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A sensitive electroanalytical sensor amplified with Pd-ZnO nanoparticle for determination of Sunset Yellow in real samples

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In the present study, ZnO nanoparticle doped with Pd (ZnO-Pd) using one simple chemical precipitation method has been synthesized and characterized by different characterization methods. The synthesized nanoparticle presented a nano-size spherical shape with d<50 nm. In continuous, ZnO-Pd as a conductive mediator has been used for modification of paste electrode (PE) in the presence of 1-butyl-3-methylimidazolium hexafluoro phosphate, ([C4mim]-[PF6]) and paraffin oil as the binder. The ZnO-Pd/[C4mim]-[PF6]/PE was used as an electroanalytical sensor for the determination of sunset yellow (SY) and obtained results proved a distinct improvement in oxidation signal about 2.57 times compared to an unmodified electrode. On the other hand, the ZnO-Pd/[C4mim]-[PF6]/PE showed a good ability for the determination of SY in concentration range 0.001-280 μM with detection limit 0.4 nM. In addition, the ability of ZnO-Pd/[C4mim]-[PF6]/PE for the determination of SY was checked in various food samples and recovery data between 97.84-104.73% confirm the powerful ability of proposed sensor for determination of SY in food samples.

KEYWORDS

Sunset yellow; ZnO-Pd nanoparticle; 1-butyl-3-methylimidazolium hexafluoro phosphate; Food analytical sensor.

Introduction

Measuring food additives is of paramount importance in the food industry to assess food quality and health [1-5]. Due to some food additives side effects as well as the side effects of adding banned ingredients in some food products, food analysis is used as an important part of food industries [6-10]. Various methods of food analysis have long been reporting, including spectroscopic and chromatographic methods for many years now [11-20]. These methods have many disadvantages such as using toxic solvent, long analysis time, low detection limit (for spectroscopic methods) and so on. [21-23]. In recent years, attention to electrochemical methods for food analysis has been enhanced due to its fast response and the ability to analyze a wide range of compounds [24-35]. What is more, the variety of electrochemical sensors has grown dramatically since their easy modification, allowing the design of selectable sensors [36-45]. Therefore, electrochemical sensors were suggested for the selective and sensitive determination of food and drug compounds [46-55].

Sunset yellow has been used as a yellow dye in the food product such as soft drinks, jams, sweets, candies, pickles, jellies, ice cream and canned juice for many years [56-58]. As an azo dye, it is highly used in food products that

cause major side effects including nasal congestion, urticaria (hives), allergies, hyperactivity, kidney tumors, rhinitis (runny nose), and chromosomal damage [59]. Because of the high use and side effects of over-consumption of this azo dye, various analytical techniques such as HPLC, electrochemistry, and spectroscopy have been reported for rapid analysis of this food additive [59-65]. Electrochemical-based techniques attractive more attention for food and drug analysis due to more benefits such as simple operation, low cost, portable ability, and using nontoxic solvents compare to other analytical techniques [66-75].

With the advent of nanoscience and the unique effects of nanomaterials in the different sciences [76-89] and especially electrochemistry [90-92], the design of electrochemical analytical sensors has undergone a major revolution [93-96].

Room-temperature ionic liquids are one of the most important mediators to the fabrication of highly sensitive electrochemical sensors [97]. Due to the presence of positive and negative charged ions in the structure of ionic liquids, this type of material strongly recommended for improving the conductivity of electrochemical sensors [98, 99].

In the present research, we modified CPE with ZnO nanoparticle doped with Pd and [C4mim]-[PF6] and fabricated a highly sensitive food analytical sensor for determination of SY. The ZnO-Pd/[C4mim]-[PF6]/PE showed excellent catalytic effect for electro-oxidation of SY and used for the determination of SY in food samples.

Experimental

Material and instrument

Sunset Yellow, Zinc nitrate hexahydrate and 1-butyl-3-methylimidazolium hexafluorophosphate were purchased from Sigma-Aldrich. The palladium (II) chloride, and graphite powder were purchased from Acros Company. The phosphoric acid and sodium hydroxide were purchased from Merck Co. and used for the preparation of phosphate buffer solution PBS. The Ivium-Vertex, Potentiostat/Galvanostat machine connected with an electrochemical cell (Azar electrode) was used for voltammetric investigation. The $Ag/Ag/Cl/KCl_{sat}$ making by Azar Electrode Company was used as a reference electrode.

