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Fe NPs and MWCNTs based electrode as FIA detector for determination of amino acids in hypothalamus microdialysis fluids

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ABSTRACT

An amperometric electrode based on multiwall carbon nanotubes (MWCNTs) and Fe nanoparticles (NPs) has been successfully fabricated. The electrode combined with Flow Injection Analysis (FIA) exhibits linear response in the concentration range of 0.1–12 μM and the sensitivity of 30.0 $\text{nA} \cdot \mu\text{M}^{-1}$ for most of amino acids, confirming good complexation of amino acids (AAs) with Fe NPs on the modified electrode. The determination of 17 amino acids in the hypothalamus microdialysis fluids of guinea pigs, illustrates that it is a powerful tool to investigate physiology and pathology mechanisms.

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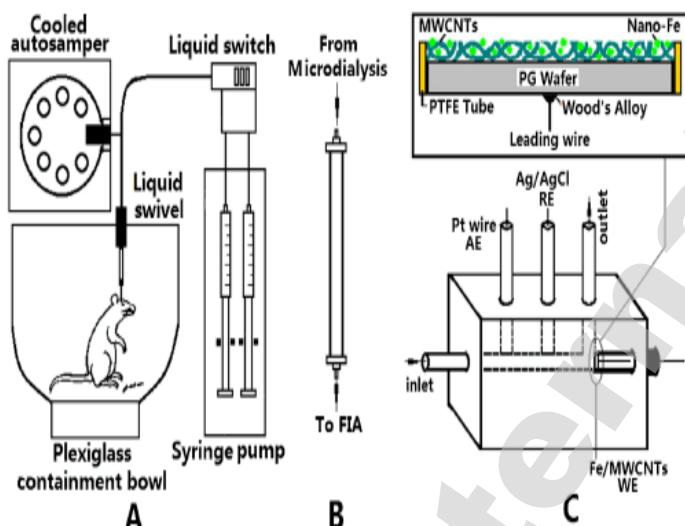


Fig. 1. Schematic diagram of the electrochemical measurement system with Fe NPs/MWCNTs electrode for determining amino acids in the hypothalamus microdialysis fluids of guinea pigs. It included A) microdialysis, B) AAs separation column, and C) Flow Injection Analysis (FIA) with Fe NPs/MWCNTs electrode as a detector (a larger view is above).

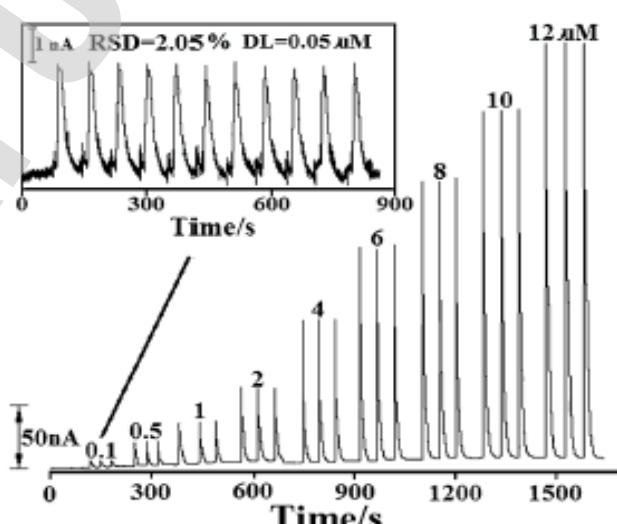


Fig. 9. When FIA system with the electrode proposed as a detector, A) responses of a series of glycine concentration (0.1–12 μM) with 250 $\mu\text{l}/\text{min}$ of flow rates in pH 8.0 of PBS carrier solution. A set of 11 replicate measurements for 0.1 μM glycine listed in inset, yields a relative standard deviation (RSD) of 2.05%, the detection limit ($S/N = 3$) is 0.05 μM .

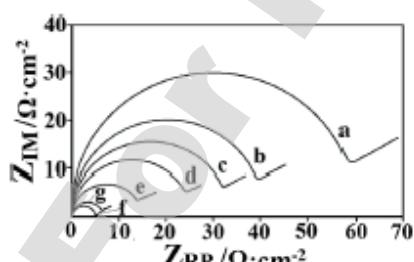
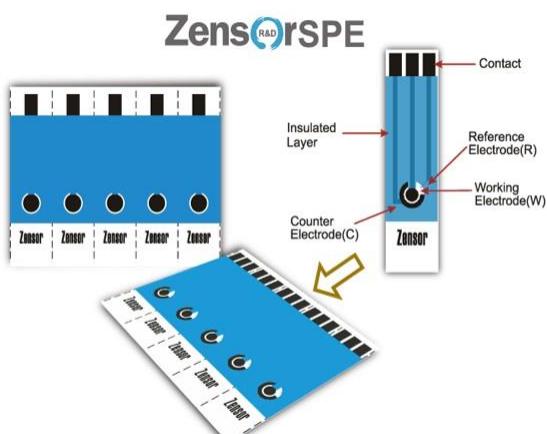


Fig. 4. EIS for Fe-MWCNTs electrode in pH 8.0 PBS containing 2 mM glycine with different ratios of $W_{\text{Fe}}/W_{\text{Fe}-\text{MWCNT}}$. From a to g, the ratios of $W_{\text{Fe}}/W_{\text{Fe}-\text{MWCNT}}$ are 0, 5, 10, 15, 20, 25 and 30%, respectively. The conductivity increased with increasing of Fe nanoparticles proportions until the ratio reached 25%. When the ratio was 30%, the conductivity is almost no change than one of 25%. Therefore, 25% of ratio was an optimization condition.





Rational Design of a Stimuli-Responsive Polymer Electrode Interface Coupled with in Vivo Microdialysis for Measurement of Sialic Acid in Live Mouse Brain in Alzheimer's Disease

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ABSTRACT: Sensitive and selective monitoring of sialic acid (SA) in cerebral nervous system is of great importance for studying the role that SA plays in the pathological process of Alzheimer's disease (AD). In this work, we first reported an electrochemical biosensor based on a novel stimuli-responsive copolymer for selective and sensitive detection of SA in mouse brain. Notably, through synergistic hydrogen-bonding interactions, the copolymer could translate the recognition of SA into their conformational transition and wettability switch, which facilitated the access and enrichment of redox labels and targets to the electrode surface, thus significantly improving the detection sensitivity with the detection limit down to 0.4 pM. Besides amplified sensing signals, the proposed method exhibited good selectivity toward SA in comparison to potential interference molecules coexisting in the complex brain system due to the combination of high affinity between phenylboronic acid (PBA) and SA and the directional hydrogen-bonding interactions in the copolymer. The electrochemical biosensor with remarkable analytical performance was successfully applied to evaluate the dynamic change of SA level in live mouse brain with AD combined with in vivo midrodialysis. The accurate concentration of SA in different brain regions of live mouse with AD has been reported for the first time, which is beneficial for progressing our understanding of the role that SA plays in physiological and pathological events in the brain.

