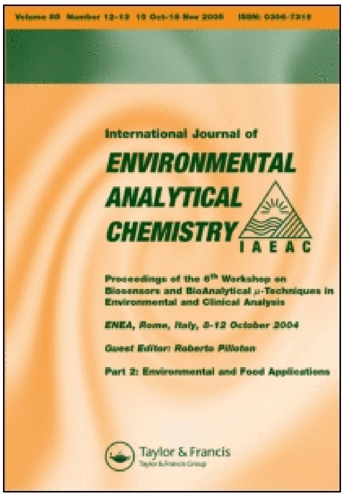

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A highly sensitive electrochemical method for the determination of Sudan I at polyvinylpyrrolidone modified acetylene black paste electrode based on enhancement effect of sodium dodecyl sulphate

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A highly sensitive electrochemical method for the determination of Sudan I at polyvinylpyrrolidone modified acetylene black paste electrode based on enhancement effect of sodium dodecyl sulphate

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A novel electrochemical method for the sensitive determination of trace Sudan I at a polyvinylpyrrolidone (PVP) modified acetylene black carbon paste electrode (PVP/CPE) based on the enhancement effect of sodium dodecyl sulphate (SDS) was reported. Compared with the poor response at a bare acetylene black paste electrode (CPE), a well-defined oxidation peak of Sudan I was observed at the PVP modified CPE. The oxidation peak current of Sudan I was further enhanced with the addition of SDS, which was attributed to the enhancement effect of SDS at PVP/CPE. Under optimal working conditions, the oxidation peak current of Sudan I was proportional to its concentration in the range of 2.0×10^{-7}–$8.0 \times 10^{-6}\,\mathrm{mol\,L^{-1}}$, with a low detection limit of $1.0 \times 10^{-8}\,\mathrm{mol\,L^{-1}}$. The proposed method was successfully applied to the detection of Sudan I in chilli products.

Keywords: Sudan I; polyvinylpyrrolidone; sodium dodecyl sulphate; acetylene black paste electrode; enhancement effect

1. Introduction

Sudan I (1-phenylazo-2-naphthalenol), also known as CI Solvent 14, was an industrial red dye involved in colouring solvents, oils, waxes, petrol, shoe and floor polishes. Sudan I was potentially genotoxic according to the International Agency for Research on Cancer (IARC) [1–3]. Experiments on rats showed that Sudan I was a dose-related higher incidence of neoplastic liver nodules, which were precursors to cancer [4]. Apparently, Sudan I was not appropriate as a food additive. In South Africa, Sudan I was prohibited in food products by the Regulations Relating to Food Colourants (R.1008) of the Foodstuffs, Cosmetics and Disinfectant Act 54 of 1972, and Sudan I has been banned as food colourant throughout the European Union since 2003. However, Sudan I has still been used illegally by some companies to manufacture chilli powder or other foodstuffs to maintain or enhance the colour of the food products. Therefore, the quantitative determination of Sudan I was of great significance.

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Several methods have been proposed for the determination of Sudan I, including spectrophotometry [5], gas chromatography [6] and high-performance liquid chromatography (HPLC) [7–9]. Compared with these methods, electrochemical techniques could offer faster and cheaper analysis. Du reported the detection of Sudan I based on its reduction at activated glassy carbon electrode [10], while Lin reported its oxidation determination at montmorillonite calcium modified carbon paste electrode (CPE) [11]. The former showed a linear range of 2.4×10^{-6} to $1.8 \times 10^{-5}\,\mathrm{mol\,L^{-1}}$ with a detect limit of $7.1 \times 10^{-7}\,\mathrm{mol\,L^{-1}}$, and the latter showed a linear range of 2.0×10^{-7} to $4.0 \times 10^{-6}\,\mathrm{mol\,L^{-1}}$ with a detect limit of $8.0 \times 10^{-8}\,\mathrm{mol\,L^{-1}}$. The two proposed methods were all applied for the determination of Sudan I in real samples.

