

Full Paper

Carmoisine A Catalytic Film on The Surface of a One-use Electrode for the Detection of Hazardous Culinary Ingredients Tartrazine in the Existence of Vanillin

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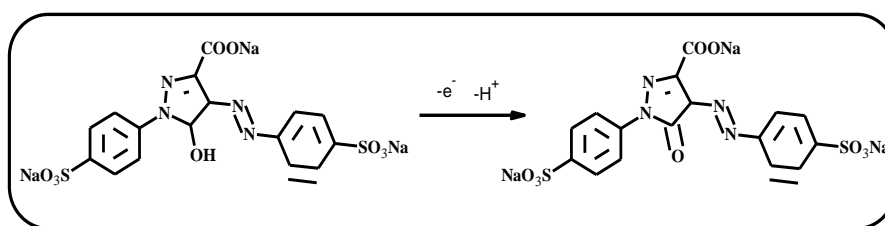
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Abstract- The present work aims to prepare a Carmoisine A polymer film on the surface of a disposable pencil graphite electrode to detect Tartrazine in the presence of Vanillin. The rise in surface area of the modified electrode using potassium ferrocyanide system through a comparative with bare electrode employing cyclic voltammetric technique confirms the catalytic film on the surface of the pencil graphite electrode. The prepared electrode was used to analyze culinary ingredients tartrazine and vanillin, both of which can cause severe health issues when consumed in excess. Tartrazine is a food coloring agent, while vanillin serves as a flavoring agent. Cyclic voltammetry (CV) and differential pulse voltammetry (DPV) techniques were employed for the electrochemical analysis of tartrazine on Poly(CARA)-modified pencil graphite electrode (MPGE). The CV method was used to study the effects of scan rate, pH, and simultaneous analysis, while the DPV method was employed for investigating concentration effects and interference. The electrode process suggests an adsorption-controlled mechanism and the pH study indicates that electrons and protons are equally shared during the reaction. The simultaneous analysis demonstrates that Poly(CARA)-MPGE can detect both tartrazine and vanillin in a binary food additive mixture. The selectivity study shows that tartrazine and vanillin can be quantified in the binary mixture at different concentrations. The limits of detection (LOD) and quantification (LOQ) for tartrazine on Poly(CARA)-MPGE were 0.852 μM and 2.84 μM , respectively, with a good stability of 89% over 25 cycles.

Keywords- Tartrazine; Vanillin; Carmoisine A; Pencil graphite electrode; Electro-polymerization

1. INTRODUCTION

Culinary ingredients are the substances utilized in making food products. They are intended to add nutritional value, enhance flavors, and improve the aesthetic appearance of the food [1]. Food dyes are common ingredients to enhance food products' aesthetics, freshness, and taste [2]. However, food dyes can negatively affect health due to their potential toxic nature [3]. Additionally, food color can stimulate human appetite, increasing food product sales. The use of food colorants dates back to ancient Egyptian civilization, where they were used to enhance the appearance of wine and confections. Initially, flower petals were used as food colorants, but now synthetic chemical compounds are commonly employed to enhance the color of food products. Tartrazine (TZ) is one of the most widely used food dyes, and its use has increased in recent years due to its vibrant appearance, low cost, and ease of availability (Scheme 1). However, TZ can harm human health when exposure exceeds the daily intake level of 7.5 mg/kg of body weight. Usage of TZ above the advisable amount can cause various health effects, such as effects on digestive enzymes [4], clastogenic effects [5], cytotoxic and mutagenic effects [6], biochemical effects [7], and embryonic effects [8] etc. are the major hazardous effects.



Scheme 1. Reaction of Tartrazine

What we see, and what we smell will affect what we eat, after the appearance, the odor is major attractions one can have. The flavoring agents add aroma and taste to the food products and intensify the appetite [9]. Vanillin (VN) is one of the major flavoring agents that is naturally available and can be synthesized in the laboratory [10]. The VN flavor is mainly used in food products, aromatherapy, and pharmaceuticals (to improve the palatability of medication). The natural VN was extracted from cured pods of vanilla orchids, non-toxic, but synthetic VN exposed above the daily intake level of 0.1 mg/kg body weight [11]. The higher intake of VN can be harmful as it causes, nausea, headache, and vomiting, and malignant cells can be induced.

