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Original scientific paper

Flower-like MoS_2 screen-printed electrode based sensor for the sensitive detection of sunset yellow FCF in food samples

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Abstract

In this study, synthesis and electrochemical sensor application of flower-like MoS_2 for sunset yellow FCF sensing were evaluated. Linear sweep voltammetry (LSV), chronoamperometry and differential pulse voltammetry (DPV) were used to determine flower-like MoS_2 electrochemical sensor performances, which had LOD of 0.04 μM in the linear working range of 0.1–150.0 μM calculated from DPV. The sensor was successfully applied for the determination of sunset yellow FCF in real samples.

Keywords

Electrochemical sensor; modified electrode; voltammetric determination

Introduction

Over the past centuries, food and beverage products used different colors to maintain nutritional and health-related advantages. These compounds actively participate in various biological processes. Nowadays, color additives are commonly applied in a variety of foods, including dairy products, beverages, cereals, snack foods and ice creams, to make them more attractive and appetitive. Among various types of food additives, food colorants are played an important role in foodstuff due to their physical appearance and consumer acceptance. Food colorants are categorized into natural and synthetic dyes. However, synthetic food dyes are widely used in the food industry compared to natural colorants due to higher light, oxygen, and pH stability. In addition, they provide a uniform color and a strong tinting ability. Synthetic color additives are organic pigments using artificial synthesis methods and are generally made from coal tar from aniline dyes as raw material [1]. Among the most used dyes by the food industry are those that contain an azo group (–N=N–), including the sunset yellow (SY), the Bordeaux red, and the tartrazine yellow. These dyes are extensively used in cereals, candies, dairy products, jellies, ice creams, fillings, liqueurs, powdered juices, soft drinks, and yogurts [2-4]. Sunset yellow (SY), also known as evening yellow, E110 or edible yellow 3, is one of the most used azo dyes. It has an orange color and is used in a great number of fruit products, like sodas, juices, candies, and ice creams. Usually, it is the only artificial dye present in soft orange beverages. SY also has a large use in the pharmaceutical industry and in cosmetics. Nevertheless, it also causes some side effects in humans, and its consumption has been related to renal failure and hepatocellular damage [5-7].

Until now, different methods such as high-performance liquid chromatography, near-infrared spectroscopy and interval random forest, surface-enhanced Raman spectroscopy and fluorescence spectroscopy have been reported for the determination of sunset yellow [8-11]. In addition, voltammetry was also used to determine sunset yellow dye based on their electrochemical activity [12-14].

Electrochemical sensor systems do not require particular expertise or expensive apparatus for operation. As a result, miniaturization of these systems for in-situ analysis of ecological contaminants is enabled. The advent of screen-printed electrodes (SPEs) has prospects for direct electrochemical mechanisms outside centralized laboratories. Features like rapid response, mass production, low power requirement, and compactness have reinforced SPEs as ideal transducers for environmental assays. Further, the sample volume required for an SPE is as low as 50 µL. Additionally, the surface of the SPEs' working electrode may be readily modified to improve the sensibility, lowering the detection limit, and increasing the selectivity of the electrochemical methods [15,16]. The chemical modification of inert substrate electrodes offers significant advantages in the design and development of electrochemical sensors [17-30]. In operations, the redox-active sites shuttle electrons between a solution of the analyte and the substrate electrodes often along with a significant reduction of the activation overpotential. A further advantage of chemically modified electrodes is that they are less prone to surface fouling and oxide formation compared to inert substrate electrodes [31-45].

The development of nanoscience and nanotechnology has allowed attempts to apply different nanomaterials in different scientific fields [46-51], including the fabrication of chemically modified electrodes [52-56]. In recent years, various nanomaterials have been used singly or in composite form for the modification of electrodes [57-69].