Cross-linked polyvinylpyrrolidone (PVP) was a water-insoluble polymer that possessed strong adsorption capacity towards phenolic compounds due to the hydrogen bonding between imide moieties of polymer and hydroxyl group in phenolic compounds. In this work, we developed a sensitive method for Sudan I determination based on the enhancement effect of sodium dodecyl sulphate (SDS) at PVP (cross-linked) modified acetylene black paste electrode. The experimental results showed that PVP significantly facilitated the oxidation of Sudan I at CPE, which might arise from the strong hydrogen bonding interactions between PVP and Sudan I. The addition of SDS was able to further improve the response of Sudan I, which is probably due to the synergistic interactions between Sudan I, SDS and PVP. The adsorptive behaviour and the enhancement effect of surfactant at CPEs have been systematically investigated by Hu *et al.* which has been proven to be an efficient and versatile electrochemical platform for sensitive determination of various species, including diethylstilbestrol [12], tetracycline [13] and thyroxine [14]. The sensitive response of Sudan I at PVP/CPE in the presence of trace SDS provided an efficient method for the determination of Sudan I in various environments including foods.

2. Experimental

2.1 *Apparatus and reagents*

All the electrochemical measurements were performed with a CHI 830 electrochemical analyser (Shanghai Chenhua Co., China) in a three-electrode system. The working electrode was a PVP modified CPE. A Pt wire and a saturated calomel electrode (SCE) were used as the counter and reference electrodes, respectively.

Acetylene black was obtained from Shanghai Reagent Corporation, China. PVP (cross-linked) was purchased from Acros. Chilli products were purchased from the local market. Sudan I (New Jersey, USA) was dissolved in acetone to form a stock solution of $1.0 \times 10^{-3}\,\mathrm{mol\,L^{-1}}$ and stored at 4°C. All surfactants including SDS were purchased from Shanghai Reagent Corporation, China, and were dissolved in double-distilled water to form $1 \times 10^{-2}\,\mathrm{mol\,L^{-1}}$ homogeneous solutions. All chemicals were used without further purification. Doubly distilled water was used to prepare all solutions except for special statements.

2.2 *Preparation of the PVP modified CPE*

The PVP modified acetylene black paste electrode was prepared as follows: 200 mg acetylene black, 100 mg PVP and 150 μL paraffin oil were mixed and ground for at least 0.5 h by hand in a small mortar to form a homogeneous acetylene black mixture. The mixture was pressed into the end cavity of a homemade poly (tetrafluoroethylene) (PTFE) cylindrical electrode body (2.0 mm i.d.). The resulting electrode was polished manually on a piece of weighting paper to get a smooth surface, which was denoted as PVP/CPE. Bare CPE was prepared in a similar way except for the absence of PVP.

2.3 *Experimental procedures*

Electrochemical experiments were carried out in a conventional electrochemical cell containing 5 mL of 0.1 mol L^{-1} phosphate buffer (pH 3.0) as the supporting electrolyte. Accumulation was carried out at open circuit for 400 s under continuous stirring, and then kept quiet for 5 s. Voltammograms were recorded at a scan rate of 0.1 V s^{-1} in the potential range of −0.40–0.60 V. After each measurement, the modified electrode was refreshed by polishing manually at weighting paper or refilling the electrode with new paste materials when necessary.

3. Results and discussion

3.1 *Enhanced voltammetric response of Sudan I at PVP/CPE by SDS*

Figure 1 illustrates the voltammetric responses of 4.0×10^{-6} mol L^{-1} Sudan I at different electrodes. No obvious peak of Sudan I was observed at bare CPE (curve a). Whereas, after the modification of CPE by PVP, a well-shaped oxidation peak at about 0.23 V (curve b) was obtained. With the addition of SDS, a similar oxidation peak of Sudan I appeared at CPE (curve c), which was greatly enlarged and a sharp-shaped oxidation peak