Carmoisine A (CARA) is a synthetic edible azo dye commonly used in food beverages, alcoholic beverages, chewing gums, ice creams, and cakes [12]. TZ was previously validated using electrochemical sensors such as ZnO/MIP-PArg [13], AuNPs/rGO/AEP/SPE [14], and H₂N-MIL-101(Cr)-CPE [15]. The naval conductive polymer film from CARA was formed from the electropolymerization method using a pencil graphite electrode. In the present work,

a CARA polymer film was formed as a modifier on the surface of a disposable pencil graphite electrode to analyze TZ in the presence of VN.

2. EXPERIMENTAL SECTION

2.1. Required chemicals and apparatus

Tartrazine, Vanillin, and Carmoisine A, are brought from Nice Chemicals, Na_2HPO_4 , NaH_2PO_4 , KCl , and $\text{K}_4[\text{Fe}(\text{CN})_6]$ are procured from Himedia. The electrochemical studies were carried out using the potentiostat CHI 660c. All the experiments were carried out at lab temperature.

2.2. Fabrication of electrode

The pencil graphite electrode was used as a bare pencil graphite electrode (BPGE). The modified pencil graphite electrode was prepared using 10 mM CARA in the presence of 0.2 M NaOH , employing the CV technique. The CV was conducted over a potential range of -0.2 to 1.2 V for various cycles. As the cycles progressed, the current decreased, and the modified electrode was prepared for different polymer cycles: 5, 10, 15, 20, 25, 30, 35, and 40. The electrodes were then immersed in potassium ferrocyanide to determine the optimum number of polymer cycles of CARA, from 5 to 35 cycles, the current increased, while it decreased at 40 cycles, as shown in Figure S1(B). The electrode with a polymer film corresponding to 35 cycles of CARA [Poly(CARA)-MPGE] exhibited the optimum catalytic activity, as represented in Figure S1(A) [16].

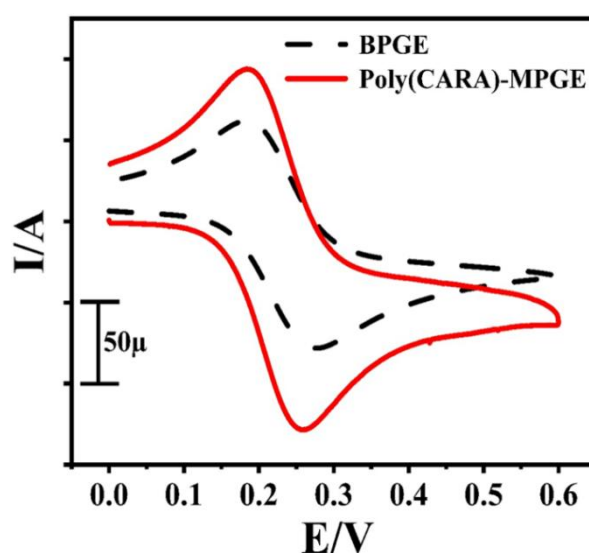


Figure 1. CVs for the comparative electrochemical activity for 25×10^{-4} $\text{K}_4[\text{Fe}(\text{CN})_6]$ at Poly(CARA)-MPGE using 1M KCl as supporting electrolyte

3. RESULTS AND DISCUSSION

3.1. Electrochemical characterization of Poly(CARA)-MPGE

In Figure 1 CVs were represented for 25×10^{-4} M $K_4[Fe(CN)_6]$ using 1 M KCl as a supporting electrolyte at Poly(CARA)-MPGE and BPGE. The solid red line represents the CV $K_4[Fe(CN)_6]$ at Poly(CARA)-MPGE and the black dashed line represents the same at BPGE. The current at Poly(CARA)-MPGE was increased compared to BPGE due to the increased catalytic activity of the fabricated electrode on the surface of the pencil graphite electrode. The area of the fabricated electrode was found to be 0.042 Cm^2 and the area of the bare electrode was 0.026 Cm^2 as determined by Randles-Sevick's equation [17–22].

3.2. pH study of TZ at Poly(CARA)-MPGE

The Poly(CARA)-MPGE was immersed in $10 \mu\text{M}$ TZ using a supporting electrolyte at different pH levels (6.2, 6.6, 7.0, 7.4, and 7.8) prepared from 0.2 M PBS, employing the CV method, as represented in Figure 2(A). The peak potential decreases simultaneously with the increase in pH, as shown in Figure 2(B). The phenomenon of anodic peak potential shift produces a straight line, and the linear regression of this line is given by the equation $E_{pa} = 1.237 - 0.0463 (\text{pH})$ ($R^2 = 0.997$) [23,24]. The slope of the graph, E_{pa} versus pH was found to be 46 mV, which is close to the theoretical slope of 59 mV, indicating the transfer of a 1:1 ratio of protons and electrons during the reaction.[25–27] Figure 2(C) illustrates the oscillation of the anodic peak current due to changes in the pH environment. The pH was selected as the optimum supporting electrolyte condition due to its higher sensitivity towards TZ at Poly(CARA)-MPGE, and the subsequent electrochemical studies were carried out at pH 7.0 [28,29].