Synthesis of ZnO nanoparticle doped with Pd

The ZnO nanoparticle doped with Pd was synthesis by mixing 50 mL zinc nitrate hexahydrate (1.0 M) + 0.008 g palladium (II) chloride. The solution containing the above materials was stirred for 25 min and then 50 mL sodium hydroxide (2.0 M) was added into the listed solution [100]. The white precipitate samples were dried at 120 °C for 17 h and calcined at 300 °C for 3 h.

Preparation of ZnO-Pd/[C4mim]-[PF6]/PE

The ZnO-Pd/[C4mim]-[PF6]/PE was prepared using a hand mixing composition containing 0.04 g ZnO-Pd +0.96 gr graphite powder and using 8 drops of paraffin oil and 2 drops of /[C4mim]-[PF6] as binders for 4 h using a mortar and pestle.

Results and discussion

Characterization of ZnO-Pd nanoparticle

The ZnO-Pd nanoparticle was characterized by EDS and FESEM methods (Figure 1). The FESEM image (Figure 1A) showed ZnO-Pd nanoparticle with a diameter less than 50 nm. The FESEM image confirms the good distribution of ZnO-Pd nanoparticle synthesized in this work. On the other hand, the presence of Zn, Pd, and O confirm accuracy synthesized ZnO-Pd nanoparticle. On the other hand, the results confirm the purity of synthesized nanoparticles.

Electrochemical behavior of SY at the surface of ZnO-Pd/[C4mim]-[PF6]/PE

The electro-oxidation behavior of 500 µM SY was studied at the surface of ZnO-Pd/[C4mim]-[PF6]/PE in the pH range 3.0-7.0

(Figure 2 inset). The negative shift in the oxidation signal of SY at the surface of ZnO-Pd/[C4mim]-[PF6]/PE was observed with moving pH=3.0 to pH=7.0 with the equation I = -0.0515 pH + 1.0575 (Figure 2).
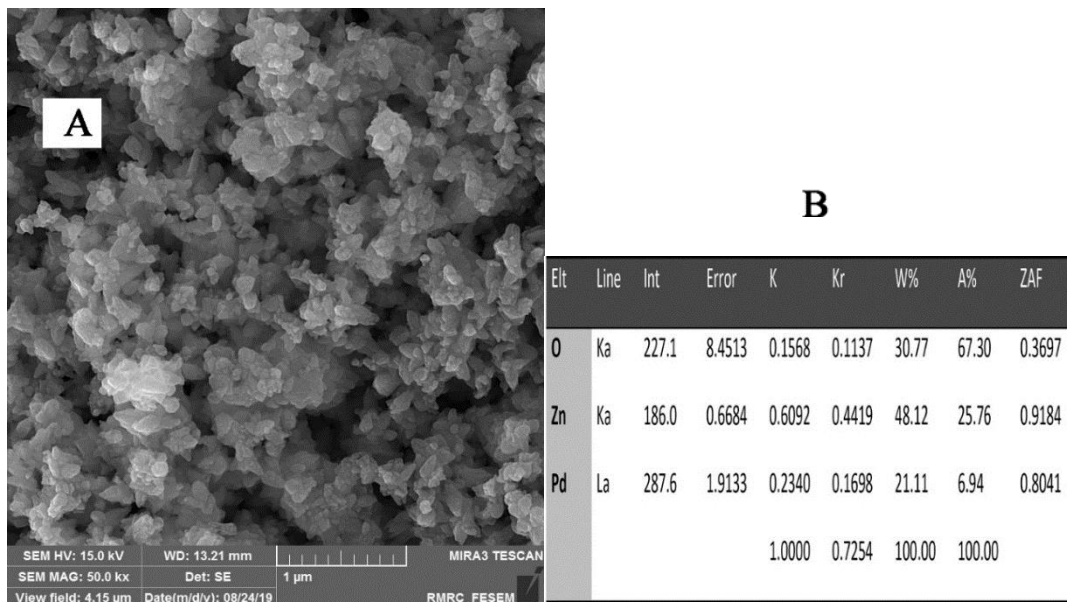

FIGURE 1 FESEM image of ZnO-Pd nanoparticle (A) and EDS analysis of ZnO-Pd nanoparticle (B)

The slope of this equation confirms the presence of H$^+$ in the electrooxidation mechanism of SY at the surface of ZnO-Pd/[C4mim]-[PF6]/PE with the equal value of electron in this mechanism (Scheme 1). On the other hand, the maximum oxidation signal of