KEYWORDS: sialic acid, stimuli-responsive polymer, electrochemical sensor, microdialysis, Alzheimer's disease

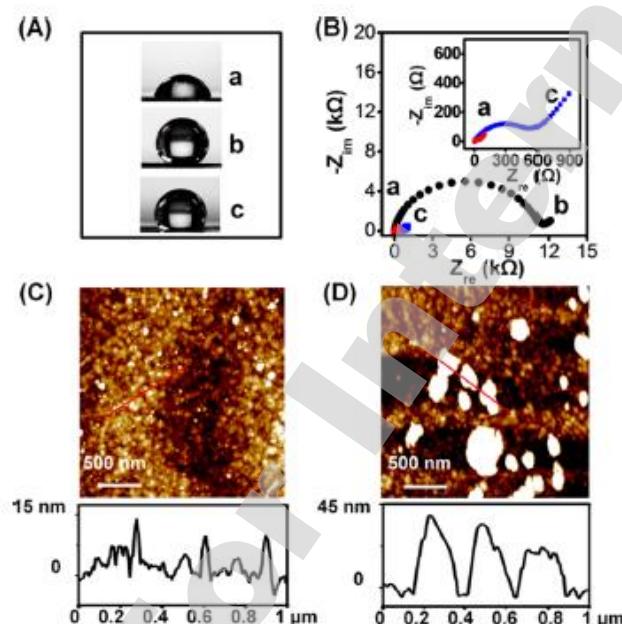


Figure 2. (A) Contact angle photographs and (B) Nyquist plots obtained at (a) SPCE/Au surface, SPCE/Au/PNI-PBA-TP surface (b) before and (c) after being exposed to SA solution (1×10^{-5} M) and typical tapping-mode AFM images for the copolymer modified Au surface (C) before and (D) after being treated by SA solution (1×10^{-3} M) together with the height profiles of the cross section analysis.

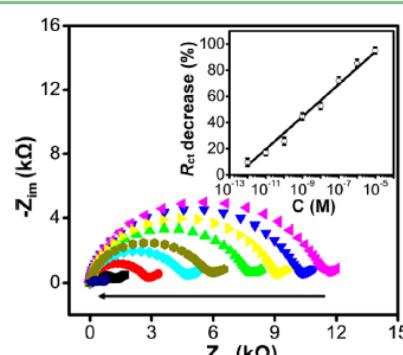
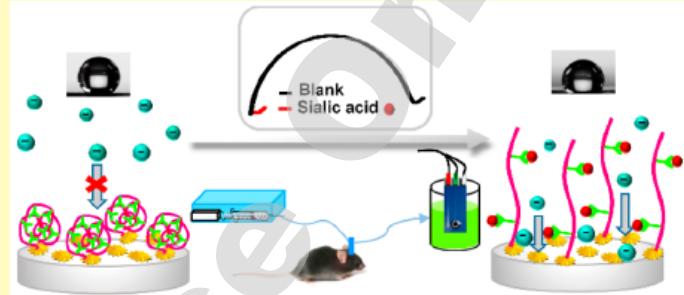
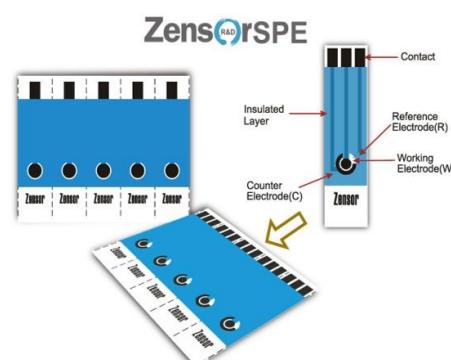


Figure 3. Nyquist plots obtained at the modified electrode surfaces in artificial cerebrospinal fluid (aCSF, pH 7.4) containing 5 mM $\text{Fe}(\text{CN})_6^{3-/4-}$ upon addition of SA solutions (1×10^{-12} – 1×10^{-5} M). Inset: relationship between the extent of R_{ct} decrease and the concentrations of SA. Error bars represent standard error measurements (SEM).





Electrocatalytic oxidation and flow injection analysis of isoniazid drug using a gold nanoparticles decorated carbon nanofibers-chitosan modified carbon screen printed electrode in neutral pH



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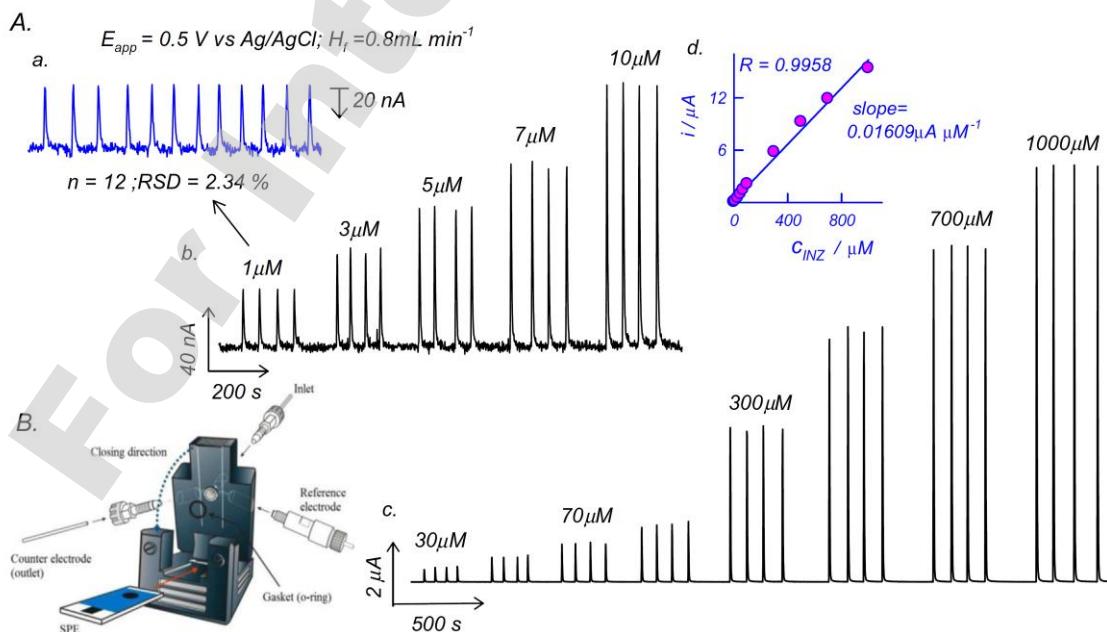
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Keywords:

