



Nicotinamide adenine dinucleotide assisted direct electrodeposition of gold nanodendrites and its electrochemical applications



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ABSTRACT

The shape tailoring in gold (Au) nanostructures is vital for tuning their optical and catalytic properties. Herein, we describe nicotinamide adenine dinucleotide (NAD⁺) assisted direct electrochemical growth of surface-confined Au nanodendrites as well as the application of these nanodendrites in the non-enzymatic detection of glucose in neutral pH and the oxidation of methanol in alkaline pH. NAD⁺ plays an important role in the growth of Au nanodendrites. The specific adsorption of NAD⁺ onto the Au (011) facet aids in the growth of Au nanodendrites. In the absence of NAD⁺, interconnected wall-like morphology is obtained. The Au nanodendrites are characterized by UV–visible spectroscopy, X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM) and electrochemical methods. FESEM and TEM analysis confirm that the Au nanostructures are dendritic, consisting of a trunk from which several branches evolve; the presence of NAD⁺ in the supporting electrolyte solution plays a vital role in the evolution of this morphology. The electrode based on Au dendrites has an electrochemically accessible surface area of 0.281 cm² and shows excellent electrocatalytic activity for both glucose and methanol. In alkaline pH, the Au nanodendrite-based electrode oxidized methanol at 0.3 V with a highly-stable response. This electrode oxidizes glucose at 0.4 V in neutral pH without the use of enzymes. The sensitivity and limit of detection of the electrode are calculated to be 0.037 ± 0.02 μA mM⁻¹ cm⁻² and 7.29 μM (S/N = 3), respectively. The surface morphology of the Au nanostructure plays an important role in the electrocatalytic performance.

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1. Introduction

In recent years, synthesis of inorganic materials in nanoscale dimension has drawn much attention because of their prospective application in various fields of science and technology. In particular, much attention has been given towards the shape tailoring of Au nanostructures due to their fascinating shape-dependent optical and catalytic properties and wide range of potential applications [1]. For the synthesis and shape tailoring of Au nanostructures, traditional wet-chemical synthetic protocol has been widely used and Au nanostructures of diverse shapes such as, stars [2], rods [3], polyhedral [4], prisms [5], flowers [6], wires [7], cubes [8], multipods [9], octahedrons [10], branched

[11], nanoplates [12] and dendrites [13–15] etc. have been obtained. To evaluate the practical application, in many cases, it is essential to immobilize these nanostructures on various solid supports, which is very difficult task. He, Goyal and Oyama's groups have developed various methods for the immobilization of Au nanostructures on different conducting supports [16–22]. However, direct electrodeposition method is convenient one, since shape tailoring of Au nanostructure and integration on the solid supports can be achieved simultaneously in a single step without using any sophisticated equipment, noxious reducing, stabilizing and immobilizing agents. As for example, a wide variety of Au nanostructures such as flower [23,24], nanocube [25], nanorod [26], branched [27], nanothorns [28], pinecones [29], porous sheets [30], quasi rods [31], urchins [32] etc. have been synthesized on different conducting supports. Among the aforementioned shapes, much emphasis has been given towards the synthesis of Au dendrites because of their potential applications in various fields

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such as in electrochemical sensing [33], electrocatalysis [34], surface-enhanced Raman scattering [35], fuel cells [36], etc. Following electrochemical approach, direct electrodeposition of Au dendrites has been achieved in the absence [34,35] and presence of different additives [36–40]. In the later case, the additives present in the electrolyte plays an important role during the electrochemical growth of Au dendrites. For instance, Wang et al. demonstrated that it is possible to synthesize catalytically active Au nanodendrites using ethylenediamine as an additive, and described how the presence of ethylenediamine in the electrolyte played a vital role in the morphology tuning of Au nanostructure [36]. In another study, Huang et al. demonstrated that Au nanodendrites can be synthesized using iodide ions as an additive [37]. Bhargava et al. demonstrated that dendritic Au nanostructures can be synthesized electrochemically by introducing $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ into the electrolyte and controlling the electrodeposition potential [38]. In another study, Lin et al. synthesized dendritic Au nanostructures using cysteine as an additive and demonstrated the role of cysteine in the electrochemical growth of catalytically active Au dendrites [39]. Recently, three-dimensional hierarchical Au dendrites were synthesized by the electrochemical method using cytosine as an additive [40]. The aforementioned synthetic protocols developed by several research groups clearly indicate the vital role of additives in the growth of Au dendrites. Though the use of several additives as shape regulating agent and their role in the direct electrodeposition of dendritic Au nanostructure is well known, yet, to the best of our knowledge there is no report available that addresses the use of NAD^+ in the direct electrodeposition of dendritic Au nanostructures. The present investigation not only demonstrates a facile method for the direct electrochemical growth of catalytically active dendritic Au nanostructures but also it opens the possibility for NAD^+ to be used as a shape regulating agent in the direct electrodeposition of other metal nanostructures.