MoS_2 obtained by different preparation methods have different morphology, such as 2D-layered structures, nanorods, nanotubes and three-dimensional structures. Among them, flower-like MoS_2 has a unique three-dimensional structure. Because of its large specific surface area, ultra-short transmission path, high interfacial transfer, and excellent catalytic activity as a matrix, it is widely used in energy storage, catalysis, sensing and other fields. MoS_2 can increase electrocatalytic activity. Molybdenum disulfide (MoS_2), which is a two-dimensional (2D) transition metal dichalcogenides (TMDs), has gained immense attention over the past few years. As a semiconductor with an indirect energy band of 1.29 eV, MoS_2 has a typical 2D layered structure, and each layer is composed of two sulfur atoms and a molybdenum (Mo) atom, forming an S–Mo–S sandwich structure. MoS_2 shows tunable electric properties, excellent electrocatalytic activity, electrochemiluminescent (ECL) signal amplification, photochemical reactivity, fluorescence quenching ability, and adsorption performance and hence is a candidate for catalysis, biology, medicine, sensor monitoring, energy storage and conversion [70, 71]. The aim of this study was to synthesize flower-like MoS_2 (FL-MoS_2) to modify the screen-printed electrodes for the detection of sunset yellow FCF food dye.

Experimental

Apparatus and chemicals

All the electrochemical measurements were carried out on a PGSTAT302N potentiostat/galvanostat Autolab. Screen-printed electrodes (SPE) (DropSens; DRP-110: Spain) consisted of a graphite counter electrode, a graphite working electrode, and a silver pseudo-reference electrode. Solution pH values were determined using a 713 pH meter combined with glass electrode (Metrohm, Switzerland). Sunset yellow and other chemicals used were of analytical grade and were purchased from Merck. Flower-like MoS_2 was synthesized in our laboratory [72]. A typical SEM is shown in Figure 1.

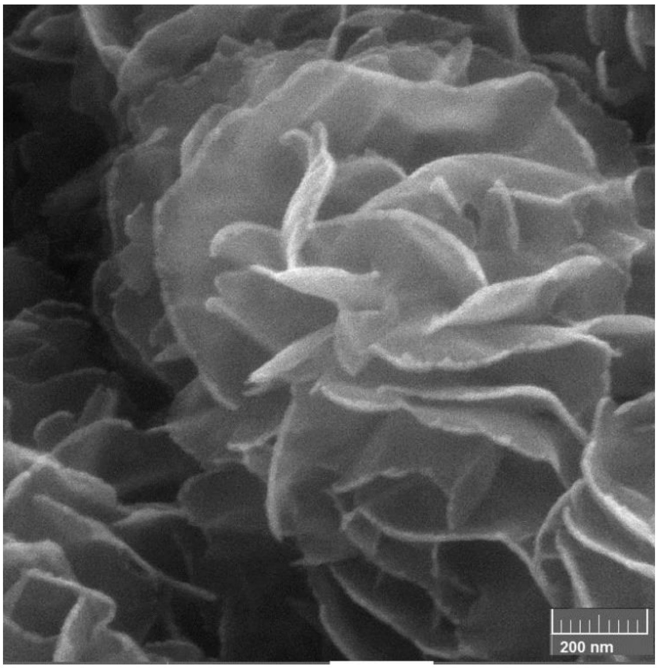

Figure 1. *SEM image of flower-like MoS_2*

Preparation of FL-MoS_2/SPE

First, 1 mg of prepared FL-MoS_2 was added into an aqueous solution (1 ml), followed by sonication for 30 min to give a homogeneous solution. Then, 4 µL of FL-MoS_2 was dispersed on the surface of SPE dropwise. Following the solvent's evaporation, the sensor's surface was washed several times with deionized water to clean free modifier molecules and subsequently air-dried. The obtained electrode was noted as FL-MoS_2/SPE.

The surface areas of the FL-MoS_2/SPE and the bare SPE were obtained by cyclic voltammetry using 1 mM $K_3Fe(CN)_6$ at various scan rates. Using the Randles–Ševčik equation for FL-MoS_2/SPE, the electrode surface was found to be 0.11 cm^2 which was about 3.5 times greater than bare SPE.