SY occurred at pH=4.0 and this pH was selected as optimum condition as the best condition. The results confirm an irreversible behavior for electro-oxidation of SY in all of pHs.
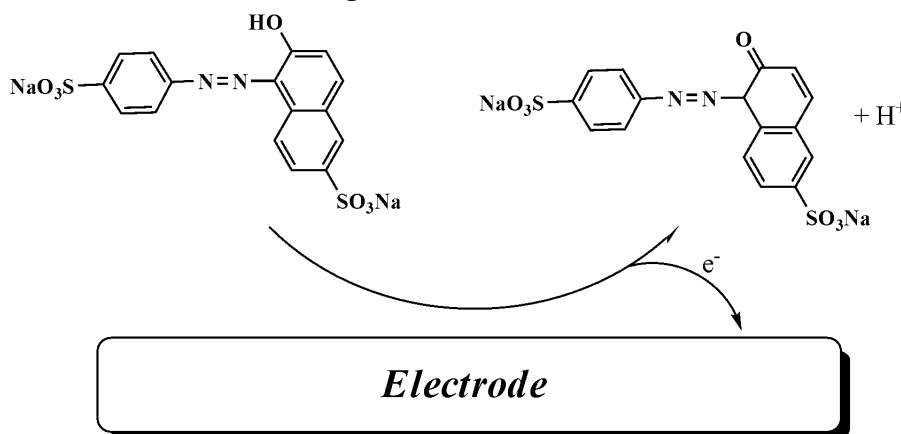
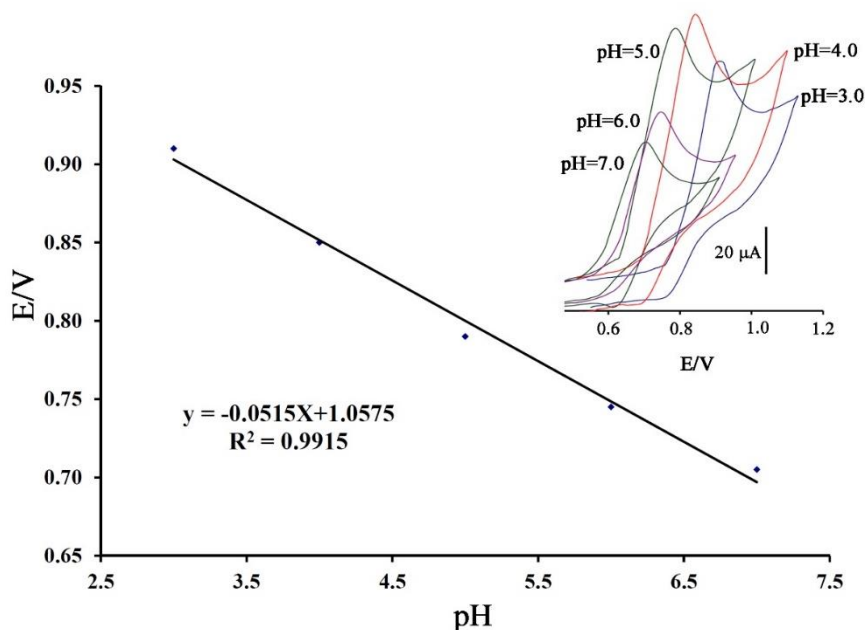**SCHEME 1** Suggestion mechanism for electrooxidation of SY

FIGURE 2 E-pH curve for electrooxidation of 500 µM SY at surface of ZnO-Pd/[C4mim]-[PF6]/PE. Cyclic voltammograms of500 µM SY at surface of ZnO-Pd/[C4mim]-[PF6]/PE in the pH range 3.0-7.0. Condition; scan rate 100 mV/s

The linear sweep voltammogram of 500.0 µM SY was recorded at the surface of ZnO-Pd/[C4mim]-[PF6]/PE (Figure 3 curve a), [C4mim]-[PF6]/PE (Figure 3 curve b), ZnO-Pd/PE (Figure 3 curve c) and PE (Figure 3 curve d) at pH=4.0 and scan rate 100 mV/s.

