

## Regular Article

# Electrochemical study of acetaminophen oxidation by gold nanoparticles supported on a leaf-like zeolitic imidazolate framework

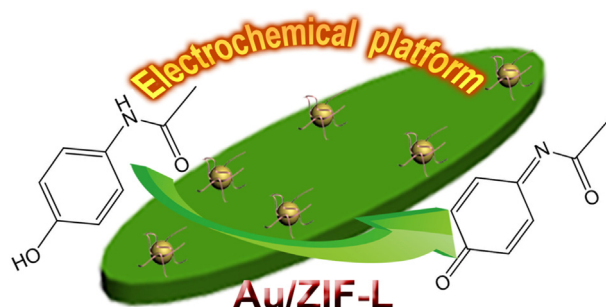


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## GRAPHICAL ABSTRACT

A novel gold nanoparticles supported on a leaf-like zeolitic imidazolate framework hybrids are used as electrocatalyst for the oxidation of acetaminophen.



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## ABSTRACT

Decoration of leaf-like zeolitic imidazolate framework (ZIF-L) with Au nanoparticles (NPs) by a simple self-assembly method was described in this work. The materials were characterized by Fourier transform infrared spectroscopy, energy-dispersive X-ray spectra,  $N_2$  adsorption, and thermogravimetric analysis. Acetaminophen (AP) is most commonly referred to as an over-the-counter antipyretic and analgesic, which is a key factor in relieving fever and pain. It is particular significant to take efforts and conduct research for the development of reliable methods to detect the AP. An electrochemical sensor for AP was constructed based on the Au/ZIF-L, which exhibited excellent electrocatalytic activity for the oxidation of AP with the catalytic rate constant ( $k_{cat}$ ) of  $4.27 \times 10^4 \text{ M}^{-1} \text{ s}^{-1}$  and diffusion coefficient ( $D$ ) of  $8.31 \times 10^{-5} \text{ cm}^2 \text{ s}^{-1}$ . Importantly, Au/ZIF-L was developed as an effective sensing platform for the detection of AP, which showed high analytical performance, such as a linear range of  $3.50 \text{ }\mu\text{M}$ – $0.056 \text{ mM}$  with sensitivity of  $37.28 \text{ }\mu\text{A mM}^{-1}$ , a linear range of  $0.056$ – $0.56 \text{ mM}$  with sensitivity of  $25.10 \text{ }\mu\text{A mM}^{-1}$ , and a low limit of detection of  $1.02 \text{ }\mu\text{M}$ . The successfully fabricated Au/ZIF-L device can be used to accurately measure the amount of AP in pharmaceutical samples. Results implied the potential application of functionalized MOF composite materials in the field of electrocatalysis, making it particularly suitable for electroanalytical chemistry.

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## 1. Introduction

Acetaminophen (AP) is most commonly referred to as an over-the-counter (OTC) antipyretic and analgesic. It is a key factor in relieving fever and pain [1]. AP plays a significant role in therapeutic field, which can inhibit the synthesis of prostaglandins in the central nervous system and also sedate the hypothalamic heat regulating centre [2,3]. However, long-term usage may cause a number of health problems like skin rashes and liver damage, even death [4]. Consequently, it is particular significant to take efforts and conduct research for the development of reliable methods to detect the AP. At present, there are various techniques having been described for the determination of AP, such as UV-spectrophotometry [5], spectrofluorimetry [6], high-performance liquid chromatography [7], gas chromatography [8], and thermogravimetric analysis [9]. Recently, the electrochemical method has been paid widely attention to analytical instrument because of its easy of monitoring, high sensitivity, small instrumentation, and low cost [10–18].

Metal-organic frameworks (MOFs) are a new number of porous materials which were constructed by assembling metal ions or clusters with organic ligands in an appropriate solvent [19,20]. Due to their high specific surface area, large internal pore volume, ultrahigh porosity, and tunable channel, MOFs have been attracting increasingly comprehensive attention in the recent years [21–26]. Presently, MOFs were attracted immense interest in the field of electrocatalytic owing to the redox behavior of metal cations inside MOFs, which can provide a pathway for electrons. For example, Qin et al. described stable polymolybdate-based MOFs as highly active electrocatalysts for hydrogen generation from water [27]. Qiao's group designed highly oxidative MOFs as electrocatalysts for urea oxidation reaction [28]. An ultrathin MOFs were reported by Zhou et al., which have been used as electrocatalysts for oxygen evolution [29]. Kornienko and co-workers synthesized a kind of MOFs and found it can be used for electrocatalytic reduction of carbon dioxide [30]. Nezamzadeh-Ejhi's group demonstrated a facile approach to construct zeolite modified electrodes for the determination of chromium and cesium [31,32]. It is well known that noble metal nanoparticles (NPs) show strong electrocatalytic performance for small molecules [33–41]. Meanwhile, noble metal NPs encapsulated inside or loaded on MOFs have been attracted much attention, due to the improved electrocatalytic and conductivity [25,42,43]. Bagheri's group has prepared Au NPs immobilized on Cu-based MOFs to fabricate hydrazine and L-cysteine electrochemical sensors [44,45]. Arul et al. demonstrated a facile approach to synthesize the Ag NPs loaded on MOFs for the determination of H<sub>2</sub>O<sub>2</sub> [46]. Yadav and co-workers reported the design of Au/MOF-5 electrochemical sensor for the nitrite and nitrobenzene [47]. Han et al. developed a facile way to synthesize the Au NPs supported on Ti-based MOFs for electrocatalytic oxidation of hydrazine [48]. Ju's group designed Pt NPs encapsulated MOFs for the electrochemical detection of telomerase activity [49].