Results and discussion

Electrochemical behavior of sunset yellow at the surface of various electrodes

The effect of the electrolyte pH on the oxidation of 60.0 µM sunset yellow was investigated at FL-MoS_2/SPE using DPV measurements in the phosphate buffer solution (PBS) in the pH range from 2.0 to 9.0. According to the results, the oxidation peak current of sunset yellow depends on the pH value and increases with increasing pH until it reaches the maximum at pH 7.0, and then decreases with higher pH values. The optimized pH corresponding to the higher peak current was 7.0, indicating that protons are involved in the reaction of sunset yellow oxidation. This result indicates the oxidation mechanism of sunset yellow involving electron transfers coupled with proton exchange. It is well-known that the hydroxyl group of sunset yellow undergoes a $1e^-/1H^+$ oxidation mechanism at the surface of reported electrodes.

The electrochemical behavior of sunset yellow was investigated by LSV. The linear sweep voltammetry was obtained using the bare SPE (curve b) and FL-MoS_2/SPE (curve a) in 0.1 M PBS (pH 7.0) in the presence of 100.0 µM sunset yellow are shown in Figure 2. On a bare SPE, a signal with a low oxidation current of ~3.3 µA was obtained with a peak potential of ~760 mV. In contrast, FL-MoS$_2$/SPE exhibited an enhanced sharp anodic peak current (I_{pa} = 14.0 μA) at much lower overpotential E_p = 740 mV. These results confirmed that the FL-MoS$_2$/SPE improved the sensitivity of the modified electrode by enhancing peak current and decreasing the overpotential of the oxidation of sunset yellow.

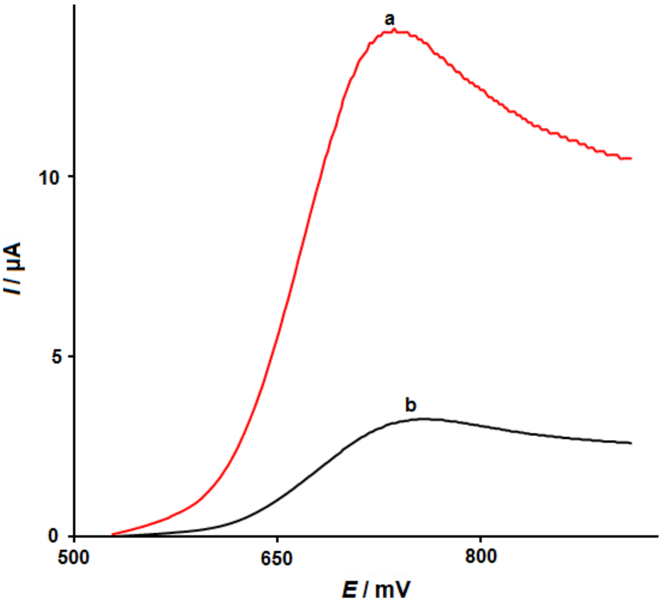

Figure 2. *Linear sweep voltammograms of (a) FL-MoS$_2$/SPE and (b) bare SPE in 0.1 M PBS (pH 7.0) in the presence of 100.0 μM sunset yellow at the scan rate 50 mVs^{-1}*

Effect of scan rate on the determination of sunset yellow at FL-MoS$_2$/SPE

The influence of the scan rate (ν) on the peak currents (I_{pa}) of sunset yellow at FL-MoS$_2$/SPE was investigated by LSV (Figure 3).

