



# Electrochemical analysis of amlodipine in some pharmaceutical formulations and biological fluid using disposable pencil graphite electrode



Nimisha Jadon <sup>\*</sup>, Rajeev Jain, Annu Pandey

School of Studies in Environmental Chemistry, Jiwaji University, Gwalior 474011, India

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## ABSTRACT

A reproducible and sensitive voltammetric procedure has been developed for the determination of amlodipine (AML) by using pencil graphite electrode (PGE) as working electrode. The experimental parameters, such as concentration, pH, amplitude, frequency, deposition potential were optimized and the peak potential was found to be 700 mV [vs. Ag/AgCl], by square-wave voltammetry (SWV) and differential pulse voltammetry (DPV). Under optimized conditions in Britton–Robinson buffer (pH 8.5), linear calibration curve was obtained in the range of 0.8 nM–51.2 nM solution. The limit of detection (LOD) and limit of quantification (LOQ) for SWV and DPV were calculated 0.02 pM, 0.04 pM, 0.06 pM and 0.2 pM respectively. The proposed method was used to estimate the amount of drug in different brands of pharmaceutical formulations with almost 99.9% recovery. Developed method was also applied for the detection of AML in spiked human serum with good recovery of almost 99% and limit of detection was found to be 0.21 pM. The proposed methodology is first attempt to develop a method on PGE which represents an effective, cheap, easily available and alternative tool instead of commonly used glassy carbon and other chemically modified electrodes for the electrochemical determination of amlodipine.

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## 1. Introduction

Amlodipine (Fig. 1) offers vital economic benefits in people suffering from heart diseases. It acts as calcium channel blocker. This drug dilates the blood vessels, thereby enhancing blood circulation. It is prescribed for patients with diabetes, if they get heart related ailments that require management using calcium channel blocker drugs. This is also using in combination with other drug for treating hypertension, angina, lower cholesterol. Its therapeutic importance and extensive use and benefits for saving the heart patient's life, need a requirement to develop a rapid and simple analytical technique for AML detection in pharmaceutical preparations and in biological fluids [1–5]. On reviewing the literature we found that several conventional techniques GC, LC, HPTLC and some spectrophotometric methods have been developed for the assay of AML in tablets and biological samples. Limitation of spectrophotometric over electrochemical method is that the differential degrades the signal-to-noise ratio. The stray light of UV–Vis spectrophotometer that caused by the faulty equipment design and other factors could influence spectra measurement accuracy of the absorption in substance. However, these methods are also dependent on many parameters, such time of the analysis, character of a compound or mixture of

compounds (extracts) and show a low sensitivity at low sample concentration over electrochemical methods. This method does not discriminate between the sample of interest and contaminants that absorb at the same wavelength [6–7]. These methods have advantage of high degree of selectivity but need sample clean-up, expensive instrumentation and a highly skilled person. A highly-sensitive, convenient and effective tool for the analysis of important biomolecules including drugs in pharmaceutical formulations and human body fluids are the electrochemical techniques, these techniques have attracted more attention nowadays due to their distinctive nature and owing to their simplicity, low cost and less time consuming as compared to the other reported analytical techniques. Some electrochemical methods have been reported for the determination of AML on different types of electrode such as carbon paste, glassy carbon, diamond electrode and gold electrode. Carbon-based materials such as glassy carbon or carbon nano-tubes or mercury electrode have also been utilized for voltammetric analysis of AML [8–16]. Demerit of unmodified bare electrodes is sluggish electron transfer and fouling of surface which reduces its sensitivity and selectivity. Different types of electrode modifiers have been applied to overcome these demerits such as a multi-wall carbon nanotubes modified glassy carbon electrode (GCE) [17–23], copper nanoparticles modified carbon paste electrode [14], fullerene-C60-modified GCE [13], polyvinylpyrrolidone modified carbon paste electrode [15,39], ruthenium hexachloroplatinate or hexacyanocobaltate film coated GCE [12] and

<sup>\*</sup> Corresponding author.

E-mail address: [nimisha09@yahoo.com](mailto:nimisha09@yahoo.com) (N. Jadon).

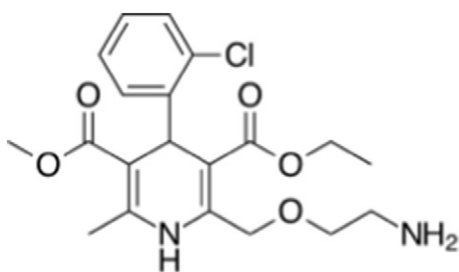


Fig. 1. Structure of amlodipine.