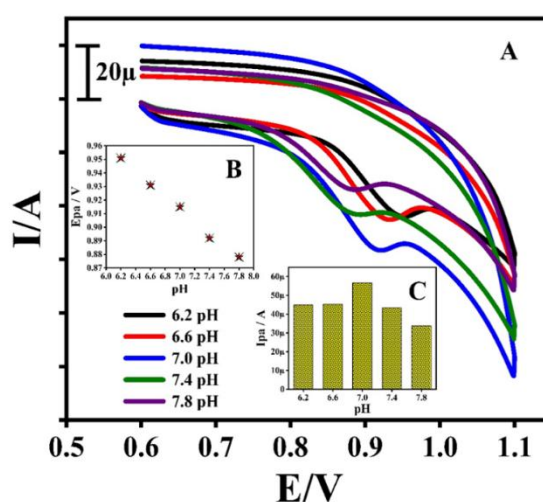


Figure 2. (A) CVs for the Variation of pH (6.2, 6.6, 7.0, 7.4, and 7.8) for $10 \mu\text{M}$ TZ at Poly(CARA)-MPGE using PBS solution; (B) Graph of E_{pa} versus pH; (C) Graph of I_{pa} versus pH

3.3. Relative Electrochemical study of TZ at Poly(CARA)-MPGE and BPGE

The CV method was employed for the comparative electrochemical study at Poly(CARA)-MPGE and BPGE of 10 μ M TZ using 0.2 M PBS of 7.0 pH, as represented in Figure 3. The solid line (red) represents the CV of TZ at Poly(CARA)-MPGE. In contrast, the dashed line (black) means the same at BPGE. Poly(CARA)-MPGE's higher sensitivity compared to BPGE is to the catalytic polymer film formed on the surface of the disposable electrode [30–35].

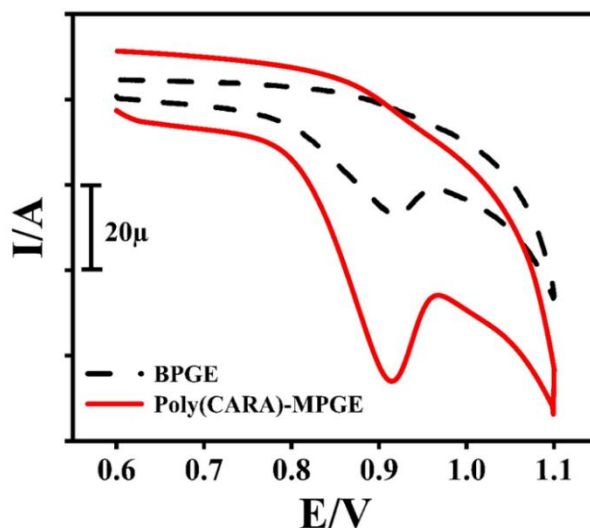


Figure 3. CVs for the comparative electrochemical study for TZ at Poly(CARA)-MPGE and BPGE using 0.2 M PBS of pH 7

3.4. Consequence of sweep rate variation

The CV study examines the effect of varying the sweep rate (0.05–0.5 V/s) for 10 μ M TZ at Poly(CARA)-MPGE using a pH 7.0 atmosphere, as indicated in Figure S2(A). The Peak current increases as the sweep rate rises, accompanied by a slight shift in peak potential. The graph of I_{pa} versus $v^{1/2}$, shown in Figure S2(B), yields a straight line, with the linear regression represented by the equation $I_{pa} = 2.939 (10^{-4}) v^{1/2} - 2.995 (10^{-5})$ ($R^2=0.997$) [36]. Figure S2(C) illustrates the graph of E_{pa} versus $\log(v)$, demonstrating that the peak potential shifts as the scan rate increases; this is a straight line with the regression equation $E_{pa} = 0.942 + 0.0265 \log(v)$ with ($R^2=0.992$) [37]. The graph of $\log(I_{pa})$ versus $\log(v)$ is shown in Figure S2(D), which has a linear regression equation of $E_{pa} = 3.542 + 0.662 \log(v)$ ($R^2=0.998$) [38]. From the above data, the slope was found to be 0.662, indicating that the electrode undergoes an adsorption-controlled process [39].