Our group is interested in the development of various synthetic protocols for the shape-controlled synthesis of Au nanostructures and their utilization in the fabrication of electrochemical interface for electrocatalytic applications. Recently, we have demonstrated the seed-mediated growth of raspberry-like Au nanostructures using electrodeposited Au nanoseeds in a growth solution composed of HAuCl_4 and hydroxylamine and have studied their electrocatalytic activity [41]. In that synthetic approach, pre-immersion of Au nanoseeds in the NAD^+ solution played an important role in the growth of raspberry-like Au nanostructures. In continuation of our earlier efforts, herein, we describe NAD^+ assisted direct electrodeposition of catalytically active Au dendrites. Presence of NAD^+ in the electrolyte plays an important role in the growth of dendritic Au nanostructure and our proposed reagent NAD^+ can also be used in the shape tailoring of other metal nanostructures. The effect of deposition pulse width, concentration of NAD^+ and HAuCl_4 on the morphology of Au nanostructures was studied in some detail. Compared to the Au nanostructures synthesized in the absence of NAD^+ , the Au dendrites synthesized in the presence of NAD^+ have higher electrocatalytic activity towards the electrochemical oxidation of glucose and methanol.

2. Experimental section

2.1. Materials

HAuCl_4 and NAD^+ were obtained from Sigma–Aldrich Co. (St. Louis, USA) and were used without any further purification. Analytical grade chemicals methanol, KOH, H_2SO_4 , Na_2HPO_4 , and NaH_2PO_4 were used as received. Millipore water was used to prepare all solutions.

2.2. Instrumentation

The spectral profile of Au dendrites was recorded using a UVS-2100 SCINCO spectrophotometer. The X-ray diffraction (XRD) profile was recorded using a Max 2500 V/PC diffractometer (Rigaku Corporation, Tokyo, Japan). The morphology of Au dendrites was obtained using a field emission scanning electron microscope (JSM-6701F, JEOL, Tokyo, Japan). TEM images and high-resolution TEM (HR-TEM) images were recorded using a field emission transmission electron microscope (FE-TEM; JEOL JEM-2200 FS, Tokyo, Japan) at the KBSI Jeonju Center in Korea. The TEM sample was prepared by scratching the Au thin film from the GC surface and dispersing it in ethanol by ultrasonication. From this dispersion 5 μL was removed and drop-casted onto a TEM grid. Electrochemical experiments were performed using a computer-controlled CHI660D electrochemical analyzer, attached with a Faraday cage/picoampere booster (CH Instruments, Austin, USA). The measurements were carried out using a two-compartment three-electrode cell, in which glassy carbon (GC), platinum (Pt) wire and Ag/AgCl (3 M NaCl) were used as the working electrode, auxiliary electrode, and reference electrode, respectively. All electrochemical measurements were performed in argon atmosphere.

2.3. Synthesis of Au nanodendrites

Au nanodendrites were synthesized electrochemically by a square-wave technique. A solution containing 2 mM HAuCl_4 and 150 mM NAD^+ in 0.5 M H_2SO_4 was used as the electrolyte. The deposition of Au nanodendrites was achieved using lower and higher potentials of -0.8 and 0.2 V, respectively, at a frequency of 40 Hz for a period of 2000 s. The electrode was then washed with copious amount of water and used in further experiments.

3. Results and Discussion

3.1. Characterization of Au nanostructures

3.1.1. UV-visible spectra

The Au nanostructures were characterized by UV-visible spectral measurement. Fig. 1 shows the spectral profile obtained for the Au nanostructures synthesized in the absence and presence

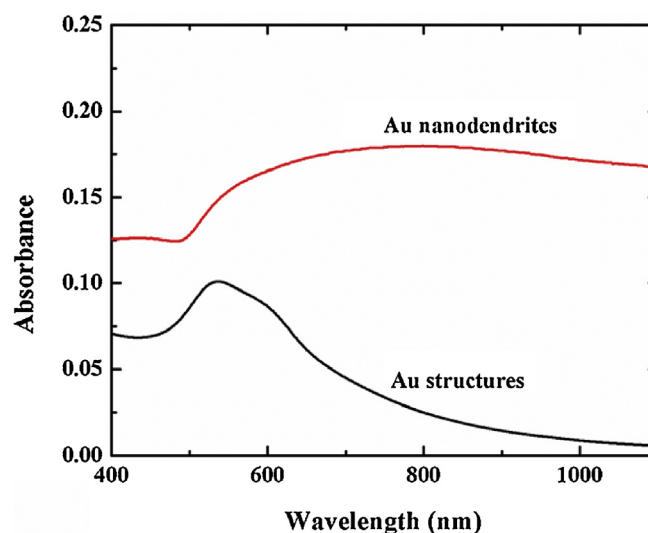


Fig. 1. UV-Vis spectra of Au nanostructures grown in the absence of NAD^+ and Au nanodendrites.

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