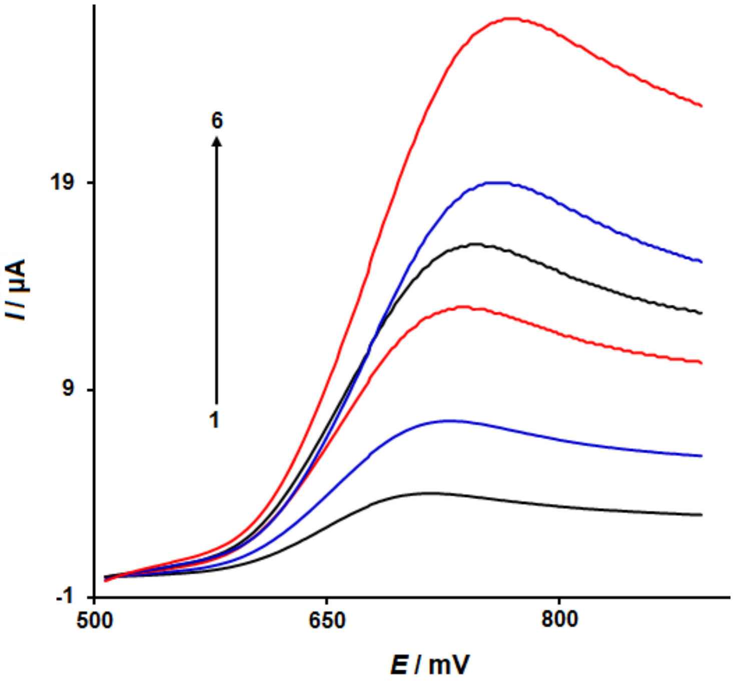

Figure 3. *Linear sweep voltammograms of FL-MoS$_2$/SPE in 0.1 M PBS (pH 7.0) containing 50.0 μM sunset yellow at various scan rates; 1-6 correspond to 5, 15, 45, 70, 100 and 200 mV s^{-1}, respectively*

Figure 4 shows the voltammetric response of 50.0 μM sunset yellow at FL-MoS$_2$/SPE at different scan rates in the range of 5 to 200 mV/s. The oxidation peak current of sunset yellow increases linearly with increasing scan rate. The linear regression equation was obtained from the plot Ipa and *vs.* $\nu^{1/2}$ (square root of scan rate) as follows; $I_{pa} = 1.9165\upsilon^{1/2} - 0.0611$ ($R^2 = 0.9996$) for the oxidation process, which indicates that the reaction of sunset yellow at FL-MoS_2/SPE is diffusion controlled.

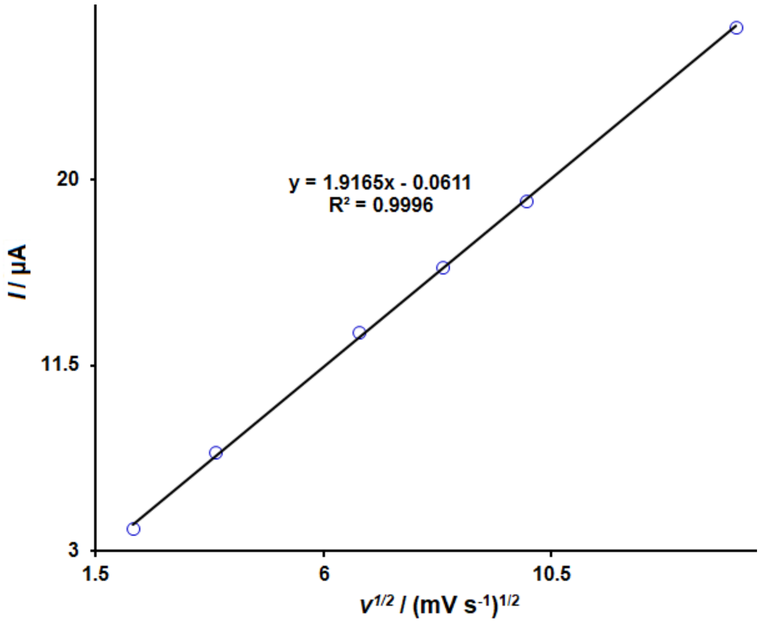

Figure 4. *Plot of anodic peak current* vs. $\nu^{1/2}$ *at different scan rates in the range of 5 to 200 mV/s.*