3.5. Result of variable concentration

The DPV method was used to determine the outcome of TZ concentration at Poly(CARA)-MPGE, using 0.2 M PBS at pH 7.0, as represented in Figure 4(A). The peak current increases as the concentration of TZ rises from 1 to 12 μ M at Poly(CARA)-MPGE. This increase in

current follows a straight line, with the linear regression equation $I_{pa} = 3.869 (10^{-6}) (C_{TZ}) + 3.061 (10^{-5})$ ($R^2=0.997$), [40] as shown in Figure 4(B). The limit of detection (LOD) and quantification (LOQ) values, calculated from the same graph, were found to be $0.852 \mu\text{M}$ and $2.84 \mu\text{M}$, respectively [41–46].

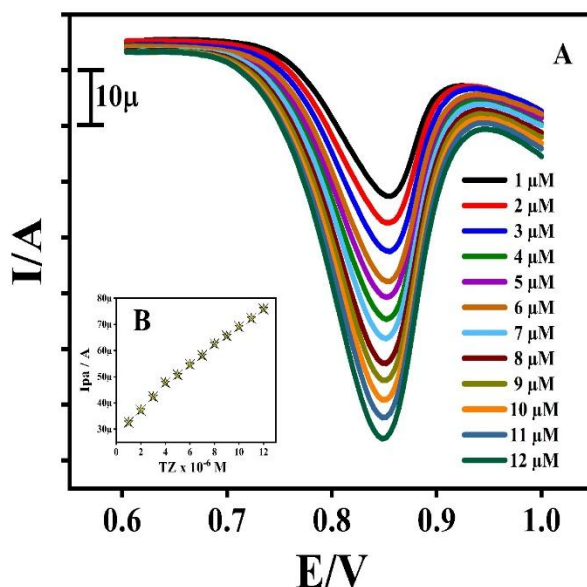


Figure 4. (A) DPVs for the variation of TZ concentration (1-12 μM) at Poly(CARA)-MPGE using 0.2 PBS of 7.0 pH; (B) Graph of I_{pa} versus concentration of TZ

3.6. Solidity of Poly(CARA)-MPGE

The CV method determines the solidity of Poly(CARA)-MPGE using $10 \mu\text{M}$ TZ employing pH 7.0 of PBS as a supportive electrolyte as indicated in Figure S3. The solidity was calculated using the formula I_{pn}/I_{p1} , [47] I_{pn} is the peak current of the n^{th} cycle and I_{p1} is the peak current of the 1st cycle. The solidity of the Poly(CARA)-MPGE was found to be 89% over 25 repeated cycles, this represents the fouling resistance ability of fabricated electrode for TZ [48].

3.7. Synchronous study of TZ and VN at Poly(CARA)-MPGE

The CV and DPV methods are employed for the simultaneous study of $10 \mu\text{M}$ TZ and $10 \mu\text{M}$ VN at Poly(CARA)-MPGE using 0.2 M PBS at pH 7.0, as represented in Figure 5(A) and (B). The solid red line signifies the curve for TZ at Poly(CARA)-MPGE, while the dashed black line signifies the same at BPGE. The current amplified nearly twofold for Poly(CARA)-MPGE compared to BPGE. This increase in sensitivity for both culinary ingredients in a mixture, along with peak separation, is attributed to the catalytic ability of the polyfilm [49–51].

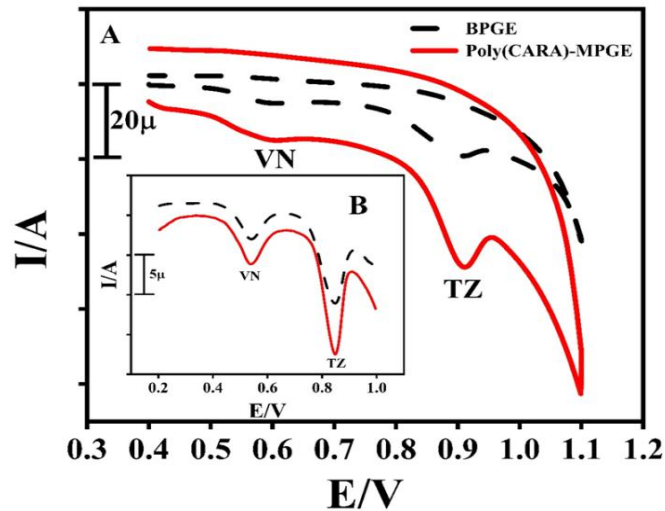


Figure 5. (A) CVs for the comparative Simultaneous study of 10 μM TZ and 10 μM VN using 0.2 PBS of 7.0 pH at Poly(CARA)-MPGE and BPGE; (B) DPVs for the comparative Simultaneous study of 10 μM TZ and 10 μM VN using 0.2 PBS of 7.0 pH at Poly(CARA)-MPGE and BPGE

