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# Tranexamic acid derived gold nanoparticles modified glassy carbon electrode as sensitive sensor for determination of nalbuphine

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# ABSTRACT

We display a simple chemical procedure for fabrication of spherical gold nanoparticles (AuNps) using tranexamic acid (Tr) as reducing as well as capping agent. As-formed Tr-AuNps were characterized by Ultra Violet Visible (UV-Vis) spectroscopy where the surface plasmon absorption band was controlled at 522 nm under already optimized conditions. Atomic force microscopy (AFM) unveiled information in relation to size and shape of Tr-AuNps. Fourier transform infra red (FTIR) spectroscopy divulged the interaction between capping agent and AuNps while X-ray diffractometry (XRD) disclosed the nature of crystalline patterns of AuNps. As-prepared Tr-AuNps were sandwiched between the surface of glassy carbon electrode (GCE) and nafion and used as sensor for highly selective and sensitive voltammetric determination of nalbuphine (NA) using square wave voltammetry (SWV) as analytical mode. Parameters such as volume of nafion, working electrodes, type and ionic strength of supporting electrolyte, pH, stirring rate, initial potential, accumulation potential and accumulation time were optimized. The mechanism regarding the oxidation of NA was also proposed. The sensor responded linearly in the range of 0.05–1.25  $\mu$ g mL<sup>-1</sup> NA with excellent limit of detection (LOD) of 13.2 ng mL<sup>-1</sup> and  $R^2$  value of 0.997. In addition, the sensor performed linearly to NA even within the matrices of serum as well as urine samples. The fabricated sensor demonstrated higher selectivity for sensitive determination of NA in the presence of various interfering species commonly found in human serum and urine. The developed sensor was successfully applied and validated for the determination of NA in human serum and urine samples with excellent recoveries.

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# 1. Introduction

Nalbuphine (NA) with chemical name, 17-(cyclobutylmethyl)-4,  $5\alpha$ -epoxymorphinan-3,  $6\alpha$ , 14-triol hydrochloride (Scheme 1) belongs to opioid class of analgesic drugs. Structurally it has resemblance with oxymorphone and naloxone [1]. It possesses agonist as well as antagonist properties at kappa and mu receptors respectively. Owing to mixed agonist/antagonist profile, it wields fewer side effects in comparison to morphine. It induces analgesia in conditions pertaining moderate to severe pain such as during labor [2], caesarian section [3], postoperative pain management

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http://dx.doi.org/10.1016/j.snb.2015.01.096 0925-4005/© 2015 Elsevier B.V. All rights reserved. [4], patient-controlled analgesia [5] and works as an analgesic element of balanced anesthesia [6]. It is the only opioid analgesic that is not controlled under the FDA regulations and albeit its clinical efficacy; the drug is susceptible to potential abuse [7]. Sufficient data with reference to the misuse of drug as doping agent by sports people have been published [8,9]. Furthermore, latest report reveals that NA transmits low appearance, pulse, grimace, activity and respiration (APGAR) in neonates in case it is administered to women prior to delivery [10]. Hence the determination of NA in human plasma is the subject of serious concern.

Various methods exploited for determining NA include, High Performance Liquid Chromatography (HPLC) [1], Liquid Chromatography–Mass Spectroscopy (LC–MS) [7], Gas Chromatography–Mass Spectroscopy (GC–MS) [11] and Liquid Chromatography–Electrochemical Detection (LC–ED) [12].



Scheme 1. Structure of nalbuphine.



Scheme 2. Structure of tranexamic acid.

However all these techniques are associated with problems like higher time consumption for sample preparation, use of harmful and costly chemicals and pricey instrumentation. Only one potentiometric method has so far been reported for the determination of NA which is not only complicated due to difficulty in construction of electrode but less sensitive as well [13].

Voltammetric analysis inherits numerous advantages in comparison to above mentioned methods on account of its instrumental simplicity, ease of sample preparation, lower cost and higher sensitivity [14]. Among voltammetric methods, SWV is considered as the most sensitive and rapid technique for trace analysis of pharmaceuticals. Moreover the exploitation of nanomaterials in the field of electroanalysis has revolutionized the sensing capability of traditional electrodes. Owing to small size (1-100 nm) metal nanoparticles exhibit unique physical, chemical and electronic properties and can be integrated into sensors with remarkable sensitivity [15]. Among metal nanoparticles, AuNps have received tremendous attention owing to their higher surface to volume ratio, exceptional surface energy, stability and higher catalytic efficiency. These qualities justify AuNps as extremely suitable candidates for sensor development via electrode modification for voltammetric scrutiny of drugs [16–18]. Several citations describe the fabrication of AuNps by employing drugs and amino acids as reducing and/or capping agents [19-23].