Chronoamperometric analysis

The analysis of chronoamperometry for sunset yellow samples was performed by use of FL-MoS_2/SPE at 0.77 V. The Chronoamperometric results of different concentrations of sunset yellow in PBS (pH 7.0) are demonstrated in Figure 5. The Cottrell equation for the chronoamperometric analysis of electroactive moieties under mass transfer limited conditions is as in eq. (1)

$$I = nFAD^{1/2}C_b\pi^{-1/2}t^{-1/2} \quad (1)$$

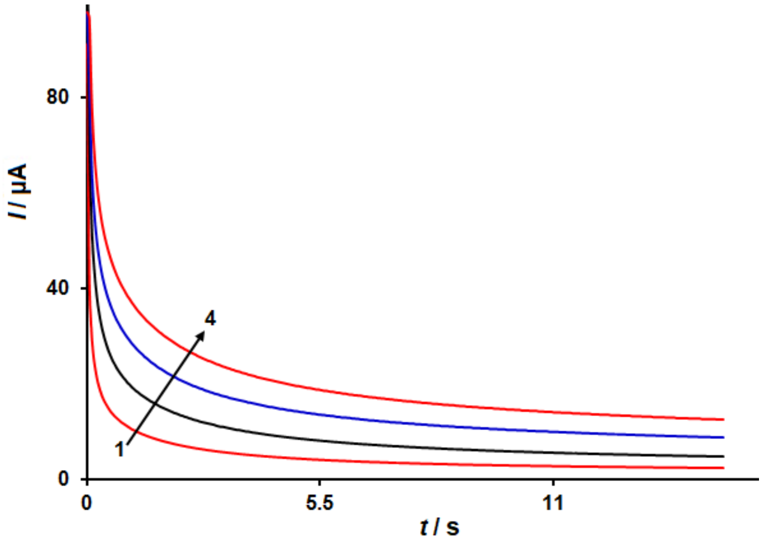

Figure 5. *Chronoamperograms obtained at FL-MoS_2/SPE in 0.1 M PBS (pH 7.0) for different concentration of sunset yellow. Numbers 1-4 correspond to 0.1, 0.75, 1.1 and 1.5 mM of sunset yellow*

where D is diffusion coefficient (cm^2 s^{-1}), and C_b is the applied bulk concentration (mol cm^{-3}). Experimental results of I *vs.* $t^{-1/2}$ were plotted in Figure 6A, with the best fits for different concentrations of sunset yellow. The resulting slopes corresponding to straight lines in Figure 6A, were then plotted against the concentration of sunset yellow (Figure 6B). The mean value of *D* was determined to be 1.1×10^{-5} cm^2/s according to the resulting slope and Cottrell equation.