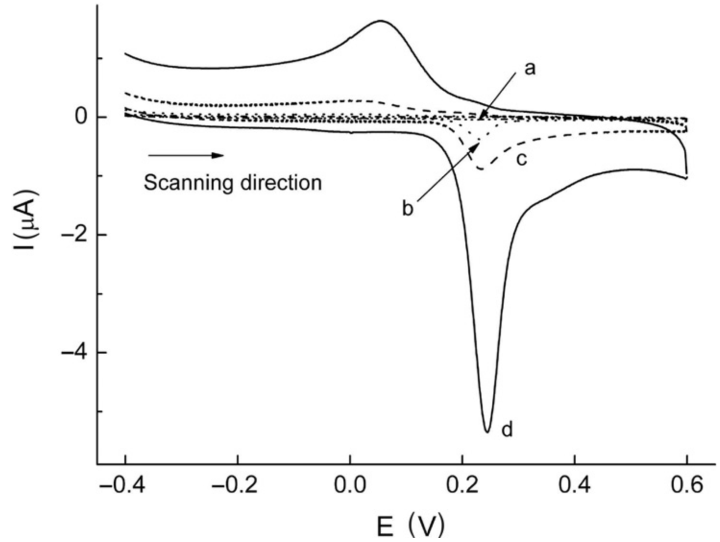

Figure 1. Cyclic voltammetric responses of 4.0×10^{-6} mol L^{-1} Sudan I at bare CPE (a, c) and PVP/CPE (b, d) in the absence (a, b) and presence (c, d) of 1.0×10^{-4} mol L^{-1} SDS in 0.1 mol L^{-1} PB (pH 3.0). Scan rate: 0.1 V s^{-1}.

was obtained when CPE was modified with PVP (curve d). In comparison with the indiscernible response at CPE, the significantly enhanced oxidation peak current of Sudan I at PVP/CPE in the presence of SDS indicated the synergistic enhancement effect of PVP and SDS for the electrochemical response of Sudan I at CPE.

As a polymeric sorbent, PVP comprised of a hydrophobic backbone and plenty of short branches of heterocyclic groups. The latter had a strong ability to form hydrogen bonds with other species. PVP doped in acetylene black paste provided many binding sites for the adsorption of Sudan I on the surface of the electrode, leading to the accumulation of Sudan I on the electrode surface and the enhanced current responses. Additionally, the blending of PVP in acetylene black paste might alter the composition and the surface properties of CPE, and influence the rate of electron transfer between the dye and the electrode surface. This was supported by the fact that the background current of PVP modified CPE is apparently larger than that of bare CPE. The large difference of the peak current at the bare and the PVP modified CPE demonstrated that the enhanced oxidation of Sudan I was based on the interaction of the dye molecule with PVP.

Hu *et al*. [15–17] have systematically investigated the influence of surfactants on the determination of many substances and a 'synergistic adsorption' mechanism was proposed to interpret the enhancement effects of surfactants. In this work, SDS might combine with Sudan I in certain forms, which facilitated the electron transfer between the electrode and the solution. As a conjugated planar molecule, Sudan I molecules might penetrate into the layer of SDS and vertically adsorb onto the electrode surface, while in the absence of SDS, Sudan I molecule might flatly adsorb onto the electrode. Apparently, the former could result in higher accumulation efficiency on the electrode surface, which in turn led to a higher current response of Sudan I.

Based on above discussions, three adsorption models were proposed for the adsorptive and the electrochemical behaviours of Sudan I on different electrodes (Figure 2): (a) few Sudan I molecules can be adsorbed onto bare CPE due to the lack of binding sites; (b) Sudan I molecules might flatly adsorb onto PVP/CPE and be accumulated on the electrode surface through hydrogen bonding; (c) Sudan I molecules might be more effectively accumulated on the surface of PVP/CPE through the possible intercalation in the SDS adsorption layer via hydrophobic interactions.

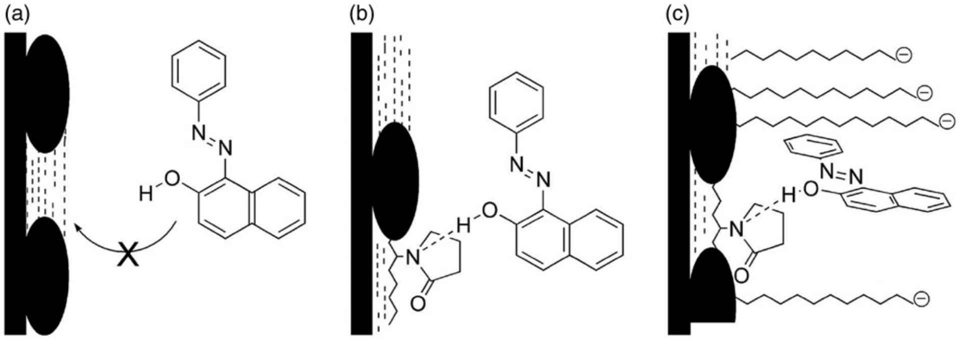

Figure 2. Models for the adsorption of Sudan I at CPE (a) and PVP/CPE (b, c) in the absence (a, b) and presence of SDS (c).