We recorded oxidation currents 49.52 µA, 72.67 µA, 103.45 µA, and 127.74 µA for electro-oxidation SY at the surface of PE, ZnO-Pd/[C4mim]-[PF6]/PE, [C4mim]-[PF6]/PE, and ZnO-Pd/[C4mim]-[PF6]/PE, respectively.
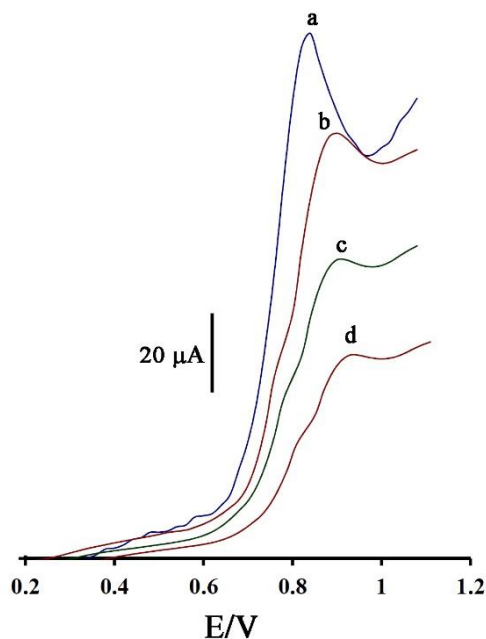

FIGURE 3 Linear sweep voltammogram of 500.0 µM SY was recorded at surface of ZnO-Pd/[C4mim]-[PF6]/PE (a), [C4mim]-[PF6]/PE (b), ZnO-Pd/PE (c) and PE (d) at pH=4.0 and scan rate 100 mV/s

As can be seen, with moving PE to ZnO-Pd/[C4mim]-[PF6]/PE, the oxidation signal of SY was improved 2.57 times. This point is relative to the high conductivity of ZnO-Pd nanoparticle and [C4mim]-[PF6]. The high surface area of ZnO-Pd nanoparticle and electrical conductivity of [C4mim]-[PF6] helps to improve the sensitivity of the fabricated sensors.

The oxidation signal of 350 μM SY was recorded in the scan rate range 20-250 mV/s at the surface of ZnO-Pd/[C4mim]-[PF6]/PE (Figure 4 inset). The linear relation with the equation I = 6.0392 $v^{1/2}$ + 44.4910 was recorded for this investigation that confirms the diffusion process for electrooxidation of SY at the surface of ZnO-Pd/[C4mim]-[PF6]/PE (Figure 4).
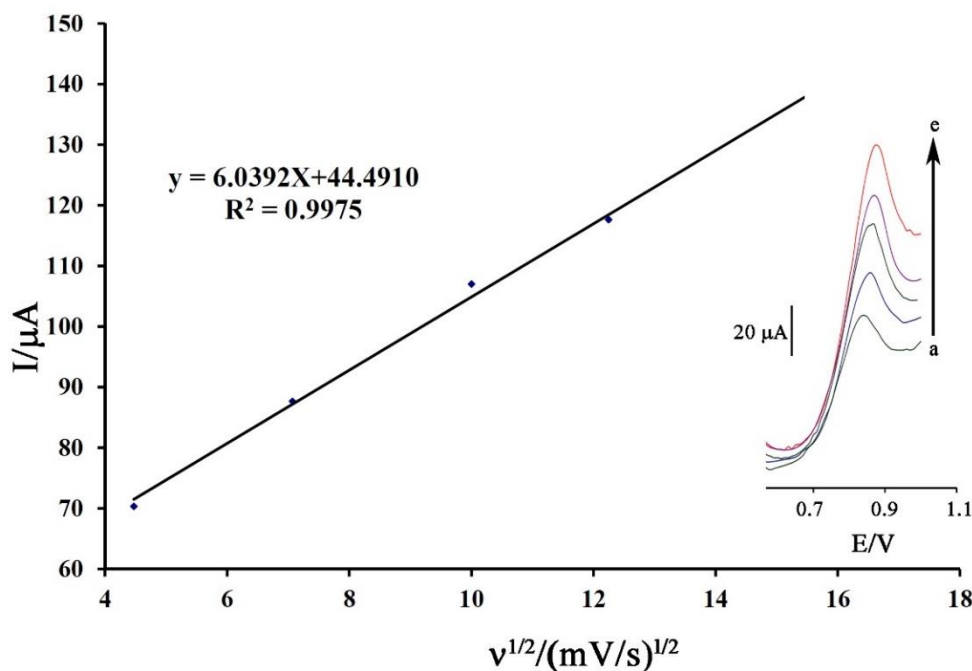

