DEVELOPMENT OF TIO_2 NANOTUBE AND SNO_2 NANOFIBER SUPPORTED GOLD NANOPARTICLE BASED NON-ENZYMATIC H_2O_2 SENSORS AND ITS PRACTICAL APPLICATIONS

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We hereby declare that We have checked this thesis, and, in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang Al-Sultan Abdullah or any other institutions.

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ABSTRAK

Sensor elektrokimia, alat teknologi mutakhir pada masa itu, menyediakan kemampuan pemantauan masa nyata, memungkinkan analisis senyawa tertentu yang nyaman dan cepat di berbagai domain. Namun, kekurangan intrinsik seperti kekuatan sinyal yang rendah, denaturasi, stabilitas yang rendah, dan biaya perawatan yang tinggi menghambat penggunaan luas dari sensor H_2O_2 berbasis protein redoks dan enzim konvensional.Untuk mengatasi batasan ini, dikembangkanlah sensor elektrokimia tanpa enzim berbasis katalis nanopartikel emas (Au NPs) yang didukung oleh oksida logam berpori, yaitu nanotube dioksida titanium (TiO_2 NTs) dan nanofiber tin(IV) oksida (SnO_2 NFs), untuk mendeteksi H_2O_2. Penelitian ini dimulai dengan sintesis Au NPs ukuran 4-5 nm, TiO_2 NTs anatase, dan SnO_2 NFs multipori melalui reduksi sitrat, anodisasi elektrokimia, dan metode elektrospinning, masing-masing. Elektrod komposit pertama, Au NPs/TiO_2 NTs disiapkan dengan mendistribusikan Au NPs dalam pori-pori TiO_2 NTs anatase melalui pengendapan titik, yang dikonfirmasi melalui karakterisasi permukaan menggunakan Field Emission Scanning Electron Microscope (FESEM) dan X-ray Powder Diffraction (XRD). Elektrod komposit lainnya, GCE/Au NPs/SnO_2 NFs, dibuat dengan mendeposisikan Au NPs bersama dengan dukungan SnO_2 NFs pada elektrod karbon kaca (GCE). Karakterisasi menggunakan Transmission Electron Microscope (TEM) dan X-ray Diffraction (XRD) menunjukkan keberadaan baik Au NPs dan SnO_2 NFs dalam komposit. Baik TiO_2 NTs berpori dan SnO_2 NFs memiliki sifat unik yang menjebak Au NPs dalam struktur berpori mereka, mencegah agregasi dan mempercepat perpindahan elektron, yang mengarah ke respons arus yang jauh lebih tinggi selama pendeteksian H_2O_2. Penelitian elektrokimia terhadap Au NPs/TiO_2 NTs dan GCE/Au NPs/SnO_2 mengungkapkan bahwa Au NPs pada elektrod menunjukkan puncak-puncak yang berbeda dan merupakan satu-satunya material yang menunjukkan respons katalitik dengan bantuan dukungan TiO_2 NTs dan SnO_2 NFs. Semasa kronoampometri pelbagai langkah pada potensi -0.35 V, elektrod komposit Au NPs/TiO_2 NTs menunjukkan tindak balas pantas dalam 1.55 s ke arah H_2O_2 di mana lineariti, sensitiviti, dan had pengesanan 1 µM hingga 5.413 mM, 519.38 µA/mM, dan 104.4 nm, masing-masing. Di sisi lain, GCE/Au NPs/SnO_2 menunjukkan respons linear yang lebih cepat terhadap penambahan H_2O_2 dari 49.98 µM hingga 3.937 mM dengan sensitivitas dan LOD berturut-turut adalah 14.157 µA/mM dan 6.67 µM. Di samping itu, kedua-dua sensor menunjukkan imuniti yang kuat ke arah mengganggu bahan, ketepatan prestasi yang baik dan kestabilan tindak balas jangka panjang. Pada bahagian terakhir, sensor komposit Au NPs / TiO_2 NTs diperiksa dengan air paip, susu dan bakteria dan sensor GCE / Au NPs / SnO_2 diuji dengan air paip, jus epal dan bakteria di mana kedua-duanya menunjukkan pemulihan yang baik H_2O_2 dengan sisihan piawai relatif yang boleh diterima. Secara keseluruhan, sensor berasaskan komposit Au NPs/TiO_2 dan Au NPs/SnO_2 sangat menjanjikan dalam teknologi penderiaan elektrokimia. Dengan batas deteksi rendah, stabilitas jangka panjang, dan pemulihan sampel nyata yang lebih tinggi, komposit ini menghembuskan kehidupan baru ke dalam kemungkinan mendeteksi H_2O_2 di berbagai lingkungan, seperti ramuan mistik yang mengatasi masalah biosensor tradisional.