3.2 *Electrochemical behaviour of Sudan I at PVP/CPE*

Scan rate could influence the current responses of Sudan I and some important electrochemical parameters could be deduced from the relationship between the scan rate and the redox potential or current of Sudan I. The dependence of oxidation peak current of $4.0 \times 10^{-6}\,\mathrm{mol\,L^{-1}}$ Sudan I on scan rate at PVP/CPE in the presence of $1.0 \times 10^{-4}\,\mathrm{mol\,L^{-1}}$ SDS is illustrated in Figure 3. Both the oxidation and the reduction peak currents are linear with scan rate (v) in the range of 0.02–0.20 $\mathrm{V\,s^{-1}}$ (Equations (1) and (2)), suggesting an adsorption-controlled process of Sudan I oxidation.

$$I_{\mathrm{pa}} = 9.00\,v + 1.073(R = 0.9976) \tag{1}$$

$$I_{\mathrm{pc}} = -2.21\,v - 0.333(R = 0.9974) \tag{2}$$

The oxidation peak potential shifted positively with the increase of scan rate in the range of 0.020–0.20 $\mathrm{V\,s^{-1}}$, which showed a linear relationship with the logarithm of scan rate ($\ln v$):

$$E_{\mathrm{pa}} = 0.02198\ \ln\ v + 0.2961(R = 0.9992) \tag{3}$$

In the case of an adsorption-controlled electrode process, E_{p} could be defined by the following equation [18] at high scan rates:

$$E_{\mathrm{p}} = E^0 + (RT/\alpha n_\alpha F)\ln(RTk^0/\alpha n_\alpha F) + (RT/\alpha n_\alpha F)\ln\ v, \tag{4}$$

where E^0 is the formal potential, T is the temperature, α is the transfer coefficient and n_α is the number of the electron transfer in the rate-determining step, k^0 is the electrochemical rate constant and F is the Faraday constant. From Equations (3) and (4), we can calculate that the value of αn_α was 1.17. Thus, the number of electrons n_α was about 2 when α was

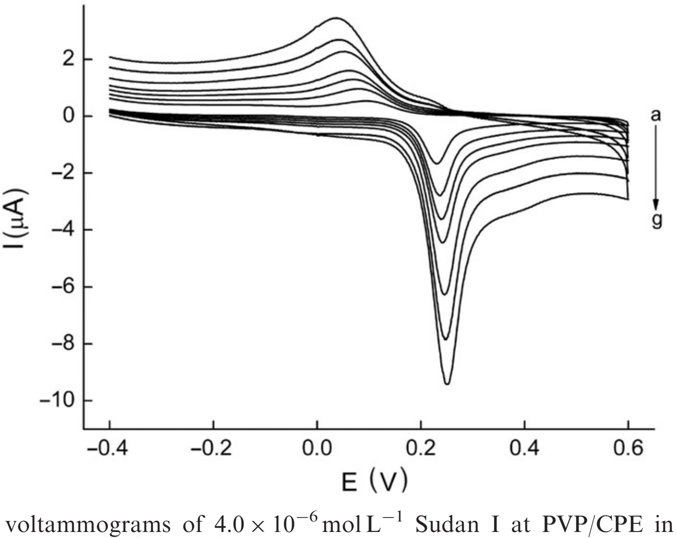

Figure 3. Cyclic voltammograms of $4.0 \times 10^{-6}\,\mathrm{mol\,L^{-1}}$ Sudan I at PVP/CPE in 0.1 $\mathrm{mol\,L^{-1}}$ PB (pH 3.0) containing $1.0 \times 10^{-4}\,\mathrm{mol\,L^{-1}}$ SDS at different scan rates (from a to g): 0.02, 0.04, 0.06, 0.12, 0.15 and 0.2 $\mathrm{V\,s^{-1}}$.