Electrochemical flow injection analysis
Isoniazid drug
Neutral pH operation
Gold nanoparticles
Carbon-nanofiber-chitosan composite

ABSTRACT

Isoniazid (INZ) is an effective anti-tuberculosis drug that has been widely used for chemotherapy of tuberculosis. Development of new and simple electroanalytical method for routine analysis of INZ is a continued research interest in the field of analytical and pharmaceutical chemistry. Although there have been several electroanalytical reports, flow injection analysis coupled electrochemical detection of INZ, which is an advanced electrochemical technique useful for practical applications, is rarely reported in the literature. Instability problem associated with the underlying electrode by the amino-functional group of the INZ is the prime reason for the limitation. Herein, we report a gold nanoparticles decorated carbon nanofibers-chitosan modified carbon screen printed electrode, designated as Au_{nano}@CNF-CHIT/SPE, prepared by drop-casting of Au³⁺ ion directly on CNF-CHIT/SPE underlying electrode followed by electrochemical potential cycling, as an elegant electrochemical detector system for high stable FIA of INZ in pH 7 phosphate buffer solution. The modified electrode showed about fifty times higher electrocatalytic current and 700 mV reduction in the over-potential values than that of a polycrystalline gold modified electrode towards INZ. Under an optimal hydrodynamic FIA condition, the present ECD showed a wide linear range from 1 μM to 1 mM with regression coefficient and sensitivity values of 0.9958 and 16.1 nA μM⁻¹ respectively. The calculated limit of detection and limit of quantification values are 172 nM (i.e., 472 pg/20 μL) and 570 nM (i.e., 1.57 μg/20 μL), respectively. The present FIA-ECD was successfully applied for the real sample analysis by detecting INZ in two pharmaceutical formulations with satisfactory good recovery values.





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Highly sensitive determination of non-steroidal anti-inflammatory drug nimesulide using electrochemically reduced graphene oxide nanoribbons

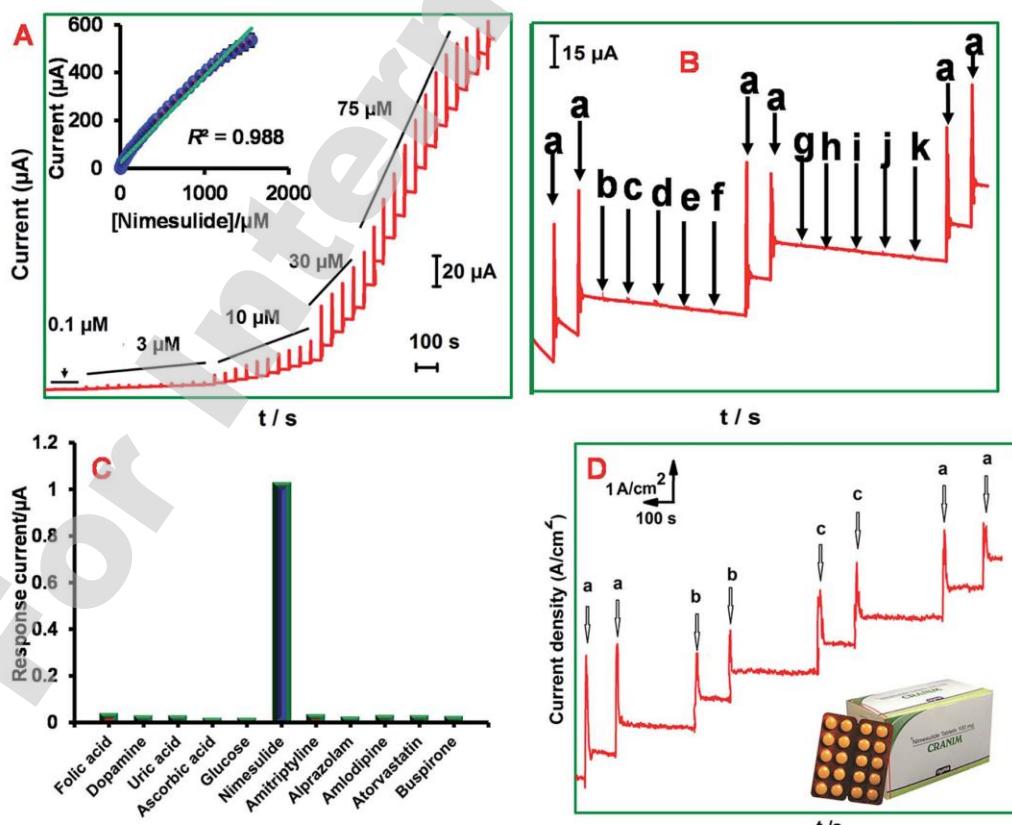
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The excess use of nimesulide (NIM) causes acute side effects to gastrointestinal, central nervous and genitourinary systems and hence its rapid, sensitive determination is highly important. We describe a robust electrochemical sensor based on electrochemically reduced graphene oxide nanoribbons (ER-GONRs) modified on a screen-printed carbon electrode (SPCE) for detecting NIM in pharmaceutical formulations and biological medium. Compared with parental multiwalled carbon nanotubes (MWCNTs), ER-GONRs possess rich edge defects, abundant functional groups, high area-normalized edge-plane structures and chemically active sites and hence they can be a superior electrocatalyst and signal amplifier for electroanalytical applications. ER-GONRs/SPCE exhibited excellent sensing performance towards NIM. The linear range was 1.0×10^{-8} to 1.50×10^{-3} M and the detection limit was 3.50 (± 1.57) nM. In addition, the ER-GONRs/SPCE showed excellent real-time sensing applications in NIM tablet and human urine samples, which could find potential applicability in drug and clinical analysis. The combined advantages of SPCE technology and ER-GONRs make this method a robust, low-cost, reproducible, sensitive and easy-to-use sensor.

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Self-monitoring of tear glucose: the development of a tear based glucose sensor as an alternative to self-monitoring of blood glucose

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Tear glucose sensing for diabetes management has long been sought as an alternative to more invasive self-monitoring of blood glucose (SMBG). However, tear glucose sensors were known to have limitations, including correlation issues with blood glucose due to low sample volume, low concentration of glucose in the tear fluid, and evaporation of the tear sample. An engineering design approach to solve these problems led to the development of an integrated device capable of collecting the tear sample from the ocular surface with little to no stress on the eye, with an extremely low limit of detection, broad dynamic range, and rapid detection and analysis of sample. Here we present the development of a prototypical self-monitoring of tear glucose (SMTG) sensor, summarizing bench studies on the enzymes and their specificity, the development of the fluid capture device and its manufacture and performance and results of system testing in an animal study where safety, lag time and tear glucose to blood glucose correlation were assessed.