MWCNT/iron-doped polypyrrole modified GCE [11] with high sensitivity and selectivity for the quantification amlodipine content in pharmaceutical formulation. In this study, PGE was used for sensitive voltammetric determination of AML without applying any modifier. Earlier it was reported that pencil lead electrodes offer a renewal surface which is simpler and faster than polishing procedures, common with solid electrodes, and result in good reproducibility for individual surface [18]. Pencil graphite electrode (PGE) is a new type of carbon electrode which has been used for the determination of a few varieties of analytes by voltammetric techniques [16,40]. Electro-analysis of AML on edge plane pyrolytic graphite electrode has been reported by Goyal et al. but the detection limit is in micromolar range [40]. PGE has several advantages compared to other carbon-based electrode such as low cost, no need for time-consuming processes like surface polishing and disposability. The surface can be modified easily, has high electrochemical reactivity and surface area. PGE can be also used for stripping voltammetric analysis instead of mercury-based electrodes [24–31]. PGE nowadays could be a better replacement of other costly carbon based electrodes for the detection of organic molecules. This paper is the first systematic report of AML quantification on PGE up to pM level.

## 2. Materials and methods

### 2.1. Reagents and solutions

All reagents used like boric acid, acetic acid, orthophosphoric acid, sodium hydroxide, and methanol were of analytical grade quality (Merck and Sigma) and were used without further purification. Amlodipine (as besylate, assay: 98.63%) standard was obtained from Cipla Limited, Chandigarh, India. Different pharmaceutical tablets containing amlodipine (Asomex-2.5, Amlovas-5, Amlodac-5, Amlogard) by different manufacturers were purchased from local market. Ultrapurified water supplied by a Milli-Q system (Millipore) with resistivity greater than 18.0  $\Omega$  cm was used for the preparation of solutions. Pencil lead, insulin syringe and bontite for making electrode were also purchased from local market.

Stock solution of 1.0 mM concentration was daily prepared in methanol and working solutions were prepared by further dilution of this stock solution. 1.0 M KCl solution was used as supporting electrolyte for all experiments.

### 2.2. Instrumentation

Electrochemical experiments were carried out by Autolab type III (potentiostat-galvanostat with 757VA Computrace software). Pencil graphite electrode (PGE) as working electrode, Ag/AgCl as reference electrode, and a graphite rod as auxiliary electrode were employed. All the measurements were performed under nitrogen purging for 5 min at room temperature. pH measurement of buffers was carried out on a pH meter fitted with a glass electrode.

### 2.3. Preparation of pencil graphite electrode (PGE)

The pencil graphite electrode was handmade. It was prepared by using insulin syringe as a holder and bontite for fixing the lead into syringe. HB pencil lead of diameter of 2.0 mm, was used. Prepared PGE was cleaned by rubbing it on butter paper and was pretreated before measurement.

### 2.4. Analytical procedure

Differential pulse voltammetry (DPV), square-wave voltammetry (SWV) and cyclic voltammetry (CV) methods were applied for the investigation of electrochemical behaviour and quantification of drug solution. Voltammograms were obtained by addition of different volumes of prepared working drug solution into the electrochemical cell containing 8.0 mL of BR buffer of desired pH (pH 8.5), 1.0 mL methanol and 1.0 mL 1 M KCl as supporting electrolyte.

### 2.5. Procedure for assay in pharmaceutical formulation

Commercial samples of pharmaceutical formulations with declared AML content were purchased from a local pharmacy. Each tablet contains a dose of 5.0 mg and 2.5 mg AML according to the information. The stock solutions of samples of the tablets were prepared as follows: five tablets were accurately weighed and powdered in mortar. A portion of powdered tablet equivalent to the average weight of one tablet was dissolved into 5.0 mL methanol followed by sonication for 15 min in a 10.0 mL volumetric flask then solution was made up to the mark using methanol to get final concentration. Aliquots of this solution were diluted to final volume of 10.0 mL was completed with buffer and supporting electrolyte and then transferred into a voltammetric cell and voltammograms were recorded.

### 2.6. Procedure for assay in spiked human serum

1.0 mL of human serum was transferred into a centrifugation tube. Aliquot of AML stock solution was added to get the final concentration. It was mixed well using a vortex mixer and then 0.5 mL methanol was added as precipitating agent after that it was centrifuged then contents of the centrifugation tube was transferred quantitatively into the voltammetric cell and voltammograms were recorded for AML. Values of the peak current ( $i_p$ ) vs. the corresponding concentration were plotted to obtain the calibration graph and LOD, LOQ were also calculated.

## 3. Results and discussion

### 3.1. Characterization of PGE

EIS is a beneficial technique to get information about properties of surface conductivity of electrodes. Conductivity of PGE was taken using the 3.0 mM potassium ferricyanide prepared in phosphate buffer of pH 7.0 (Fig. 2). Electron charge transfer resistance ( $R_{ct}$ ) was calculated for by using the Randles-Sevcik equation. The  $R_{ct}$  value was found for bare PGE as 476.56  $\Omega$ , which shows better electrical conductivity and has a faster electron transfer rate on bare PGE. Surface area was also calculated by cyclic voltammograms of 1.0 mM potassium ferricyanide at different scan rate and was found to be 0.165 cm<sup>2</sup> using equation given below. SEM images of PGE surface shows regular graphite layer with little rough surface.

$$i_p = 0.4463 \left( F^3 / RT \right)^{1/2} A n^{3/2} D_R^{1/2} C_0 v^{1/2}$$

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