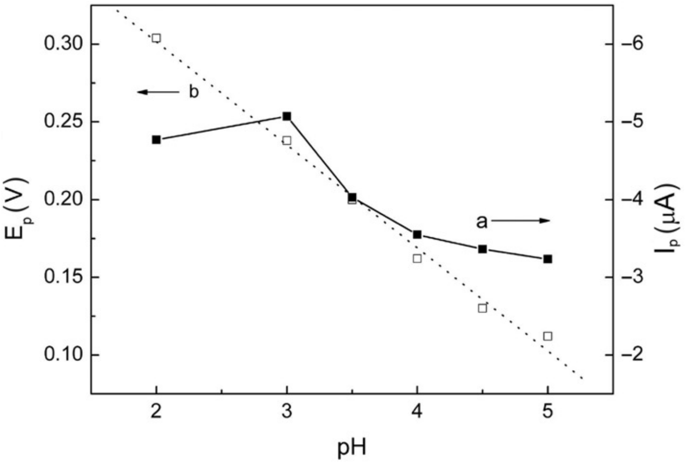

Figure 4. Effects of solution pH on the peak current (I_p) and the peak potential (E_p). Scan rate: $0.1\,V\,s^{-1}$. Other conditions are the same as in Figure 3.

assumed as 0.5. The origin of the oxidation response of Sudan I was still unclear because both the –OH and the –N=N– groups might be involved in the oxidation reaction. Though it was much more probable for the naphthyloxy group to be oxidised in this oxidation process, there was no sufficient experimental data to support this assumption.

The influence of solution pH on the voltammetric behaviours of Sudan I was also examined (Figure 4). It can be seen that the oxidation peak current of Sudan I increases in the range of pH 2.0–3.0 and then decreases from pH 3.0 to 5.0 (curve a). The peak potential shifted linearly to the negative direction with the increasing solution pH (curve b) and a slope of 0.066 V/pH was obtained, suggesting that the number of electrons involved in the oxidation of Sudan I was equal to that of protons. Therefore, Sudan I underwent a two proton–two electron oxidation process at the PVP/CPE.

3.3 *Chronocoulometry*

Chronocoulometry was used to characterise the oxidation of $4.0 \times 10^{-6}\,mol\,L^{-1}$ Sudan I at the PVP modified CPE in the presence of $1.0 \times 10^{-4}\,mol\,L^{-1}$ SDS and to determine the diffusion coefficient D and Q_{ads} according to the Anson equation [19]:

$$Q = 2nFAcD^{1/2}\pi^{-1/2}t^{1/2} + Q_{dl} + Q_{ads}, \tag{5}$$

where A is the apparent area of the acetylene black paste electrode, c is the concentration of Sudan I, D is the diffusion coefficient and Q_{ads} is the adsorption charge. Other symbols have their usual significances. The effect of double-layer charge Q_{dl} could be eliminated by subtraction of the background charge. The corresponding results are displayed in Figure 5. The charge (Q) linearly varies with the square roots of time ($t^{1/2}$) for the oxidation reaction with a slope of 0.0121 $\mu C\ s^{-1/2}$ in the absence of SDS. According to Equation (5), D is calculated to be $1.96 \times 10^{-7}\,cm^2\,s^{-1}$. Q_{ads} can be obtained by the subtraction of the intercepts of the plot Q *versus* $t^{1/2}$ in the presence and absence of Sudan I. Here, Q_{ads} is 0.0380 μC, corresponding to an adsorption amount (Γ) of $3.14 \times 10^{-12}\,mol\,cm^{-2}$ for Sudan I on the basis of the Faraday equation. In the presence of SDS, D is calculated to be

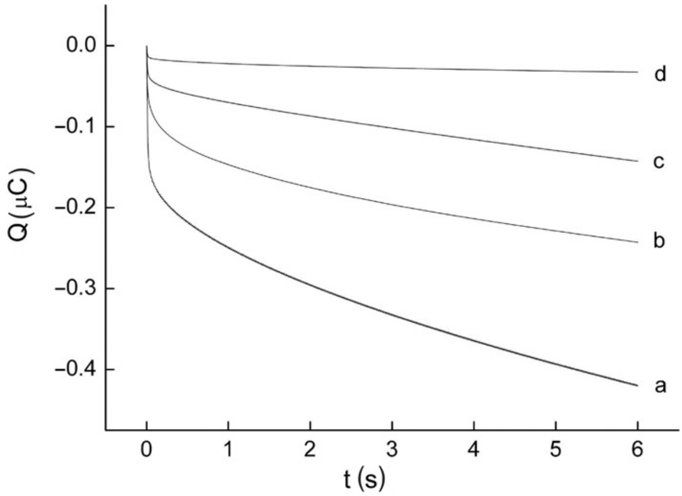

Figure 5. Chronocoulometry at PVP/CPE in 0.1 mol L^{-1} PB (pH 3.0) in the presence (a, c) and absence (b, d) of 4.0×10^{-6} mol L^{-1} Sudan I with (a, b) and without (c, d) 1.0×10^{-4} mol L^{-1} SDS. Initial potential: 0.10 V, final potential: 0.24 V, and pulse width: 6 s.