In the present study, we have demonstrated a facile strategy for the load of Au NPs on leaf-like zeolitic imidazolate framework (Au/ZIF-L). Moreover, the unique Au/ZIF-L exhibited remarkable electrochemical activity for AP, indicating that the Au/ZIF-L composite may hold great promise for the design of electrochemical sensors in pharmaceutical products.

## 2. Experimental

### 2.1. Chemical reagents

HAuCl<sub>4</sub>·3H<sub>2</sub>O, sodium citrate dehydrate, polyvinylpyrrolidone (PVP), zinc nitrate hexahydrate (Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O), 2-

methylimidazole (C<sub>4</sub>H<sub>6</sub>N<sub>2</sub>), and other reagents were of analytical grade from Shanghai Macklin Biochemical Co. Ltd. All solutions were freshly prepared using double distilled water. The supporting electrolyte was prepared for 0.1 M phosphate buffer solution (PBS pH = 7.4), which was made up from NaH<sub>2</sub>PO<sub>4</sub>, Na<sub>2</sub>HPO<sub>4</sub>, and H<sub>3</sub>PO<sub>4</sub>.

### 2.2. Instrumentation

All the electrochemical measurements were performed using an AUTOLAB (Metrohm Instruments, Switzerland) electrochemical workstation and a CHI760E electrochemical Analyzer (CH Instruments, Shanghai Chenhua Instrument Corporation, China) at laboratory temperature. A typical three electrode was used, including the glassy carbon electrode (GCE) as the working electrode; a platinum electrode as counter electrode; and an Ag/AgCl (insaturated KCl solution) electrode as a reference electrode. Scanning electron microscopy (SEM) image was carried out with a PhilipsXL-30 ESEM. Energy-dispersive X-ray (EDX) spectra were characterized with JEOL (JEM-2100F). X-Ray diffraction pattern (XRD, Rigaku X-ray D/max-2200vpc, Japan) was recorded with Cu K $\alpha$  radiation operating at 40 kV/20 mA ( $k = 0.15406$  nm). Fourier transform infrared (FTIR) for the various samples were examined using Nicolet Magna 560 FT-IR spectrometer by mixing the materials with KBr. Thermogravimetric analysis (TGA) image was taken by a PerkinElmer Diamond TG analyzer.

### 2.3. Preparation of the modified electrodes

Prior to use, GCE ( $d = 3$  mm) was polished carefully before each experiment with 0.05  $\mu\text{m}$  alumina power. After that, the electrode was sonicated successively in HNO<sub>3</sub> (1:1) and absolute alcohol. Then the electrode was rinsed thoroughly with double-distilled water and dried in high-purity nitrogen steam. A drop (5  $\mu\text{L}$ ) of the suspension (2  $\text{g}\cdot\text{L}^{-1}$ ) was dip-coated onto the GCE surface and dried at 25  $^{\circ}\text{C}$ .

### 2.4. Synthesis of PVP-Au NPs

PVP-Au NPs were prepared following a modified literature procedure by reduction of HAuCl<sub>4</sub> with sodium citrate [50]. Typically, HAuCl<sub>4</sub> aqueous solution (0.2 mL, 0.1  $\text{g}\cdot\text{mL}^{-1}$ ) and H<sub>2</sub>O (200 mL) was added into a 500 mL flask, and was heated at 110  $^{\circ}\text{C}$  for 15 min under vigorously stirring. Then, sodium citrate aqueous solution (3 mL, 1 wt%) was induced to the solution quickly and keep stirring at 110  $^{\circ}\text{C}$  for another 30 min. PVP solution (0.4 mL, 0.0255  $\text{g}\cdot\text{mL}^{-1}$ ) was added to 20 mL citrate-stabilized Au NPs solution, and the mixture was stirred for 24 h at 25  $^{\circ}\text{C}$ .

### 2.5. Synthesis of Au/ZIF-L

ZIF-L was prepared according to the reported method with a minor modification [51]. Briefly, 0.2 mL PVP-Au NPs, PVP aqueous solution (1 mL, 0.01  $\text{g}\cdot\text{mL}^{-1}$ ), Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (40 mL, 0.0148  $\text{g}\cdot\text{mL}^{-1}$ ) and 2-methylimidazole aqueous solution (40 mL, 0.0328  $\text{g}\cdot\text{mL}^{-1}$ ) were mixed. Subsequently, the mixture was allowed to react at ice-bath for 4 h under vigorous stirring. The product was obtained by centrifugation, washed several times with methanol and vacuum-dried overnight.

### 2.6. Pharmaceutical real samples solution preparation

The tablets of AP were finely grinded. Calculated amounts of the AP powder were dissolved in absolute alcohol. The solution was filtered carefully and then stored in the refrigerator at 4  $^{\circ}\text{C}$  and used for analysis.

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