



Short communication

Determination of tranexamic acid in cosmetic products by high-performance liquid chromatography coupled with barrel plating nickel electrode

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ABSTRACT

Tranexamic acid (TA) is an important reagent in cosmetic skin-whitening formulation and a drug for the inhibition of plasminogen to plasmin in blood. Since there is no chromophore in tranexamic acid molecule to enable direct analysis by UV–visible absorption spectrophotometry, derivatization is thus required by excluding use of UV or fluorescence detection. We report here a relatively simple electrochemical TA detection method by using a barrel plating nickel electrode. Chromatographic separation was performed on a Hamilton PRP-X100 anion-exchange column (150 mm × 4.1 mm i.d., 10 μm particle size) with a (85:15, v/v) mixture of 0.1 mol l⁻¹ NaOH and acetonitrile as mobile phase and pumped at a flow rate of 0.9 ml min⁻¹. By detecting at +0.55 V vs. Ag/AgCl, the calibration plot was linear in the concentration window of 3–1000 ppm with regression coefficient and detection limit (S/N = 3) of 0.9993 and 0.13 ppm (0.84 μmol l⁻¹), respectively. Successive injections (n = 10) of 50 ppm tranexamic acid showed a R.S.D. value of only 0.3% indicating good reproducibility of the proposed system. The method was successfully applied to the analysis of the content of tranexamic acid in cosmetic products and proved to be suitable for rapid and reliable quality control.

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1. Introduction

Tranexamic acid (TA), *trans*-4-(aminomethyl) cyclohexanecarboxylic acid, is a synthetic derivative of lysine and marketed as Cyklokapron in USA and as Transamin in Asia. It is effective on the skin in healing of wounds and can prevent skin from roughening [1,2]. Examples for TA in cosmetics products are skin-whitening lotions, creams, milky lotions, facial packs and scalp care cosmetics. It is also an antifibrinolytic drug that competitively inhibits the activation of plasminogen to plasmin, a molecule responsible for the degradation of fibrin [3–6]. Sensitive detection and determination of TA is thus of interest not only in cosmetics but also in clinical and pharmaceutical samples. Since there is no chromophore in TA molecule to enable direct analysis by UV–visible absorption spectrophotometry, derivatization is thus required by excluding use of UV or fluorescence detection [7–13]. A more reliable HPLC–tandem MS-based TA assay [14], on the other hand, always requires expensive instrumentation and skilled technician.

We report here a simple, inexpensive and sensitive electrochemical method for TA detection using a barrel plating nickel electrode

(Ni-BPE). The primary function of barrel plating is to provide an economical means to electroplate manufactured parts that meet specific finishing requirements [15,16]. Since the fabrication cost of the Ni-BPE is low, it can be disposable and as such manual polishing is not necessary. Our previous studies have successfully used this kind of disposable-type electrode for analytical applications [17–19]. In this study, we further demonstrate that the Ni-BPE coupled with a specifically designed electrochemical cell allows us to obtain an electrocatalytic profile towards the oxidation of TA. The user-friendly design of electrochemical cell is suitable for use in flow injection analysis (FIA) and is especially attractive in practical applications. Both the mechanism of electrocatalytic oxidation in FIA and the use as a detector in HPLC for the determination of TA at the Ni-BPE are discussed and investigated. It was finally applied to the determination of TA in cosmetics without the necessity of laborious sample preparation.

2. Experimental

TA (TCI), sodium hydroxide (Showa) and acetonitrile (ACN, ECHO) were used as received. All reagents were of analytical-reagent grade and prepared with de-ionized water. Cosmetic samples were purchased from a local supermarket and prepared by dissolving 1 g of the sample in mobile phase with suitable

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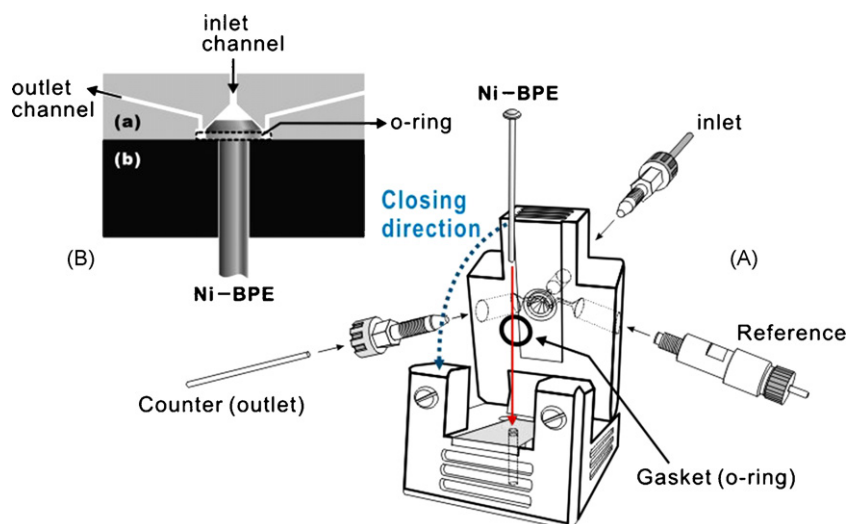


