



Oct 21 Oct 23, 2015

Bitec, Bkk

SYNTHESIS AND CHARACTERIZATION OF Ni AND Cu HEXACYANOFERRATE NANOPARTICLES FOR HYDROGEN PEROXIDE SENSOR

Anurak Chitbuengphrao,¹ Tanin Tangkuaram,² Anchana Preechaworapun^{1*}

¹ Program of Chemistry, Faculty of Science and Technology, Pibulsongkram Rajabhat University, Phitsanulok, Thailand 65000

² Department of Chemistry, Faculty of Science, Maejo University, Chiang Mai, Thailand 50290

*e-mail: anchanaaph@yahoo.com

(27)

Oct 20

Abstract: The Ni hexacyanoferrate (NiHCF) and Cu hexacyanoferrate (CuHCF) nanoparticles have been synthesized, characterized, and used as electrocatalyst for the hydrogen peroxide determination. Metal hexacyanoferrate nanoparticles were synthesized by mixing each metal chloride and $K_3[Fe(CN)_6]$ in 0.05 mM KCl. X-ray powder diffraction, scanning electron microscopy, FTIR-spectroscopy and UV data suggest the formation of nanocrystalline compound. NiHCF and CuHCF nanoparticles with glutaraldehyde were modified on a glassy carbon electrode (GC), and characterized with cyclic voltammetric analysis. Their voltammograms exhibit a well pair of reversible redox peaks and excellent ability electrocatalysis for H_2O_2 determination by amperometry at the potential 0.0 V and -0.1 V, respectively. The linear calibration curve the concentration range 0.1–2.0 mM and 0.5–2.8 mM H_2O_2 was constructed for NiHCF and CuHCF nanoparticles modified GC electrode. Limit of detection (S/N = 3) of 30 mM and 45 mM H_2O_2 was calculated for nanoparticles modified GC electrodes, respectively.

Introduction: The determination of hydrogen peroxide (H_2O_2) has been widely used in chromatography, chemiluminescence and electrochemistry for application in many areas such as environment, biology, food, industry and clinical laboratory. Generally, electrochemical technique has been frequently applied in many areas, because of advantages of this method e.g. simple equipment, low operating expense and fast analytical time. The objective of paper is to study the characteristic and electrocatalytic properties towards H_2O_2 of NiCNF and CuCNF nanoparticles. The NiCNF and CuCNF were found to be dispersed well in water and stable without stabilizer addition. Their modified glassy carbon electrodes were shown facilitates low potential and sensitive detection of hydrogen peroxide.

Methodology: $K_3[Fe(CN)_6]$ was purchased from Ajax Finechem (Newzealand). $NiCl_2 \cdot 6H_2O$ was purchased from Carloerba (UN). $CuCl_2 \cdot 2H_2O$ was purchased from J.T.BAKER (USA). 25% Glutaraldehyde solution was purchased from Wako (Japan). Cyclic voltammetric measurements were carried out on CHI 1230A Electrochemical analyzer Serial: F1030 (HCH Instruments, USA.). A three electrode cell (10 ml) with the modified glassy carbon electrode as the working electrode, a saturated silver/silver chloride electrode (Ag/AgCl) as reference electrode and a stainless steel electrode as counter electrode was used. Fourier transform infrared spectra (FT-IR) measured by KBr pellets was obtained on a WQF-410 Fourier transform infrared spectrophotometer (FTIR-8900, SHIMADZU Japan). Transmission electron microscope (SEM) image was taken with a transmission electron microscope (LEO 1450 VP LEO, Germany). X-ray Diffractometer (XRD) (D-500 SIEMENS, Germany) UV-vis adsorption spectra were recorded on a Perkin-Elmer FT-IR spectrometer (FTIR-8900, SHIMADZU, Japan).