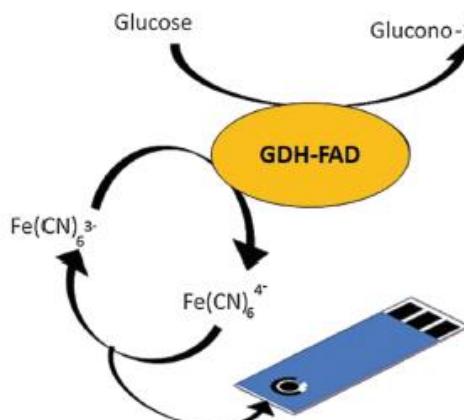


Fig. 3 Schematic diagram of the catalytic reaction between glucose and GDH-FAD using ferricyanide as an electron mediator. The electrons detected by the sensor are then correlated to glucose concentrations.

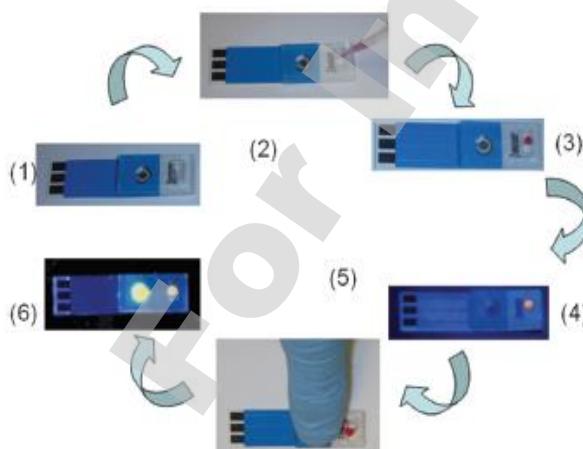


Fig. 6 Schematic detailing the operation of the microfluidic device.

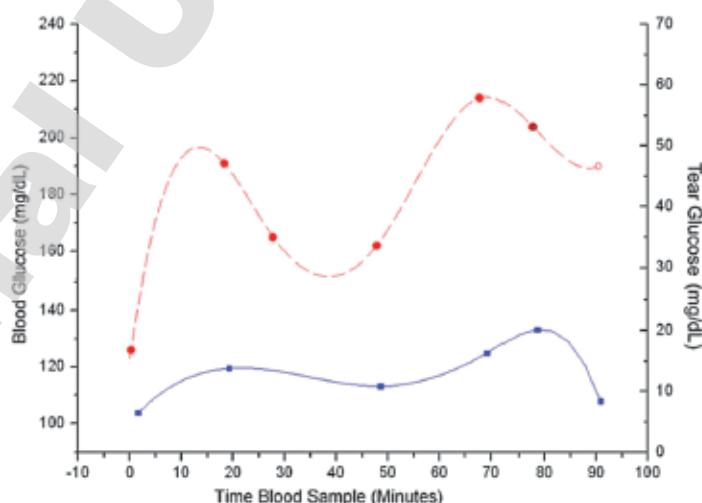
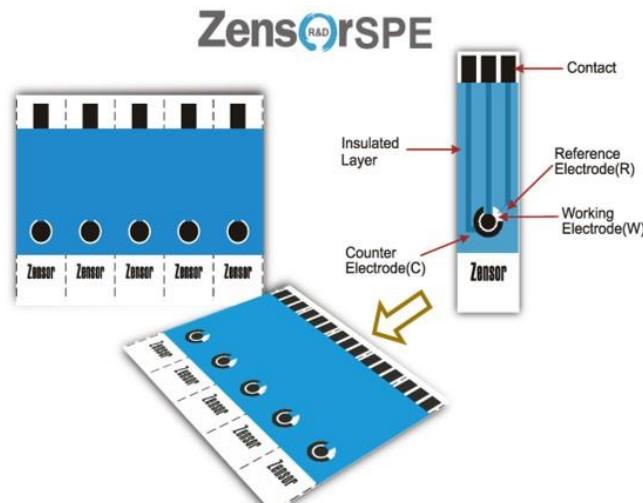


Fig. 9 Experimental representation of the lag time of blood glucose (BG) versus TG for a single animal.



Non-invasive screening for early Alzheimer's disease diagnosis by a sensitively immunomagnetic biosensor

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Amyloid-beta peptide 1–42 ($\text{A}\beta_{42}$) is considered as a reliable biomarker for the early diagnosis of Alzheimer's disease (AD). Thus, it is urgent to develop a simple and efficient method for the detection of $\text{A}\beta_{42}$. In this work, a reusable biosensor based on magnetic nitrogen-doped graphene (MNG) modified Au electrode for the detection of $\text{A}\beta_{42}$ has been developed. The antibodies of $\text{A}\beta$ 1–28 ($\text{A}\beta_{\text{ab}}$) are used as the specific biorecognition element for $\text{A}\beta_{42}$ that were conjugated on the surface of MNG. In the presence of magnetic nanoparticles on MNG, the electrode coating material, the biosensor can be quickly constructed, without requiring an electrode drying process, which reduce the analysis time and is convenient for proceeding to detection. The reusable biosensor with good reproducibility and stability was linear within the range from 5 pg mL^{-1} to 800 pg mL^{-1} , covering the cut-off level of $\text{A}\beta_{42}$ and a detection limit of 5 pg mL^{-1} had been achieved. Furthermore, the fabricated biosensor for $\text{A}\beta_{42}$ detection not only improves the detection performance but also reduces the cost and shortens the response time, demonstrating its potential in diagnosing applications.