Literature review however reveals that no attempt has so far been furnished until this work to report the determination of NA by voltammetry. This compelled us to develop a sensitive and selective NA sensor based on a green chemical drug tranexamic acid (derivative of amino acid lysine) capped AuNps GCE as sensing platform. So this is a novel and simpler approach using tranexamic acid as reducing and capping agent for the facile synthesis of AuNps and their exploitation to develop highly sensitive voltammetric NA sensor as the first report. The structure of tranexamic acid is illustrated in Scheme 2.

#### 2. Materials and methods

#### 2.1. Instrumentation

Aqueous solutions of Tr-AuNps were characterized by UV–Vis spectrophotometer, lambda 35 of Perkin Elmer. FTIR spectra of respective materials were obtained by means of Nicolet 5700

FTIR spectrophotometer of Thermo. AFM imaging of AuNps was performed with the help of Atomic force microscope of Agilent 5500, USA. X-Ray Diffractometer Model D-8 of Bruker was used for recording XRD patterns of AuNps. All voltammetric measurements were recorded with the help of model 797 trace analyzer from Metrohm.

## 2.2. Materials

Chemicals and reagents used in this work were of analytical grade (Merck quality). Solutions of tranexamic acid, chloroauric acid (HAuCl<sub>4</sub>), nalbuphine HCl, codeine, tramadol and pentazocine were prepared in deionized water. Other solutions used in interference study were also prepared in deionized water. Nafion (1%) solution was prepared by dissolving its accurately weighed amount in 2-propanol and diluted to 0.2% with deionized water. Acetate buffer (0.1 M) solution of pH 6 was prepared by mixing appropriate volumes of acetic acid and sodium acetate solutions. Phosphate buffer solution (0.1 M) of pH 6 was prepared by mixing Na<sub>2</sub>HPO<sub>4</sub> and NaH<sub>2</sub>PO<sub>4</sub> in desired ratio. Britton-Robinson buffer (BRB) solution was prepared by mixing 0.04 M solution of each of glacial acetic acid, orthophosphoric acid, and boric acid. The pH of BRB solution was adjusted to the desired value by addition of appropriate volume of 0.1 M NaOH solution.

## 2.3. Synthesis of Tr-AuNps

Tr-AuNps were synthesized by adding 200  $\mu$ L of 0.2 M NaOH solution in 5 mL of deionized water followed by addition of 120  $\mu$ L of 0.5% tranexamic acid solution and 150  $\mu$ L of 0.5% HAuCl<sub>4</sub> solution. The pH of resulting mixture was adjusted to 10.0  $\pm$  0.1 by adding 0.1 M HCl and made up to 10 mL with deionized water. The resultant solution mixture was heated at 150 °C on hot plate (preheated for 5 min) for 10 min with stirring at 200 rpm until the appearance of wine red color which confirmed the formation of Tr-AuNps.

### 2.4. Sample preparation for FTIR and XRD

A large volume (500 mL) of Tr-AuNps solution was prepared following similar procedure as mentioned earlier. The solution was transferred into large petri dish and evaporated to dryness on a water bath already regulated at 100 °C. The resultant Tr-AuNps were simultaneously washed with water and methanol twice and finally subjected to N<sub>2</sub> atmosphere for drying purpose. The Tr-AuNps thus formed were scratched with a clean glass slide, collected and further dried in an oven at 105 °C. As dried Tr-AuNps were cooled to room temperature, incorporated in KBR pellet and characterized by FTIR spectroscopy. For further characterization, a thick film was deposited via simultaneous deposition and drying of Tr-AuNps solution on circularly cut glass which possessed a diameter equal to that of XRD sampler. As loaded circular glass with thick film of Tr-AuNps was fitted in the depression of sampler and forwarded for XRD analysis.

#### 2.5. Sample preparation for AFM

According to procedure, 250  $\mu$ L of Tr-AuNps solution was drop casted on clean cleaved mica surface, evaporated under N<sub>2</sub> to form thin film and imaged via AFM technique under contact mode. In addition the surface of GCE was modified with Tr-AuNps in similar fashion as true in case of mica surface and subjected to AFM imaging. Download English Version:

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