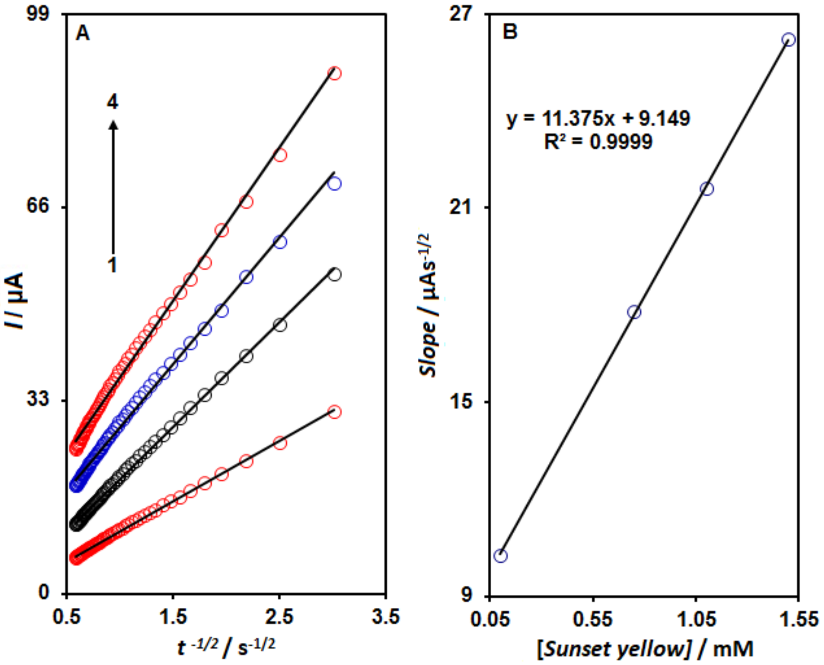

Figure 6. *(A) Plots of* I vs. t$^{-1/2}$ *obtained from chronoamperograms 1-4,*
(B) plot of the slope of the straight lines against sunset yellow concentration (0.1-1.5 mM)

Calibration curve

Because DPV commonly has a higher sensitivity than cyclic voltammetry technology, the DPV technique was applied for the quantitative detection of sunset yellow. Figure 7 shows the differential pulse voltammograms of sunset yellow at various concentrations using FL-MoS$_2$/SPE (Step potential = 0.01 V and pulse amplitude = 0.025 V).

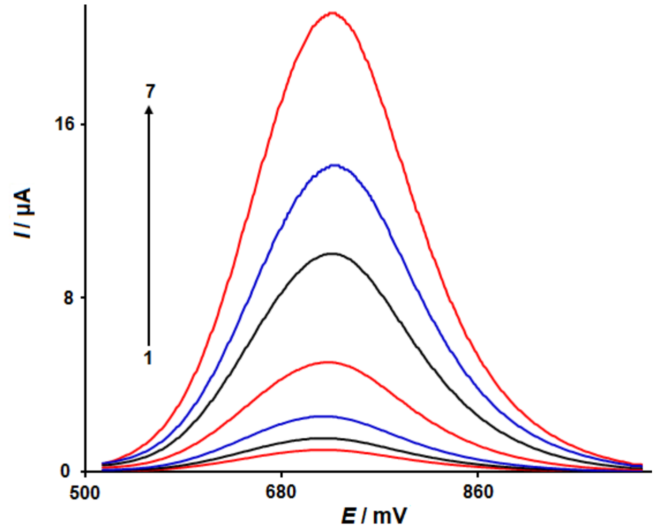

Figure 7. *DPVs of FL-MoS$_2$/SPE in 0.1 M (pH 7.0) containing different concentrations of sunset yellow. Numbers 1–7 correspond to 0.1, 2.5, 10.0, 30.0, 70.0, 100.0 and 150.0 µM of sunset yellow*

As seen, the oxidation peak currents of sunset yellow enhance gradually by increasing its concentration. The oxidation peak currents show a good linear relationship with the concentrations of sunset yellow ranging from 0.1 M to 150.0 μM. The linear equation is $I_{pa} = 0.1315C_{\text{sunset yellow}} + 1.0472$ ($R^2 = 0.9994$) (Figure 8). Also, the limit of detection (LOD) was estimated to be 0.04 μM.