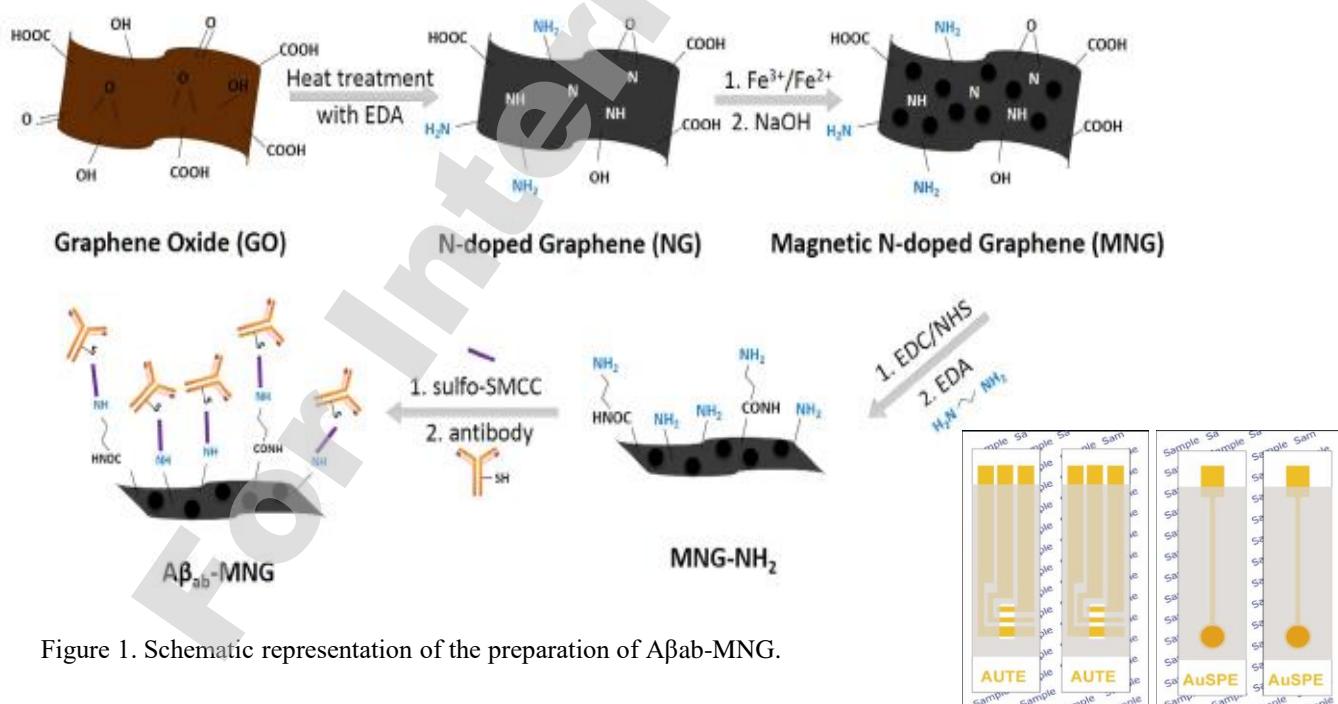


Figure 1. Schematic representation of the preparation of $\text{A}\beta_{\text{ab}}\text{-MNG}$.



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Regular Article

Electrochemical properties of the acetaminophen on the screen printed carbon electrode towards the high performance practical sensor applications



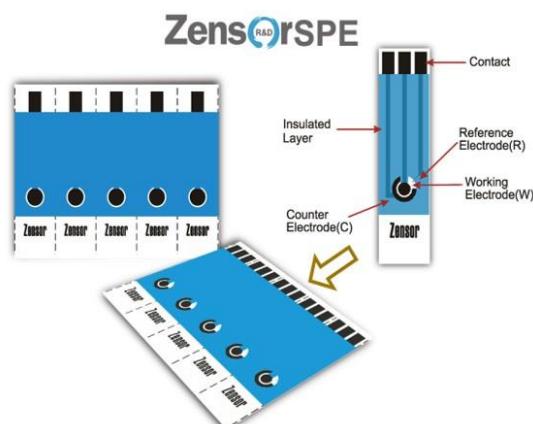
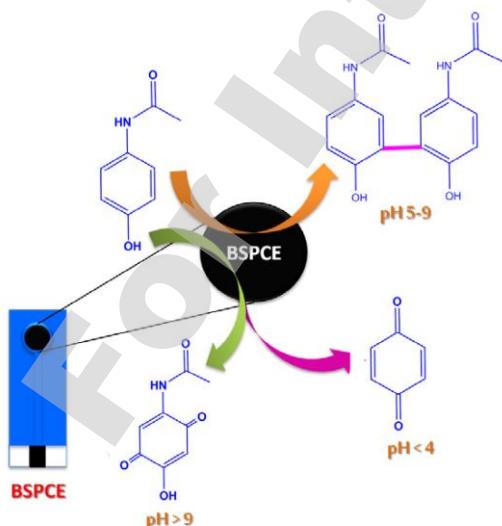
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ABSTRACT

Acetaminophen is a non-steroidal anti-inflammatory drug used as an antipyretic agent for the alternative to aspirin. Conversely, the overdoses of acetaminophen can cause hepatic toxicity and kidney damage. Hence, the determination of acetaminophen receives much more attention in biological samples and also in pharmaceutical formulations. Here, we report a rapid and sensitive detection of the acetaminophen based on the bare (unmodified) screen printed carbon electrode (BSPCE) and its electrochemistry was studied in various pHs. From the observed results, the mechanism of the electro-oxidation of acetaminophen was derived for various pHs. The acetaminophen is not stable in strong acidic and strong alkaline media, which is hydrolyzed and hydroxylated. However, it is stable in intermediate pHs due to the dimerization of acetaminophen. The kinetics of the acetaminophen oxidation was briefly studied and documented in the schemes. In addition, the surface morphology and disorders of BSPCE was probed by scanning electron microscope (SEM) and Raman spectroscopy. Moreover, the BSPCE determined the acetaminophen with the linear concentration ranging from 0.05 to 190 μ M and the lower detection limit of 0.013 μ M. Besides that it reveals the good recoveries towards the pharmaceutical samples and shows the





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Label-free electrochemical immunosensor for the rapid and sensitive detection of the oxidative stress marker superoxide dismutase 1 at the point-of-care

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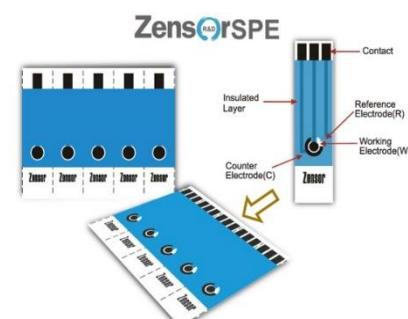
A B S T R A C T

In this work, we have fabricated a label-free electrochemical immunosensor for the detection of Cu,Zn superoxide dismutase (SOD1) which is clinically important to a wide variety of neurodegenerative, cardiovascular, and chronic immune diseases. The immunosensor is comprised of a screen printed carbon electrode (SPCE) modified with self-assembled monolayers (SAMs) of gold nanoparticles (GNPs) in electropolymerized polypyrrole (PPy) and biofunctionalized with monoclonal anti-SOD1 antibody. The morphological changes of each electrode modification step were analyzed by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) while electrochemical changes were monitored via cyclic voltammetry (CV) and impedance spectroscopy (EIS) by using ferri-ferrocyanide as electrochemical redox probe. The GNP nanostructured immunosensor indirectly monitors the SOD1 levels via electrocatalytic oxidation of nitrite, due to inherent nitrite oxidase activity of SOD1, with a wide linear sensing range (0.5 nM to 5 μ M), low detection limit (0.5 nM), and high sensitivity ($46.6 \pm 3.5 \text{ nA nM}^{-1}$). SOD1 concentration levels were also measured in real biological samples (i.e., cultured human epidermal keratinocytes) and the results correlated well with a western blot densitometry assay. Such rapid detection of SOD1 concentration levels in real biological samples is well-suited for point-of-care (POC) diagnostics.