FIGURE 4 Plot of I-$v^{1/2}$ for electrooxidation of 350 μM SY at surface of ZnO-Pd/[C4mim]-[PF6]/PE in the scan rate a) 20; b) 50; c) 100; d) 150 and e) 250 mV/s

The value of the diffusion coefficient of SY was determined by recording chronoamperograms 700 μM, 800 μM and 900 μM SY at the surface of ZnO-Pd/[C4mim]-[PF6]/PE using applied potential 950 mV (Figure 5A). The Cottrell plots relative to electro-oxidation of SY are the presence of in figure 5 B and using obtained slopes the value of diffusion coefficient was determined 4.11 ×10^{-5} cm²/s.

The stability of ZnO-Pd/[C4mim]-[PF6]/PE for determination of 50 μM SY was checked in period time 45 days. We detected an 8% decreased in the oxidation signal of SY at the surface of ZnO-Pd/[C4mim]-[PF6]/PE in this period time that confirms good stability for fabricated ZnO-Pd/[C4mim]-[PF6]/PE as the electroanalytical sensor.
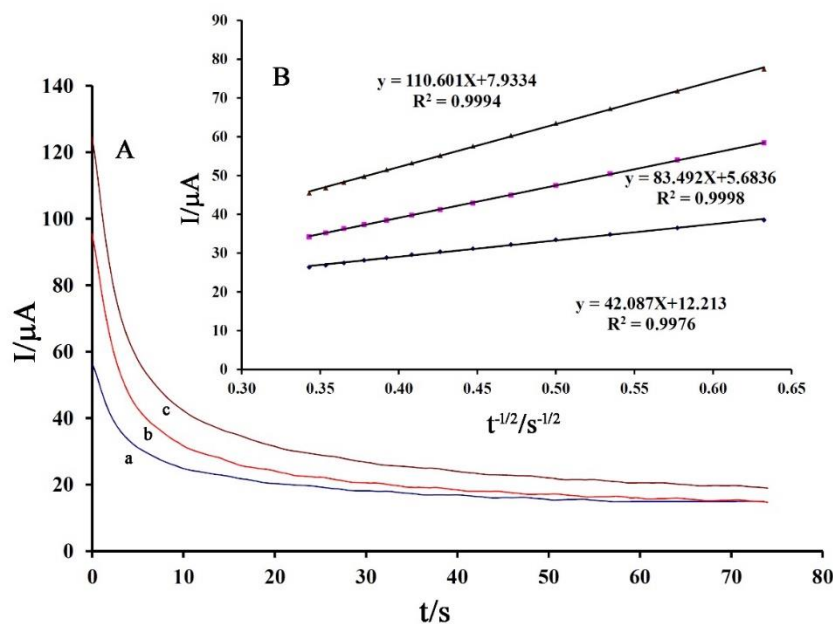
FIGURE 5 A) Chronoamperograms a) 700 μM, b) 800 μM and c) 900 μM SY at surface of ZnO-Pd/[C4mim]-[PF6]/PE. B) Cottrell plots relative to electro-oxidation of SY.

The linear dynamic range and detection limit of SY at the surface of ZnO-Pd/[C4mim]-[PF6]/PE was determined by recording square wave voltammograms at optimum condition (Figure 6 inset). Results showed a linear dynamic range between 0.001-280 μM with detection limit 0.4 nM for determination of SY at the surface of ZnO-Pd/[C4mim]-[PF6]/PE (Figure 6). The recorded linear dynamic range or detection limit for the determination of SY using ZnO-Pd/[C4mim]-[PF6]/PE is comparable to other reported sensors (please see table 1).
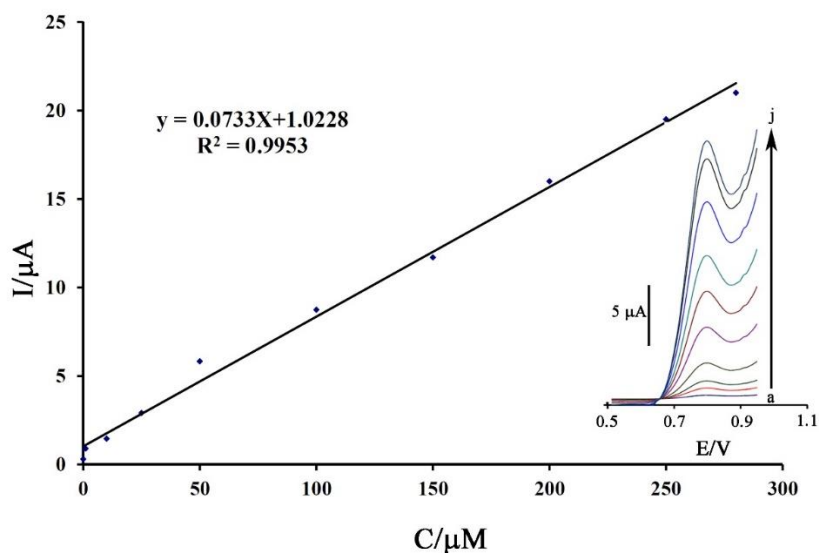