3.8. Selective studies for TZ and VN at Poly(CARA)-MPGE

DPV technique was employed for the selective study of TZ and VN at Poly(CARA)-MPGE using 0.2 M PBS of pH 7.0 as represented in Figure 6 and Figure S4. The rise in the concentration of TZ (10-50 μM) in the presence of 10 μM VN was represented in Figure 6(A).

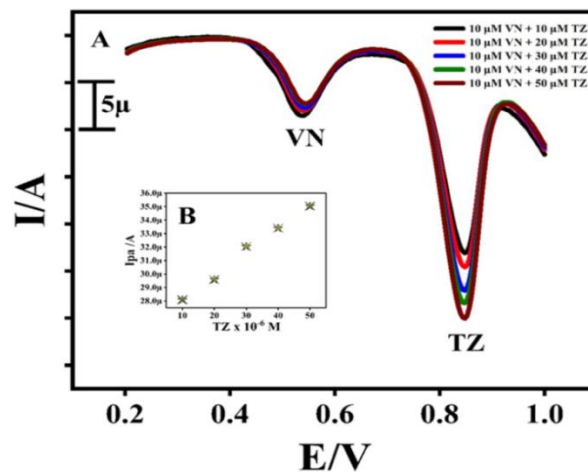


Figure 6. (A) DPVs for the selectivity study for TZ (10-50 μM) and VN (10 μM) at Poly(CARA)-MPGE using 0.2 M PBS 7.0 pH. (B) Graph of I_{pa} versus concentration of TZ

The peak current of TZ increases as we increase the concentration of TZ by taking VN concentration as a constant. The peak current of VN was not disturbed a bit which represents the selective ability of the modified electrode. The increase in peak current of TZ with

an increase in concentration gives a straight line having a regression equation of $I_{pa} = 1.771 (10^{-7}) (C_{TZ}) + 2.632 (10^{-5})$ with the $R^2=0.996$ as represented in Figure 6(B) [52–54].

Figure S4(A) shows the increase in the concentration of VN (10-50 μM) while maintaining a constant concentration of TZ (10 μM). The peak current of VN increased linearly with concentration, while the peak current of TZ remained relatively steady, showing negligible fluctuations. The linear regression of this relationship is depicted in Figure S4(B), with the equation $I_{pa} = 1.914 (10^{-7}) (C_{VN}) + 1.19 (10^{-5})$, ($R^2=0.997$) [55]. In the selective study, the results indicate that the fabricated electrode can be used to analyze TZ and VN in the presence of either of these compounds at a given concentration [56–59].

Table 1 compares the LODs and detection methods of other previous reports [60-63] in electrochemical determination of TZ.

Table 1. LOD of TZ with other reported electrodes

S.I no	Electrodes	Reaction	Method	LOD=3s/m(μM)	Reference
1	CNF/AuNP-CPE	Oxidation	DPV	2.64	[60]
2	Gr/PLPA/PGE	Oxidation	DPV	1.54	[61]
3	SDSMCPE	Oxidation	CV	5.2	[62]
4	PGMCPE	Oxidation	CV	2.045	[63]
5	Poly(CARA)-MPGE	Oxidation	DPV	0.852	Present work

4. CONCLUSION

The modified electrode was fabricated using CARA through the electro-polymerization method employing the CV technique. The prepared Poly(CARA)-MPGE exhibited high sensitivity toward TZ, and the electrode underwent an adsorption-controlled process, as determined by the scan rate study. The pH study revealed that the reaction involves sharing an equal number of electrons and protons. The simultaneous elucidation of TZ and VN demonstrated a higher current, and the selectivity study between these two culinary ingredients showed good peak separation. The electrode exhibited a good LOD and LOQ of 0.852 μM and 2.84 μM for TZ, with excellent stability of 89% toward TZ over 25 cycles. This invention can be utilized to determine real samples of culinary ingredients TZ and VN in a binary blend or any other food dye.

Declarations of interest

The authors declare no conflict of interest in this reported work.

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