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Scheme 1. Schematic representation of the label-free SOD1 immunosensor.



SCIENTIFIC REPORTS



Quantification of ethanol in plasma by electrochemical detection with an unmodified screen printed carbon electrode

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Simple, rapid and accurate detection of ethanol concentration in blood is very crucial in the diagnosis and management of potential acute ethanol intoxication patients. A novel electrochemical detection method was developed for the quantification of ethanol in human plasma with disposable unmodified screen-printed carbon electrode (SPCE) without sample preparation procedure. Ethanol was detected indirectly by the reaction product of ethanol dehydrogenase (ADH) and cofactor nicotinamide adenine dinucleotide (NAD^+). Method validation indicated good quantitation precisions with intra-day and inter-day relative standard deviations of $\leq 9.4\%$ and 8.0%, respectively. Ethanol concentration in plasma is linear ranging from 0.10 to 3.20 mg/mL, and the detection limit is 40.0 $\mu\text{g}/\text{mL}$ ($S/\text{N} > 3$). The method shows satisfactory correlation with the reference method of headspace gas chromatography in twenty human plasma samples (correlation coefficient 0.9311). The proposed method could be applied to diagnose acute ethanol toxicity or ethanol-related death.

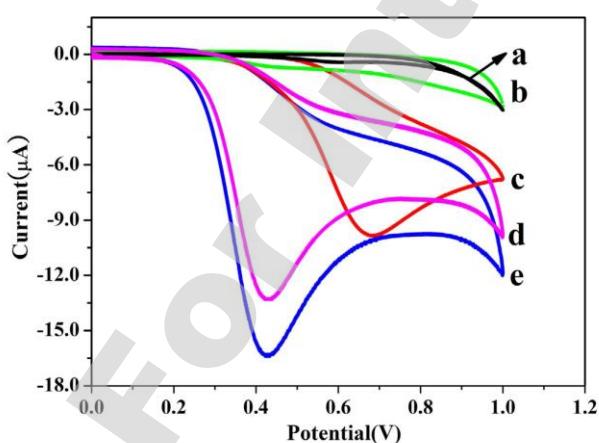
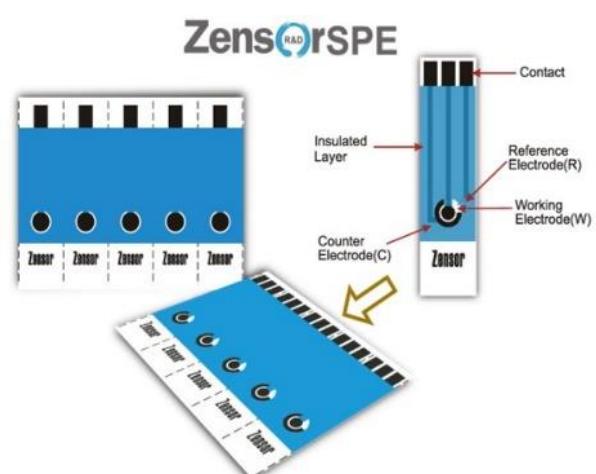


Figure 3. Cyclic voltammetry (CV) curves in 220 mM PBS (pH 10.4) (a), blank plasma in PBS (b), 4 μL NAD⁺ (100 mM), 4 μL ADH (360 U/L) and 4 μL spiked ethanol (20.0 mg/mL) plasma in 80 μL PBS (c), 4 μL NAD⁺ (100 mM), 4 μL ADH (360 U/L) and 4 μL 100-fold dilution spiked ethanol (20.0 mg/mL) plasma in 80 μL PBS (d), 0.05 mM NADH in PBS (e).





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Author manuscript

Wound Repair Regen. Author manuscript; available in PMC 2017 March 06.



Electrochemical detection of *Pseudomonas* in wound exudate samples from patients with chronic wounds

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Abstract

In clinical practice, point-of-care diagnostic testing has progressed rapidly in the last decade. For the field of wound care, there is a compelling need to develop rapid alternatives for bacterial identification in the clinical setting, where it generally takes over 24 hours to receive a positive identification. Even new molecular and biochemical identification methods require an initial incubation period of several hours to obtain a sufficient number of cells prior to performing the analysis. Here we report the use of an inexpensive, disposable electrochemical sensor to detect pyocyanin, a unique, redox-active quorum sensing molecule released by *Pseudomonas aeruginosa*, in wound fluid from patients with chronic wounds enrolled in the WE-HEAL Study. By measuring the metabolite excreted by the cells, this electrochemical detection strategy eliminates sample preparation, takes less than a minute to complete, and requires only 7.5 microliters of sample to complete the analysis. The electrochemical results were compared against 16S rRNA profiling using 454 pyrosequencing. Blind identification yielded 9 correct matches, 2 false negatives, and 3 false positives giving a sensitivity of 71% and specificity of 57% for detection of *Pseudomonas*. Ongoing enhancement and development of this approach with a view to develop a rapid point-of-care diagnostic tool is planned.

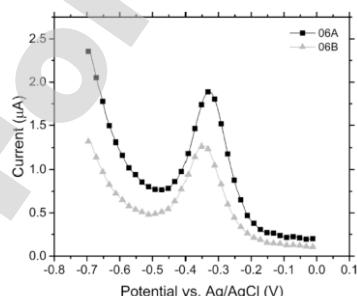


Figure 2.
Square-wave voltammograms of wound fluid exudate. Pyocyanin peak indicates the presence of *Pseudomonas aeruginosa* in the sample.

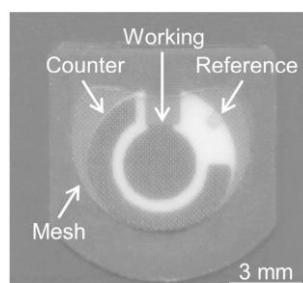


Figure 3.
Disposable, screen-printed electrode sensor with mesh modification for small-volume analysis.





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Rudimentary simple method for the decoration of graphene oxide with silver nanoparticles: Their application for the amperometric detection of glucose in the human blood samples

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A B S T R A C T

Graphene oxide decorated with silver nanoparticles (GO-Ag) was prepared by anodic dissolution of silver in the aqueous dispersion of GO. The composites were characterized by XRD, XPS, TEM, AFM, and Raman spectroscopy. The electrooxidation of glucose on GO-Ag modified electrodes have been tested by cyclic voltammetry and chronoamperometry. The detail mechanism of redox processes on the GO-Ag electrodes has been studied. A few μg loading of silver has demonstrated to give current in μA for the mM concentration of glucose. A linear relationship between peak height in the voltammograms and glucose concentration in the range 1–14 mM has been proposed for amperometric detection of glucose. From the results, the detection limit for glucose sensing is estimated to be as small as 4 μM . The selectivity for glucose in presence of interfering molecules viz. ascorbic and uric acids is tested. Proof-of concept is presented by carrying out the measurements in real human blood samples.