$2.71 \times 10^{-7}\,cm^2\,s^{-1}$ and Γ is estimated to be $7.64 \times 10^{-12}\,mol\,cm^{-2}$. These results also confirm the great enhancement effect of SDS for the electrochemical response of Sudan I.

3.4 *Optimal working conditions for the determination of Sudan I*

3.4.1 *Surfactant type*

As is well-known, surfactants in solution could not only endow the electrode/solution interface with different electrical properties, but also change the electrochemical process by forming adsorption layers on the electrode surface or aggregating into supramolecular structures in solution [16,17,20–22]. Surfactants could adsorb on the acetylene black paste via hydrophobic interactions with the paraffin oil, and form a hydrophilic film on the electrode surface with the polar head group directing to the bulk solution [23,24]. Figure 6 shows the electrochemical behaviours of Sudan I at PVP/in the presence of various kinds of surfactants such as anionic SDS, sodium dodecyl benzyl sulphonate (SDBS), neutral Triton X-100 and cationic cetyltrimethyl ammonium bromide (CTAB). Although all the four surfactants show an apparent enhancement effect on the oxidation of Sudan I, the highest oxidation peak current occurs in the case of SDS (curve a) instead of CTAB, a cationic surfactant that usually exhibited the best performance in many electroanalytical systems [13,14,16,17]. This may be explained with the possible interaction between positively charged azo dyes and negatively charged SDS in acidic media [25,26]. That is to say, besides the hydrophobic interactions between the hydrophobic backbone of Sudan I and the hydrophobic chain of surfactants, electrostatic interactions might also exist between Sudan I and SDS, which played an important role in the enhanced response of Sudan I at PVP/CPE. This conclusion was further supported by the higher response of Sudan I in the presence of another anionic surfactant SDBS (curve b) than that in the case of CTAB (curve c) or Triton-X 100 (curve d). The difference in structure and the

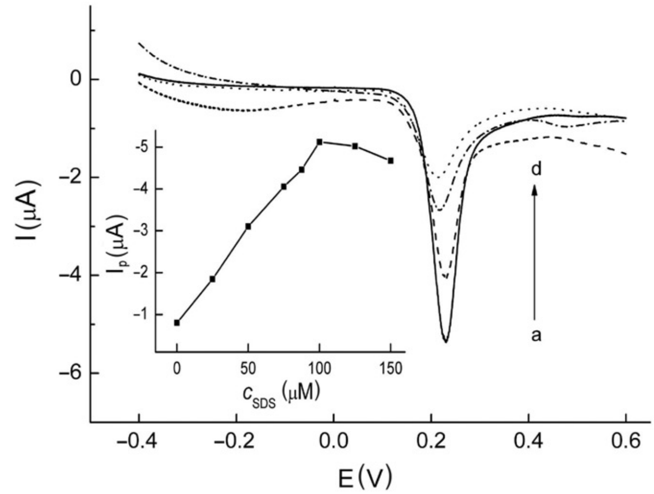

Figure 6. Influences of $1.0 \times 10^{-4}\,\mathrm{mol\,L^{-1}}$ different kinds of surfactants on the peak currents of $4.0 \times 10^{-6}\,\mathrm{mol\,L^{-1}}$ Sudan I: (a) SDS, (b) SDBS, (c) CTAB, (d) Triton X-100. Inset shows the effects of SDS concentration on the peak currents of Sudan I. Scan rate: $0.1\,\mathrm{V\,s^{-1}}$.

corresponding hydrophobic interaction with Sudan I for SDBS and SDS might explain their different enhancement effects (curve a, b). Thus, the anion surfactant SDS was employed for the determination of Sudan I.