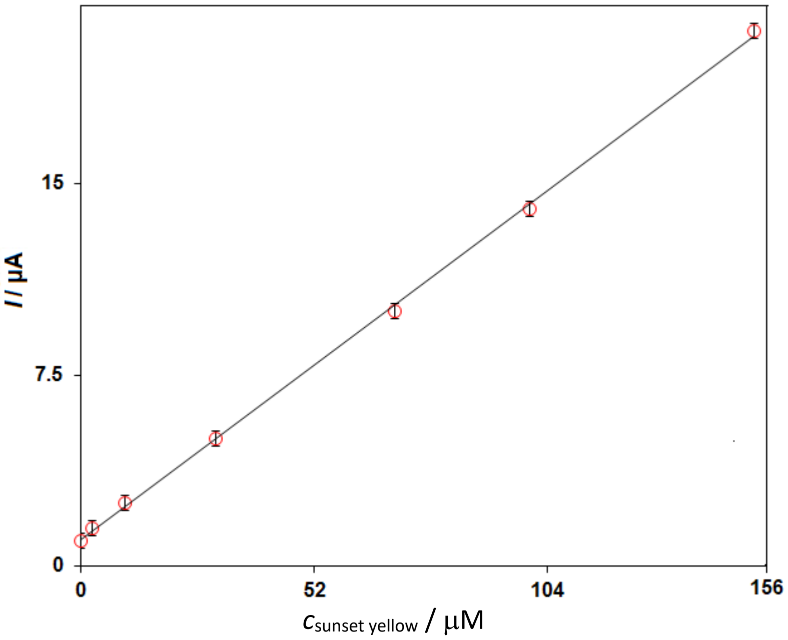

Figure 8. *Plot of the electrocatalytic peak current as a function of sunset yellow concentration in the range of 0.1-150.0 μM*

The performance of this sensor is compared with some of the recently reported electrodes for sunset yellow quantification (see Table 1) [73-76].

Table 1. *Comparison the sensing performances toward the detection of sunset yellow between the existing modified electrodes and the proposed FL-MoS₂/SPE*

Electrochemical Sensor	Method	Linear range, μM	LOD, μM	Ref.
Poly(L-phenylalanine)-modified glassy carbon electrode	DPV	0.04–14.0	0.04	[73]
Graphene oxide decorated with silver nanoparticles–molecular imprinted polymers-modified glassy carbon electrode	LSV	0.1-12.0	0.02	[74]
Gold nanoparticles - modified carbon paste electrode	DPV	0.1-2.0	0.03	[75]
Multi-walled carbon nanotubes modified glassy carbon electrode	DPV	0.55-7.0	0.12	[76]
FL-MoS_2/SPE	DPV	0.1–150.0	0.04	This work

Stability and repeatability

The long-term stability test of the FL-MoS_2/SPE sensor using DPV was performed at room temperature. The results exhibited that the peak current of 35.0 μM sunset yellow at the FL-MoS_2/SPE stayed at 93.2 % of its primary current after 7 days, 90.8 % after 14 days, and 89.97 % after 21 days, indicating the superior long-term stability of the proposed sensor.

The oxidation of sunset yellow (35.0 μM) on the same FL-MoS_2/SPE sensor was performed by ten repeated voltammetric measurements, the results of which confirmed the superior repeatability of the fabricated sensor with the relative standard deviation (RSD) of 2.9 %.

Analysis of real samples

The real samples for the analysis were prepared and quantified by DPV method. The developed sensor was applied to detect sunset yellow in orange and apple juice samples. The results are summarized in Table 2. Each measurement was repeated five times. The recovery and relative standard deviation (RSD) values confirmed that the FL-MoS$_2$/SPE sensor has a great potential for analytical application.

Table 2. *The application of FL-MoS$_2$/SPE for the determination of sunset yellow in real samples (n=3)*

Sample	Concentration, µM		Recovery, %	RSD, %
	Spiked	Found		
Apple juice	0.0	3.0	-	3.4
	2.0			1.9
	3.0			2.2
Orange juice	0.0	2.5	-	2.1
	3.0			3.0
	4.0			2.8

Conclusion

In the present study, sensing of sunset yellow FCF was attempted and implemented successfully with flower-like MoS$_2$ (FL-MoS$_2$) modified screen-printed electrodes. The morphology of flower-like MoS$_2$ was characterized by SEM. Using linear sweep voltammetry and differential pulse voltammetry, the electrochemical oxidative sensing of sunset yellow FCF was investigated. The FL-MoS$_2$/SPE showed a linear response of 0.1 µM to 150.0 µM for sunset yellow FCF, respectively, and their detection limit was equal to 0.04 µM. The stability of the modified electrode was indeed excellent.

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