

Analytical Methods

Electrochemical determination of Sudan I using
montmorillonite calcium modified carbon paste electrode

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Abstract

In the current work, a sensitive, rapid and convenient electrochemical method was developed for the determination of Sudan I utilizing the excellent properties of montmorillonite calcium (MMT-Ca). Compared with the unmodified carbon paste electrode (CPE), MMT-Ca modified CPE not only significantly enhances the oxidation peak current of Sudan I but also lowers the oxidation overpotential, suggesting that the MMT-Ca modified CPE can remarkably improve the determining sensitivity of Sudan I. The experimental conditions such as determining medium, the content of MMT-Ca and accumulation time were optimized for the determination of Sudan I. The oxidation peak current is proportional to the concentration of Sudan I over the range from 0.05 mg L⁻¹ (2.01×10^{-7} mol L⁻¹) to 1.0 mg L⁻¹ (4.03×10^{-6} mol L⁻¹), and the limit of detection is 0.02 mg L⁻¹ (8.06×10^{-8} mol L⁻¹) for 2-min accumulation. Finally this newly-proposed sensing method was successfully employed to detect Sudan I in practical samples and good recovery was achieved.

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1. Introduction

Food safety analysis is a very important branch of analytical chemistry, and attracts increasing attention since it plays crucial role in human public health. Sudan dyes are phenyl-azoic derivatives extensively used in chemical industries, household commodities, textile, leather and wood industries for coloring materials such as hydrocarbon solvents, oils, fats, plastics, printing inks, shoe and floor polishes (Nohynek, Fautz, Benech-Kieffer, & Toutain, 2004). It is generally known that Sudan dyes mainly include four types: Sudan I, Sudan II, Sudan III and Sudan IV (see Fig. 1), which have been proved through laboratory experiments to cause cancer to both animals and human beings (Stiborova, Martinek, Rydlova, Hodek, & Frei, 2002). Therefore, Sudan I has been classified as a category 3 carcinogen by the International Agency for Research on Can-

cer (IARC). On this point, developing a sensitive, rapid and convenient method for the determination of Sudan dyes is of great importance and interest.

To date, the most commonly used method for the determination of Sudan dyes is high performance liquid chromatography (HPLC) (Cornet, Yasmine, Goedele, Loco, & Degroodt, 2006; Tateo & Bononi, 2004) or HPLC-MS (Calbani et al., 2004; Zhang, Zhang, & Suna, 2006) due to their high sensitivity and excellent selectivity. Otherwise, very limited electrochemical methods have also been proposed for the determination of Sudan dyes. For example, an electrochemically activated glassy carbon electrode (AGCE) was utilized to determine Sudan I based on its reduction (Du, Han, Zhou, & Wu, 2007). The linearity range is from 2.4×10^{-6} to 1.8×10^{-5} mol L⁻¹, and the detection limit is 7.1×10^{-7} mol L⁻¹. However, to the best of our knowledge, electrochemical determination of Sudan dyes based on its oxidation and using montmorillonite calcium-modified carbon paste electrode has been not reported.

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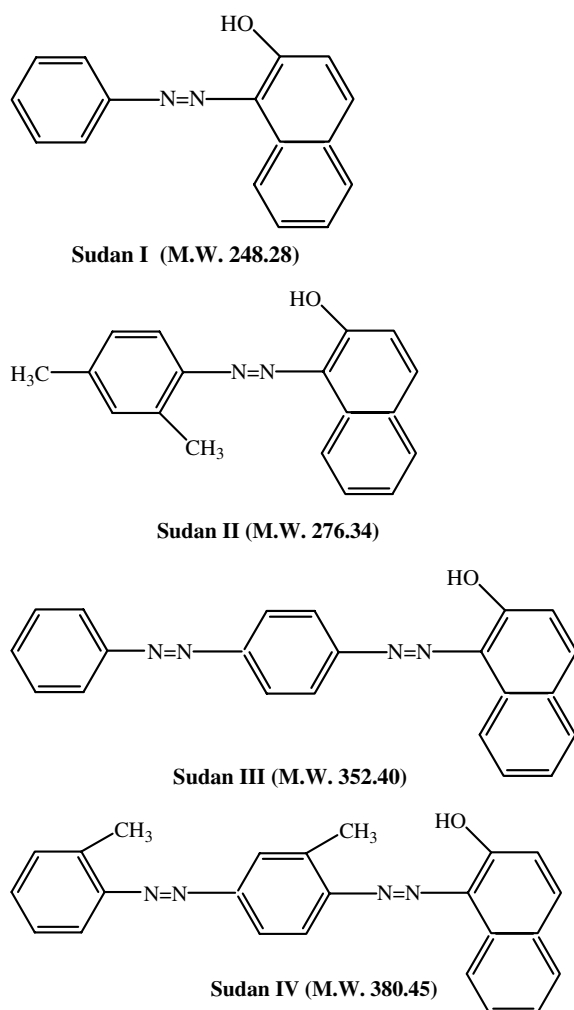


