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Characterization of $COOH-Fe_3O_4/NCC-CTA^+$ on Screen Printed Carbon Electrode Using Field Emission Scanning Electron Microscope and Energy Dispersive X-Ray for DNA Biosensor

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ABSTRACT

A novel DNA biosensing platform was designed by the functionalization of iron oxide (Fe_3O_4) with the carboxylic group via capping agent, mercaptopropionic acid (MPA) and conjugated with nanocellulose crystalline (NCC) surface modified with surfactant cetyltrimethylammonium bromide (CTAB) to assist in the DNA sensing capability. The product of nanocomposites compound was drop-casted on screen printed carbon electrode (SPCE). Characterization by field emission scanning electron microscope (FESEM) and energy dispersive X-Ray (EDX) spectroscopy showing that carboxyl functionalized iron oxide ($COOH-Fe_3O_4$) can be hybridized with $NCC-CTA^+$ via electrostatic interaction.

INTRODUCTION

The Field emission scanning electron microscopy (FE-SEM) is a magnification tool that utilized focused beams of electrons to generate a high resolution, three- dimensional images that provide information on topography and morphology of various surface modified SPCE [1]. Energy Dispersive X-Ray (EDX) spectroscopy is a powerful tool used in an elemental analysis. An x-ray beam from the spectrometer was focused on the working electrode of the modified SPCEs [2]. The excitation of an electron from inner shell of the sample is replaced by electron from outer shell; subsequently, give out a difference in energy between the two shells in the form of characteristics x-rays. The characteristics of the X-rays can be analyzed and the element associated identified [3]. The modified SPCEs show different set of peaks depending on the element present on its surface. Screen printed carbon electrodes (SPCEs) appeared in the 1990s and has attracted a considerable attention for biosensor application. Its production by screen printing technology offer massive benefit attributes over traditional electrode due to their reproducibility in production and repeatability in measurement along with low cost for marketing [4,5]. Fe_3O_4 have been previously reported in many literatures for their potential in sensor, medical and clinical diagnostic 6–8].

[6–8]. Since Fe_3O_4 is highly hydrophobic, hydrophilic surface modification can be done by its covalent bond and physical adsorption of biocompatible polymer to its surface [9]. The surface modification of NCC was previously reported by using surfactant. One the study was found to stabilize nanoparticles by surface modification of Cetyl trymethyl ammonium bromide (CTAB)[10]. The terminology used for surfactant modified nanocrystalline celluloses (NCCs) is CTA-NCCs because the NCC sulfate ester group counterions are exchanged from H^+ or Na^+ to cetyltrimethylammonium (CTA^+). There are some electroactive indicators that is used in detection of hybridization DNA. Electroactive indicators for DNA hybridization work in different affinities for ssDNA and dsDNA. There are four modes of interaction reported, which is interaction involving electrostatic interaction with the negative sugar phosphates structure, interaction of planar aromatic ring systems, preferential binding with guanine bases and groove binding [11].

MATERIALS AND METHODS

Field emission scanning electron microscope (FESEM) The morphological studies of modified SPCEs were carried out using Nova NanoSEM 230. Prior to analysis, modified SPCEs were mounted on sample holder using carbon adhesive and coated with gold ensuring good conducting material for easing the flow of the incident electrons to the samples [1].

Energy dispersive X-Ray (EDX) spectroscopy Nova NanoSEM 230 was used in combination with EDX Spectrometers. The same procedure as FE-SEM was used [2].

RESULTS AND DISCUSSION

FESEM image reveals the images of prepared Fe_3O_4, COOH-Fe_3O_4, NCC/CTAB, and COOH-Fe_3O_4/NCC/CTAB through drop casting on screen printed carbon electrode (SPCE). **Fig.** 1 shows the image of FESEM for (A) bare SPCE (B) SPCE/Fe_3O_4, (C) SPCE/COOH-Fe_3O_4, (D) SPCE/NCC, (E) SPCE/NCC/CTAB, and (F) COOH-Fe_3O_4/ NCC/CTAB

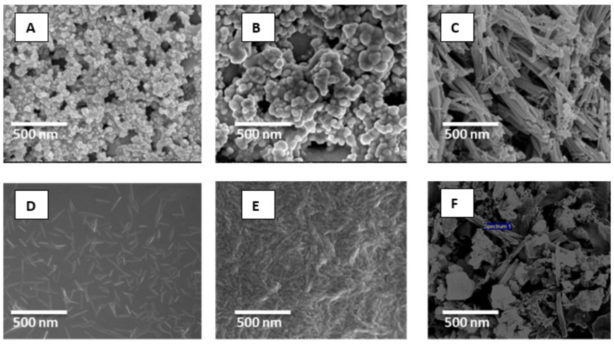
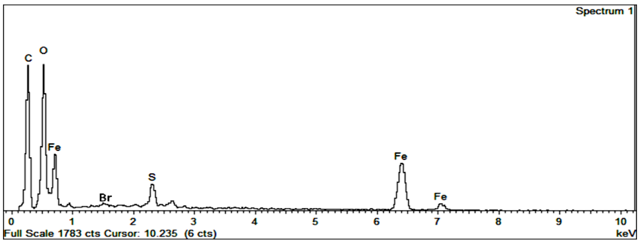


Fig. 1. FESEM of (A) bare SPCE (B) SPCE/Fe_3O_4, (C) SPCE/COOH-Fe_3O_4, (D) SPCE/NCC, (E) SPCE/NCC-CTA^+, and (F) COOH-Fe_3O_4/NCC-CTA^+.

Fig. 1A shows the image of bare SPCE with a globular shape as previously reported by [12,13] and **Fig.** 1B shows a quite spherical shape of Fe_3O_4 close to the FESEM image reported elsewhere [14]. Fe_3O_4 functionalized COOH (**Fig. 1C**) shows a changing morphology with a cylindrical shape compared to unfunctionalized Fe_3O_4 as observed from the previous study [15–17]. These results proved that the deposition of Fe_3O_4 capped with MPA (COOH- Fe_3O_4) can be obtained for SPCE modification.

Fig. 1D shows the image of SPCE/NCC with a rod-like nanocrystal which is in a good agreement with the image previously reported [18,19]. On the other hand, **Fig. 1E** shows the image of randomly oriented whisker NCC-CTA^+**.** This similar image had also been reported by [20,21]. **Fig. 1F** concluded that the compound of COOH-Fe_3O_4/NCC-CTA^+ can be deposited on working surface of SPCE. This image indicates that the deposition of COOH-Fe_3O_4/NCC-CTA^+ on SPCE surface was succeed and supported by the EDX spectra result (**Fig. 2**) which confirm the present of Fe, S, C, Br and O.



Element	Weight%	Atomic%
C K	37.16	50.63
O K	29.83	32.95
Br K	1.23	5.85
S K	2.20	1.21
Fe K	29.58	9.36
Totals	100.00	

Fig. 2. The EDX spectra of SPCE/COOH-Fe_3O_4/NCC-CTA^+.

The physical characterization of FESEM and EDX proved the morphology changing and element contents present indicates the successful of nanocomposites (COOH-Fe_3O_4/NCC-CTA^+) material deposition on SPCE.

CONFLICT OF INTEREST

The author declares that there is no conflict of interest.

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