochemical sensor based on a CuCo<sub>2</sub>O<sub>4</sub> NRs/IL/CPE for the detection of SY. The CuCo<sub>2</sub>O<sub>4</sub> NRs/IL/CPE exhibited good electrochemical activity towards the electrochemical reaction of SY, as evidenced by the significant reduction in the peak potential and enhancement of the oxidation current compared to the bare CPE. In quantitative measurements, the developed sensor displayed a wide linear concentration range of 0.2 to 160.0  $\mu\text{M}$  for the determination of SY. The sensor demonstrated a low LOD of 0.06  $\mu\text{M}$  for SY, indicating its good performance. The proposed electrochemical method was successfully applied for the detection and quantification of SY in real samples, such as orange jelly powder and orange juice, demonstrating the practical applicability of the developed sensor.

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