Fig. 1. Chemical structures of Sudan I, Sudan II, Sudan III and Sudan IV.

The main objective of the current work is to develop a sensitive and convenient electrochemical method for the determination of Sudan dyes utilizing the excellent properties of montmorillonite calcium (MMT-Ca). Montmorillonite belongs to the smectite group of clays with a layer lattice and includes two types: montmorillonite sodium (MMT-Na) and montmorillonite calcium (MMT-Ca). Because montmorillonite has high chemical and mechanical stability, a well-layered structure, strong adsorptive properties attributed to the expandability of the internal layers, it has been widely used in electroanalytical chemistry for different purposes (Shumyantseva et al., 2004; Xiang, Sato, Umemura, & Yamagishi, 2005; Zen & Kumar, 2004).

To complete the goal, MMT-Ca was homogeneously mixed with graphite powder and paraffin oil, and then a MMT-Ca modified carbon paste electrode (CPE) was achieved. In pH 7.0 phosphate buffer, the oxidation peak current of Sudan I significantly increases and the oxidation peak potential shifts negatively at the MMT-Ca modified CPE, suggesting that MMT-Ca facilitates the electron

transfer of Sudan I and then remarkably improves the determining sensitivity. After all the experimental parameters were optimized, a novel electrochemical method was proposed for the determination of Sudan I, demonstrating with chilli and ketchup samples. This new sensing and determining system possesses following advantages: high sensitivity, extreme simplicity, good accuracy and low cost.

2. Experimental section

2.1. Reagents

Montmorillonite calcium (MMT-Ca) was obtained from source clay Minerals Repository, University of Missouri (Columbia Mo). Graphite powder (spectral reagent) and paraffin oil were purchased from Sinopharm Group Chemical Reagent Co., Ltd., China (<http://www.sinoreagent.com>).

Sudan I (1-[(2,4-dimethylphenyl)azo]-2-naphthalenol), Sudan II (1-(phenylazo)-2-naphthalenol), Sudan III (1-(4-phenylazophenylazo)-2-naphthalenol), Sudan IV (*o*-tolylazo-*o*-tolylazo-beta-naphthalenol), were purchased from Beijing Chemical Reagent Company, China. 0.10 mg mL⁻¹ Stock solutions of Sudan I, Sudan II, Sudan III and Sudan IV were prepared in ethanol, and stored at 4 °C in the dark.

Other chemicals used were of analytical reagents, and all the chemicals were used without further purification.

2.2. Apparatus

All the electrochemical measurements were carried out using a VersaStat™ II Potentiostat/Galvanostat (Princeton Applied Research, USA), which was controlled by a PC using the Powersuit Software.

A conventional three-electrode system, consisting of a carbon paste working electrode, a saturated calomel reference electrode (SCE) and a platinum wire auxiliary electrode, was employed. The body of working electrode was a polytetrafluoroethylene (PTFE) cylinder that was tightly packed with carbon paste, while a copper wire inserts into the carbon paste to provide electrical contact.

2.3. Fabrication of MMT-Ca modified CPE

At first, 30.0 mg of MMT-Ca and 150.0 mg graphite powder were mixed uniformly by milling in a small ceramic mortar, then 30.0 µL paraffin oil was added into and milled again to give a homogenous MMT-Ca modified carbon paste. After that, the MMT-Ca modified carbon paste was pressed into the end cavity (3-mm in diameter, 1-mm in depth) of the electrode body, and the surface was smoothed against weighing paper. It is necessary to note that the amount of paraffin oil must be carefully controlled because excessive paraffin oil will lower the conductivity, while insufficient paraffin oil is not beneficial to obtain uniform MMT-Ca modified carbon paste.

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