Fig. 1. Schematic representation of the Ni-BPE working system.

dilution. The two cosmetic products were labeled to contain 2% (w/w) and 1% (w/w) of TA, respectively. The excipients present are distilled water, POE-20 behenyl ether, glyceryl monostearate, methyl glucose sesquistearate and PEG-20 glyceryl stearate, PEG-100 stearate/glyceryl stearate, cetyl alcohol, myristal myristate, squalane, shea butter, cetearyl octanoate, isohexadecane, lanolin, dimethicone, tocopherol acetate, propylene glycol, carbomer, 1,3-butylene glycol, disodium edetate, sodium pyrrolidone carboxylate and methyl paraben. The real samples were ultrasonically treated for 15 min; then centrifuged at 25 °C and 4500 rpm for 5 min. After filtered sequentially through 0.45 μm Critical syringe filters (Nashua, NH, USA), the final filtrates were analyzed by HPLC.

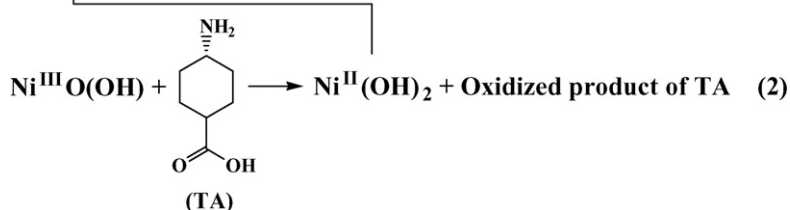
Cyclic voltammetric (CV) and chronoamperometric experiments were carried out using a CHI 900 electrochemical workstation (Austin, TX, USA). The Ni-BPE three-electrode system is the same as reported in our previous study and is activated by cycling from +0.6 to −0.2 V in 0.1 mol l^{−1} NaOH [18]. The Ni-BPE (1.25 mm diameter and 31 mm length) with an average weight of 392.2 ± 0.5 mg (*n* = 10) was a special order from Zensor R&D

solution at +0.55 V vs. Ag/AgCl until the current became constant. It usually takes 5 min at room temperature. The TA oxidation peak signal was uniformly taken as a quantitative parameter.

3. Results and discussion

3.1. Electrochemical studies

Fig. 2 compares CV responses at a conventional glassy carbon electrode (GCE) and the activated Ni-BPE in the absence/presence of 60 ppm TA in 0.1 mol l^{−1} NaOH at a scan rate (*v*) of 1 mV s^{−1}. As can be seen, no faradic responses are observed at GCE in presence/absence of TA. In contrast, the bare Ni-BPE shows a profound redox response centered at +0.45 V vs. Ag/AgCl and there is an increase in the anodic peak current together with a decrease in the cathodic peak current upon 60 ppm TA addition. The redox potential is similar to those of Ni-based electrodes reported earlier [20–23]. The electrocatalytic oxidation mechanism of TA at the Ni-BPE surface represents a mediated catalytic oxidation:



(Taichung, Taiwan). Working geometric surface area of the Ni-BPE is ~0.012 cm². Fig. 1 illustrates the schematic representation of the Ni-BPE working system used in this work. It consists of a Ni-BPE working electrode, a stainless tube counter electrode (outlet) and an Ag/AgCl reference electrode. The HPLC system consisted of a BAS-PM92E high-pressure microprocessor pump drive, a Rheodyne 7125 sample injection valve (20 μl loop) and the proposed electrochemical detector. The HPLC system with an anion-exchange column (Hamilton PRP-X100, 150 mm × 4.1 mm i.d., 10 μm particle size) was equilibrated in (15:85, v/v) ACN + 0.1 mol l^{−1} NaOH carrier

As per the above scheme, the higher oxidation state (Ni^{III}O(OH)) appearing at +0.45 V vs. Ag/AgCl is responsible for the mediation. The electrochemical observation clearly indicates the electrocatalytic behavior of TA at the Ni-BPE. Nevertheless, the electrooxidation of TA on the oxide metals can be very complex and may involve strong adsorption steps of reactants and/or reaction products [24–27]. In this study, the interaction between NiOOH and the terminal −NH₂ functional groups of TA was believed to be essential for the detection. Most importantly, when applying a potential around the oxidation peak (i.e., +0.45 V) to the Ni-BPE, the increase in the oxidation current can be used for the amperometric determination of TA.

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