FIGURE 6 Current-concentration plot relative electro-oxidation of SY at the surface of ZnO-Pd/[C4mim]-[PF6]/PE. Inset) SW voltammograms of SY in the concentration range a) 0.001, b) 1.0, c) 10.0, d) 25.0, e) 50.0, f) 100.0, g) 150.0, h) 200.0, i) 250.0 and j) 280.0 μM at the surface of ZnO-Pd/[C4mim]-[PF6]/PE.

The selectivity investigation was studied by acceptable error 5% in current and potential in the presence of 15 µM SY at the surface of ZnO-Pd/[C4mim]-[PF6]/PE. The results showed 1000-fold of Ca^{2+}, NO_3^-, Br^-, and K^+ and 400-fold glycine, methionine and vitamin B_2, and 50-fold vitamin C don't show any interference for the determination of SY at the surface of ZnO-Pd/[C4mim]-[PF6]/PE that confirm good selectivity for the proposed sensor for the determination of SY in the present study.

Table 1 Comparison of the capability of ZnO-Pd/[C4mim]-[PF6]/PE and previous suggested sensor in the determination of SY

Electrode	Mediator	Linear dynamic range (µM)	Limit of detection (µM)	Ref.
Au	poly(aniline-co-o-anisidine-co-o-toluidine)/graphene oxide nanocomposite	5.0-500	0.142	[101]
Glassy carbon electrode	exfoliated graphene oxide	0.05-1.0	0.019	[102]
Glassy carbon electrode	Graphene oxide/multi wall carbon nanotubes composite	0.09-8.0	0.025	[103]
Graphite Paste Electrode	Attapulgite	0.0025-1.5	0.001	[104]
Carbon paste	ZnO-Pd/[C4mim]-[PF6]	0.001-280	0.0004	This work

Real sample analysis

The ability of ZnO-Pd/[C4mim]-[PF6]/PE for determination of SY in fruit juices, was tested for investigation of sensor ability in the real sample analysis. The orange juice and fruit juice were purchased from the local market and selected for this investigation. The recovery data for determination of SY using ZnO-Pd/[C4mim]-[PF6]/PE are presence in table 2. As can be seen, the acceptable recovery data confirm highly performance ability of ZnO-Pd/[C4mim]-[PF6]/PE for the determination of SY in real samples.

TABLE 2 Determination and recovery data of SY using ZnO-Pd/[C4mim]-[PF6]/PE in food samples (n=4)

Sample	Exist (mg/L)	Spiked (mg/L)	Found (mg/L)	Recovery %
Orange juice	25.22	---	26.11±1.11	---
	---	10.00	34.92±1.08	99.14
Fruit juice	---	30.00	31.42±1.25	104.73
	---	50.00	48.92±2.11	97.84
Drinking water	---	---	<Limit of detection	---
	---	20.00	20.85±1.01	104.25

Conclusion

In this investigation, we have reported an easy and one-pot synthesis procedure for synthesized ZnO-Pd nanoparticle with d<50 nm. The ZnO-Pd nanoparticle showed good electrical conductivity and was used for the fabrication of ZnO-Pd/[C4mim]-[PF6]/PE as a high-performance food electrochemical

sensor for the determination of SY. The ZnO-Pd/[C4mim]-[PF6]/PE improved electrochemical redox reaction SY about 2.57 times and used for determination of this food azo dye in the concentration range 0.001-280 μM with detection limit 0.4 nM. In addition, ZnO-Pd/[C4mim]-[PF6]/PE was showed the powerful ability for determination of SY in real food samples like orange and fruit juices.

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