NiCNF and CuHCF nanoparticles were synthesized by dropwise addition of 100 ml of a 0.05M aqueous solution of metal chloride ($NiCl_2$ or $CuCl_2$) to a 100 ml solution of 0.05M $K_3Fe(CN)_6$ containing 0.05 M KCl under stirring condition. After complete addition, precipitation was occurred, then rotated in centrifugal machine and washed with deionized water for several times. After filtration, they were dried overnight in a vacuum at room

temperature to give a powdered substance. To fabricate the sensor, NiHCF was suspended in glutaraldehyde solution, then coated on the GC electrode and dried at room temperature.

Results, Discussion and Conclusion: The NiNPs modified electrode was characterized by SEM images in Figure 1(B) that images compared with bare GC electrode in Figure 1(A). The NiNPs modified electrode was prepared with drop a mix solution of NiNPs and glutaraldehyde onto GC electrode. The NiNPs were dispersed on GC surface at the average particle size as 100 nm.

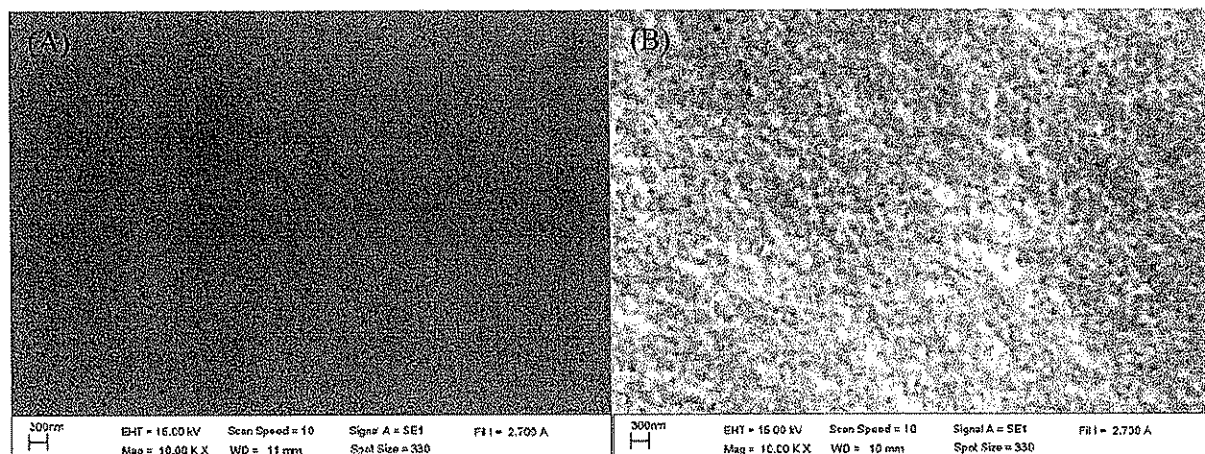


Figure 1. Scanning electron micrograph images of the surface of bare GC electrode (A) and NiHCF modified GC electrode (B).

To further demonstrate the advantages of the materials, a control experiment was conducted. Firstly, the NiHCF was analyzed by X-Ray diffraction (XRD) and the result is shown in Figure 2(A) where showed a typical face-centered cubic (fcc) structure, indicating the presence of NiHCF pattern [1]. It can be observed that the peaks are broaden, indicating the existence of crystallites of nanometric dimensions. Figure 3 shows the FT-IR spectra of the NiHCF and CuHCF nanoparticles. The FT-IR spectra presence of metal cyanoferrate nanoparticle fingerprint [2, 3].