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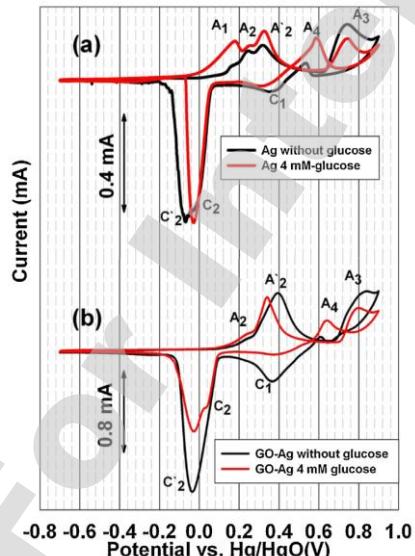
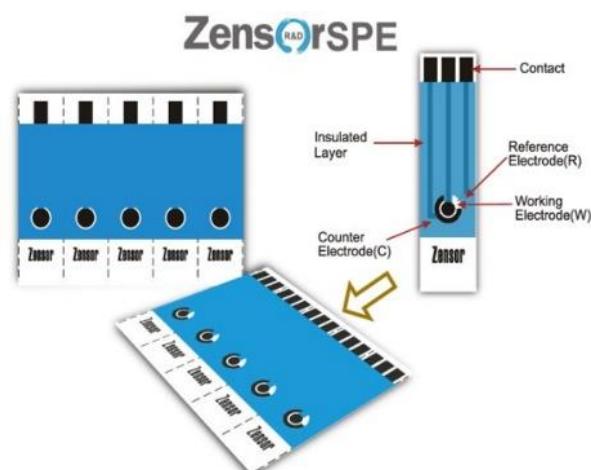


Fig. 5. CVs recorded on (a) silver nanoparticles (b) GO-Ag composite, drop-casted on glassy carbon electrode. Black curve is for without glucose and the red curve is for 4.0 mM glucose. The electrolyte was 0.1 M NaOH. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)





A Disposable Tear Glucose Biosensor— Part 4: Preliminary Animal Model Study Assessing Efficacy, Safety, and Feasibility

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Pankti Shah, BSE^{1,2}, Neil Saez, BSE^{1,2}, Stephanie Maxwell^{1,2},
Teagan Adamson, BSE, MS^{1,2}, Michelle Abou-Eid^{1,2}, Kenyon McAferty, BSE^{1,2},
Dharmendra R. Patel, MD³, and Curtiss B. Cook, MD⁴**

Abstract

Objective: A prototype tear glucose (TG) sensor was tested in New Zealand white rabbits to assess eye irritation, blood glucose (BG) and TG lag time, and correlation with BG.

Methods: A total of 4 animals were used. Eye irritation was monitored by Lissamine green dye and analyzed using image analysis software. Lag time was correlated with an oral glucose load while recording TG and BG readings. Correlation between TG and BG were plotted against one another to form a correlation diagram, using a Yellow Springs Instrument (YSI) and self-monitoring of blood glucose as the reference measurements. Finally, TG levels were calculated using analytically derived expressions.

Results: From repeated testing carried over the course of 12 months, little to no eye irritation was detected. TG fluctuations over time visually appeared to trace the same pattern as BG with an average lag times of 13 minutes. TG levels calculated from the device current measurements ranged from 4 to 20 mg/dL and correlated linearly with BG levels of 75–160 mg/dL ($TG = 0.1723 BG + 7.9448 \text{ mg/dL}; R^2 = .7544$).

Conclusion: The first steps were taken toward preliminary development of a sensor for self-monitoring of tear glucose (SMTG). No conjunctival irritation in any of the animals was noted. Lag time between TG and BG was found to be noticeable, but a quantitative modeling to correlate lag time in this study is unnecessary. Measured currents from the sensors and the calculated TG showed promising correlation to BG levels. Previous analytical bench marking showed BG and TG levels consistent with other literature.

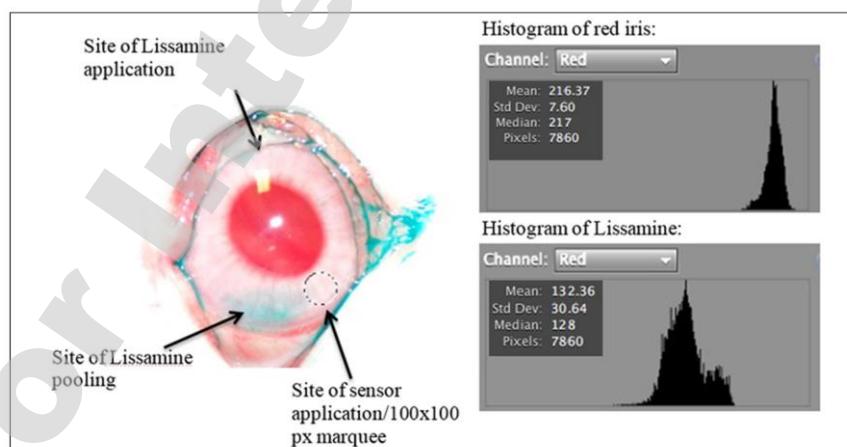


Figure 2. Example of rabbit eye after Lissamine green dye has been applied. Red channel was used to assess potential damage to the eye. Shown in the figure is what is perceived to be an area of Lissamine dye pooling, the site where the tear sensor was applied, and the site of Lissamine application. Also shown are the histograms and the standard deviation of the red iris and Lissamine, showing clear differences between an area of eye with no Lissamine and an area of eye with Lissamine. If the area is suspected to be damaged, an increase in standard deviation will be observed.





A Disposable Tear Glucose Biosensor—Part 3: Assessment of Enzymatic Specificity

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Abstract

Background:

A concept for a tear glucose sensor based on amperometric measurement of enzymatic oxidation of glucose was previously presented, using glucose dehydrogenase flavin adenine dinucleotide (GDH-FAD) as the enzyme. Glucose dehydrogenase flavin adenine dinucleotide is further characterized in this article and evaluated for suitability in glucose-sensing applications in purified tear-like saline, with specific attention to the effect of interfering substances only. These interferents are specifically saccharides that could interact with the enzymatic activity seen in the sensor's performance.