ABSTRACT

Electrochemical sensors, a state-of-the-art technological tool at the time, provide real-time monitoring capabilities, allowing convenient and rapid analysis of specific compounds in various domains. However, the intrinsic shortcomings such as low signal strength, denaturation, low stability and high maintenance cost plague the widespread use of conventional redox protein and enzyme-based H_2O_2 sensors. To address these limitations, porous metal oxide, titanium dioxide nanotube (TiO_2 NTs) and Tin(IV) oxide nanofiber (SnO_2 NFs) supported gold nanoparticle (Au NPs) catalyst-based enzyme free electrochemical sensor was developed to detect H_2O_2. The work commenced with the synthesis of 4-5 nm size Au NPs, anatase TiO_2 NTs and multiporous SnO_2 NFs via citrate reduction, electrochemical anodization and electrospinning method, respectively. The first composite electrode, Au NPs/TiO_2 NTs was prepared by distributing Au NPs within the pores of anatase TiO_2 NTs via drop casting, confirmed via surface characterization using Field Emission Scanning Electron Microscope (FESEM) and X-ray Powder Diffraction (XRD). Another GCE/Au NPs/SnO_2 NFs composite electrode was fabricated by depositing Au NPs along with SnO_2 NFs support onto a glassy carbon electrode (GCE). Characterization using Transmission Electron Microscope (TEM) and X-ray Diffraction (XRD) showcasing the coexistence of both Au NPs and SnO_2 NFs within the composite. Both porous TiO_2 NTs and SnO_2 NFs have unique properties that trap Au NPs in their porous structure, preventing aggregation and accelerating electron transfer, leading to significantly higher current response during H_2O_2 sensing. The electrochemical investigation of Au NPs/TiO_2 NTs and GCE/Au NPs/SnO_2 revealed that Au NPs on the electrode exhibited distinct peaks and were the sole material showing catalytic response with the help of TiO_2 NTs and SnO_2 NFs support. During multiple step chrono-amperometry at a potential of -0.35 V, the Au NPs/TiO_2 NTs composite electrode demonstrated a speedy response within 1.55 s towards H_2O_2 where linearity, sensitivity, and detection limits of 1 µM to 5.413 mM, 519.38 µA/mM, and 104.4 nm, respectively. On the other hand, GCE/Au NPs/SnO_2 displayed quite faster linear response towards the addition of 49.98 µM to 3.937 mM of H_2O_2 where sensitivity and LOD were calculated to be 14.157 µA/mM and 6.67 µM, respectively. In addition to this, both sensors exhibited strong immunity towards interfering substances, good performance accuracy and long-term response stability. In the last part, the Au NPs/TiO_2 NTs composite sensor was examined with tap water, milk and bacteria and the GCE/Au NPs/SnO_2 sensor was tested with tap water, apple juice and bacteria where they both exhibited good recoveries of H_2O_2 with acceptable relative standard deviations. Overall, Au NPs/TiO_2 NTs and Au NPs/SnO_2 composite based sensors are very promising in electrochemical sensing technology. With their low detection limit, long-term stability and higher real sample recovery, these composites breathe new life into the possibility of detecting H_2O_2 in various environments, like a mystical potion that overcomes the problems of traditional biosensors.

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