The influence of SDS concentration on Sudan I oxidation peak current of Sudan I is also studied (inset in Figure 6). The oxidation peak current increases gradually at the beginning of the increase of the concentration of SDS and reaches the maximum at $1 \times 10^{-4}\,\mathrm{mol\,L^{-1}}$. The increase of SDS concentration in solution results in the increase of the adsorbed amount of Sudan I and the current responses of Sudan I also increase. However, the peak current decreases with the further increase of the SDS concentration, which may be caused by the micelle effect. Namely, the electron transfer between Sudan I and electrode surface might be inhibited at rather high SDS concentrations because supramolecular structures, i.e. aggregates of micelles, may form and adsorb onto the electrode surface over the critical micelle concentration (CMC) of SDS. To sum up, $1.0 \times 10^{-4}\,\mathrm{M}$ was the optimal concentration for SDS to obtain the strongest enhancement effect.

3.4.2 *Supporting electrolyte*

The type of supporting electrolytes and solution pH played key roles in the voltammetric responses of Sudan I. Different supporting electrolytes were used to estimate the current responses of $4.0 \times 10^{-6}\,\mathrm{mol\,L^{-1}}$ Sudan I. No obvious oxidation peak of Sudan I was observed in several electrolytes, including $0.1\,\mathrm{mol\,L^{-1}}$ NaOH and $0.1\,\mathrm{mol\,L^{-1}}$ acetate buffer (NaAc–HAc, pH 3.5–5.6), $0.1\,\mathrm{mol\,L^{-1}}$ NH_3OH–NH_4Cl buffer (pH 8.0–11.0) and Palitzsch buffer (pH 6.8–9.2). The results showed that the best peak shape could be obtained in $0.1\,\mathrm{mol\,L^{-1}}$ phosphate buffer and the highest oxidation peak current was observed at pH 3.0. Consequently, $0.1\,\mathrm{mol\,L^{-1}}$ PB (pH 3.0) was chosen as the supporting electrolyte for the determination of Sudan I.

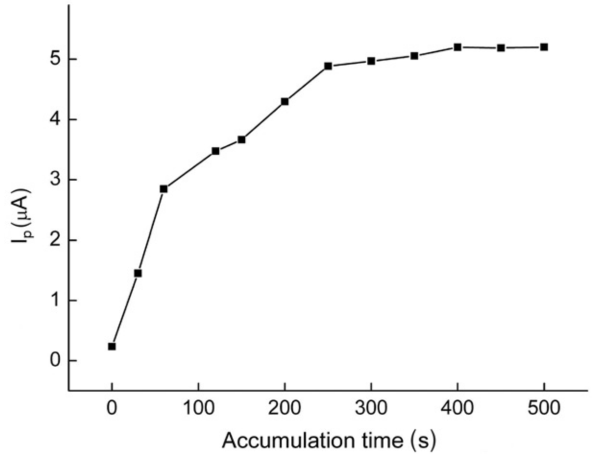

Figure 7. Influences of accumulation time on the peak current of 4.0×10^{-6} mol L^{-1} Sudan I at PVP/CPE in the presence of 1.0×10^{-4} mol L^{-1} SDS.

3.4.3 *Accumulation condition*

The effect of accumulation condition on the current responses of Sudan I was also investigated. In the range of –0.40–0.60 V, the accumulation potential showed a negligible influence on the electrochemical oxidation of Sudan I. Thus, the measurement was performed at an open circuit. The accumulation time significantly influences the current response of 4.0×10^{-6} mol L^{-1} Sudan I (Figure 7). The peak current of Sudan I rapidly increases with the accumulation time within 400 s and then increases slowly for a longer accumulation time, due to the saturated adsorption of Sudan I on the modified electrode surface. Therefore, an accumulation time of 400 s was chosen for the quantitative detection of Sudan I.

3.5 *Analytical performances*

3.5.1 *Calibration curve and reproducibility*

The calibration curve of Sudan I in the presence of SDS at the PVP/CPE under the optimal working conditions was investigated (Figure 8). The oxidation peak current was linearly related to the concentration of Sudan I in the range of 2.0×10^{-7}–8.0×10^{-6} mol L^{-1} (inset of Figure 8) with a low detection limit of 1.0×10^{-8} mol L^{-1}. The linear regression equation could be described as follows: I_p (μA) = 1.208 c (μmol L^{-1}) + 0.3164 ($R = 0.9986$). The relative standard deviation (RSD) for six parallel detections of 1.0×10^{-6} mol L^{-1} Sudan I was calculated as 4.8%, suggesting the excellent reproducibility.