Methods:

Bench top amperometric glucose assays were performed using an assay solution of GDH-FAD and ferricyanide redox mediator with samples of glucose, mannose, lactose, maltose, galactose, fructose, sucrose, and xylose at varying concentrations to evaluate specificity, linear dynamic range, signal size, and signal-to-noise ratio. A comparison study was done by substituting an equivalent activity unit concentration of glucose oxidase (GOx) for GDH-FAD.

Results:

Glucose dehydrogenase flavin adenine dinucleotide was found to be more sensitive than GOx, producing larger oxidation currents than GOx on an identical glucose concentration gradient, and GDH-FAD exhibited larger slope response (-5.65×10^{-7} versus -3.11×10^{-7} A/mM), signal-to-noise ratio (18.04 versus 2.62), and linear dynamic range (0–30 versus 0–10 mM), and lower background signal (-7.12 versus -261.63 nA) than GOx under the same assay conditions. GDH-FAD responds equally to glucose and xylose but is otherwise specific for glucose.

continued →

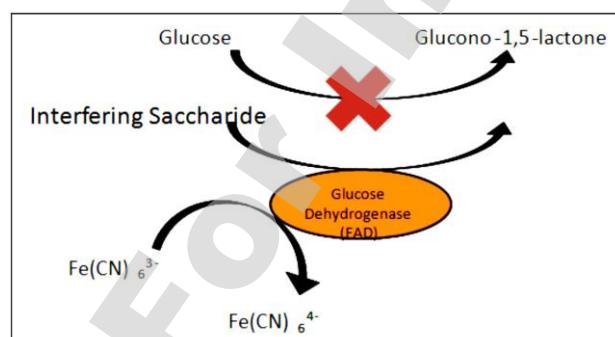
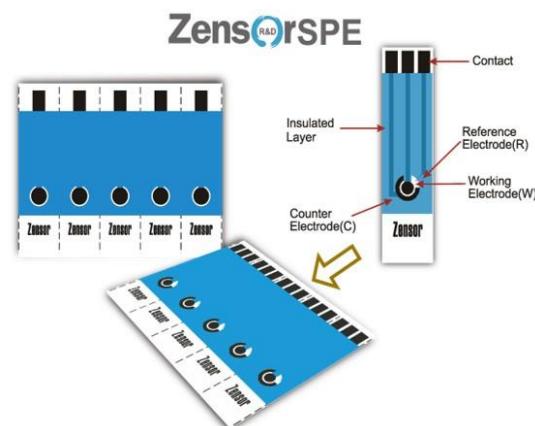


Figure 1. Schematic diagram of saccharide interference that may compete with glucose as a substrate for enzyme activity, thereby falsely increasing the signal strength and masking the true glucose value. $\text{Fe}(\text{CN})_6^{3-}$, ferricyanide.





Highly selective immobilization of amoxicillin antibiotic on carbon nanotube modified electrodes and its antibacterial activity†

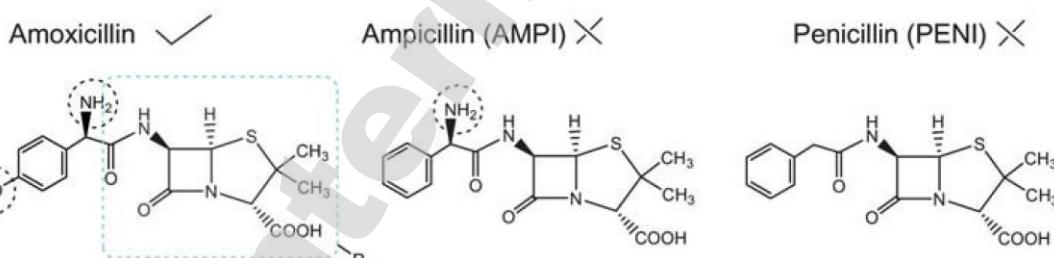
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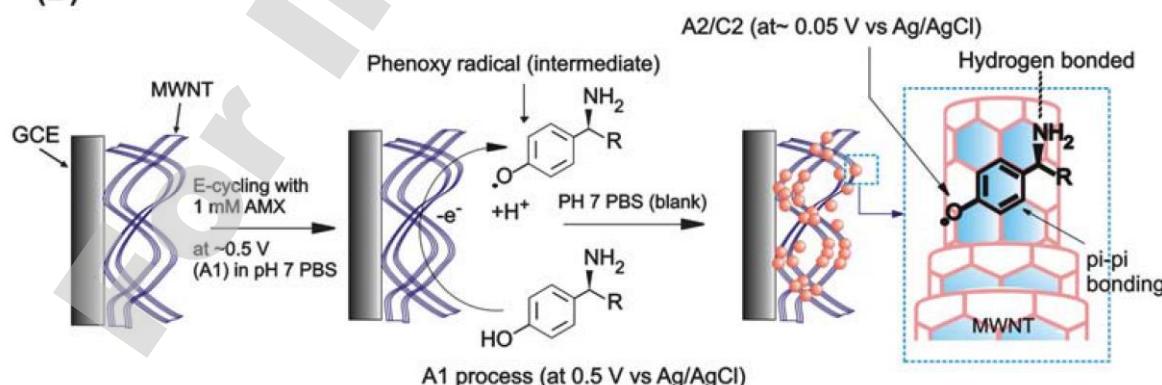
DOI: 10.1039/c0jm02262d

An electrochemical route for highly selective immobilization of a β -lactam family antibiotic, amoxicillin (AMX), from the other drugs, penicillin and ampicillin, on multiwalled carbon nanotube modified glassy carbon electrodes (GCE/AMX@MWNT), without any linkers and surface functionalization, has been successfully demonstrated. The electrochemical response of the AMX on GCE/MWNT showed an irreversible oxidation peak at 0.5 V vs. Ag/AgCl (A1), followed by the growth of a new redox peak at 0 V vs. Ag/AgCl (A2/C2) in pH 7 phosphate buffer solution, which is in parallel to a control phenol electrochemical response, revealed that the phenoxy radical electrogenerated at A1 gets subsequently adsorbed on the underlying MWNT modified electrode with a specific surface confined A2/C2 redox peak with proton-coupled electron transfer behaviour. Physicochemical characterization from X-ray diffraction, transmission electron microscopy and scanning electron microscopy collectively evidenced the immobilization of AMX both on the inner and outer (surface) walls of the carbon nanotubes. Further, the AMX@MWNT hybrid material was found to show enhanced antibacterial activity against three bacterial pathogens, *Escherichia coli*, *Staphylococcus aureus* and *Bacillus subtilis*, over the unmodified AMX and MWNT. Finally, as an environmental pollution remedy, the uptake of the AMX drug from five different simulated sources: river water, sea water, river soil, sea soil and farm milk, was successfully demonstrated by this new electrochemical methodology.

(A)



(B)



(A). GCE/MWNT

(B). GCE/MWNT with AMX

(C). GCE/AMX@MWNT