3.5.2 *Interferences*

Under the optimal experimental conditions, the interferences from some metal ions and organic compounds were evaluated. The experimental results showed that 1000-fold of Zn^{2+}, Na^+, K^+, Al^{3+}, NH_4^+, Fe^{3+}, glucose, oxalic acid, citric acid, amylum, 100-fold of cholesterin and 6-fold of hypoxanthine had no apparent effects on the current responses

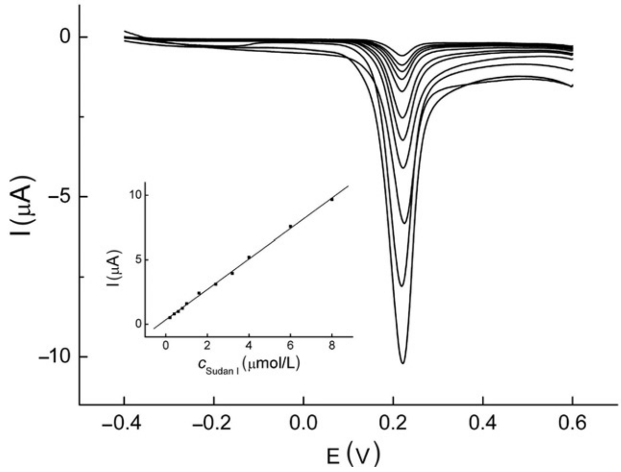

Figure 8. Linear sweep voltammograms in the presence of 0.2, 0.4, 0.6, 0.8, 1.0, 1.6, 2.4, 3.2, 4.0, 6.0 and 8.0 (from top to bottom) μmol L^{-1} Sudan I. Inset: plot peak height *vs*. Sudan concentration.

Table 1. Determination of Sudan I in capsicum samples.

Sample	Sudan I added (mol L^{-1})	Sudan I expected (mol L^{-1})	Sudan I found (mol L^{-1})	Recovery (%)
A	0.00	–	0.00	103.4
	4.027×10^{-7}	4.027×10^{-7}	4.164×10^{-7}	
B	0.00	–	0.00	98.37
	8.054×10^{-6}	8.054×10^{-6}	7.923×10^{-6}	
C	0.00	–	0.00	98.85
	1.006×10^{-5}	1.006×10^{-5}	9.945×10^{-5}	

of 4.0×10^{-6} mol L^{-1} Sudan I (signal change $<5\%$). At the same concentration, the analogues of Sudan I, e.g. Sudan II, Sudan III and Sudan IV apparently interfered with the determination of Sudan I (signal change ranged from 5% to 11%). Hundred fold of other foreign species potentially contained in chilli powder, such as capsanthin, capsorubin, zeaxanthin, β-carotene, had no influence on the determination of Sudan I. Such results indicated that the proposed analytical method had excellent anti-jamming ability.

3.5.3 *Real sample determination*

The enhanced oxidation of Sudan I at PVP/CPE in the presence of 1.0×10^{-4} mol L^{-1} SDS was applied for the determination of Sudan I in foodstuff samples. The capsicum samples were finely ground, and an amount of about 5 g was exactly weighed. Extraction was performed with 30 mL anhydrous acetone for 10 min. The extract was concentrated to 10 mL by evaporation and then centrifuged at 8000 rpm for 10 min. Ten microlitre of the supernatant was added to 5 mL 0.1 mol L^{-1} PB (pH 3.0) under stirring. The concentration of Sudan I was examined using a standard addition method. The RSD of each sample for three parallel detections was 4.5%. The recovered ratio varied between 98.37% and 103.4% (as shown in Table 1). These experimental results indicated that the proposed method for Sudan I determination was effective and highly sensitive.

4. Conclusion

A highly sensitive electrochemical method for the determination of Sudan I at PVP/CPE in the presence of SDS was developed. Sudan I underwent a sensitive oxidation process at PVP/CPE and a sharp-shaped peak was observed in the presence of SDS. The enhanced oxidation of Sudan I was attributed to the synergistic effect of PVP and SDS on the electrode surface. The electrochemical behaviour of Sudan I in the presence of different surfactants showed that both the length of the hydrophobic chain and the charge of the surfactants could influence the current responses. The proposed method was applied to the detection of Sudan I in chilli products and the results suggested that this method might have promising applications for the determination of Sudan I in real samples.

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