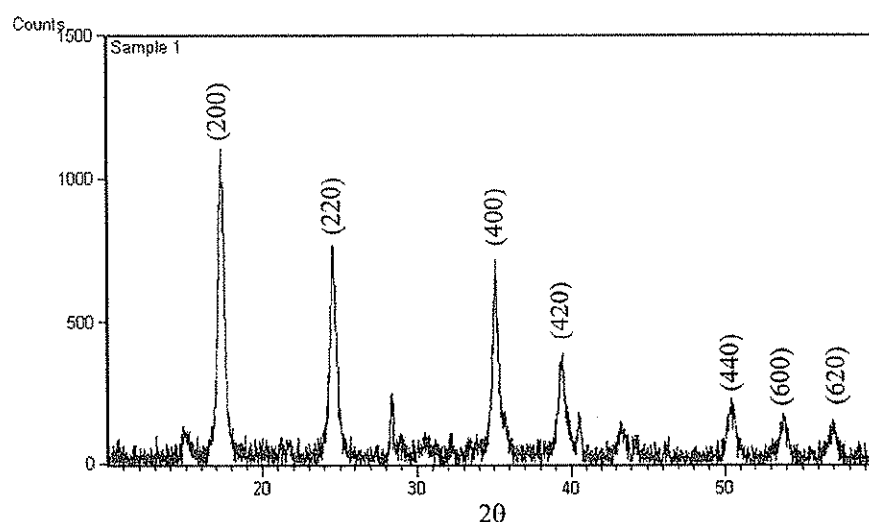


Figure 2. X-Ray diffraction pattern of NiHCF nanoparticles synthesis

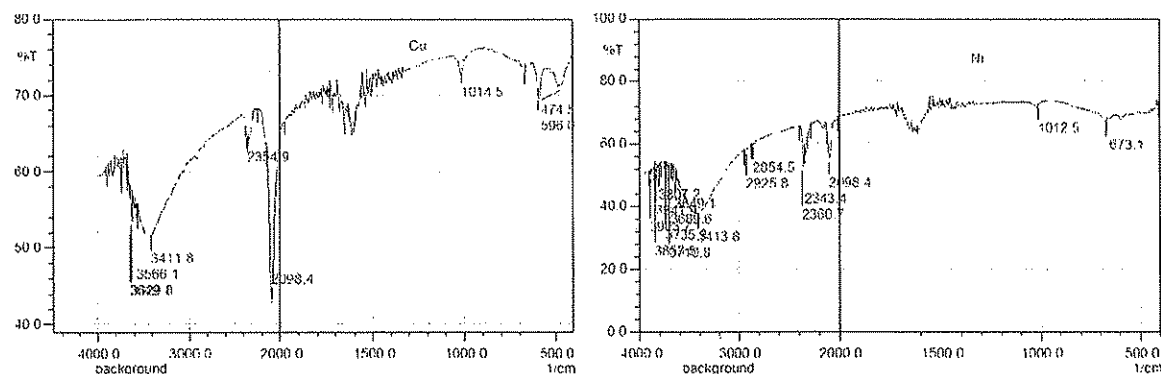


Figure 3. FT-IR spectra of CuHCF (A) and NiHCF (B)

Dropping 2 μ l of 2.5% w/v NiHCF and CuHCF in glutaraldehyde solution on two electrodes, the CVs of the recorded was shown in Figure 4. It is noticed only one redox couple, independently of the cations responsible for the charge compensation. This redox process is related to the $\text{NiII/FeII} \rightarrow \text{NiII/FeIII}$ and $\text{CuII/FeII} \rightarrow \text{CuII/FeIII}$ transition and is equivalent to the second redox couple in PB system [1]. The result can be attributed to the particles attached on the GC electrode. To obtain the optimal potential for amperometric detection at NiHCF/GC and CuHCF electrode, the electrochemical behavior of H_2O_2 was studied by apple potential at 0.3 to -0.3 V and 0.1 to -0.2 V, respectively. The maximum current occurred at 0.0 and 0.1 V, respectively. Hence, this potential was set for quantitative amperometric potential detection in H_2O_2 determines experiments. The linear calibration curve the concentration range 0.1–2.0 mM and 0.5–2.8 mM H_2O_2 was constructed at the NiHCF and CuHCF nanoparticles, respectively. Limit of detection ($\text{S/N} = 3$) of 30 mM and 45 mM H_2O_2 was calculated for NiHCF and CuHCF nanoparticles modified GC electrodes, respectively. The excellent sensitivity was received 73 A/ mM at $R^2 = 0.9960$.

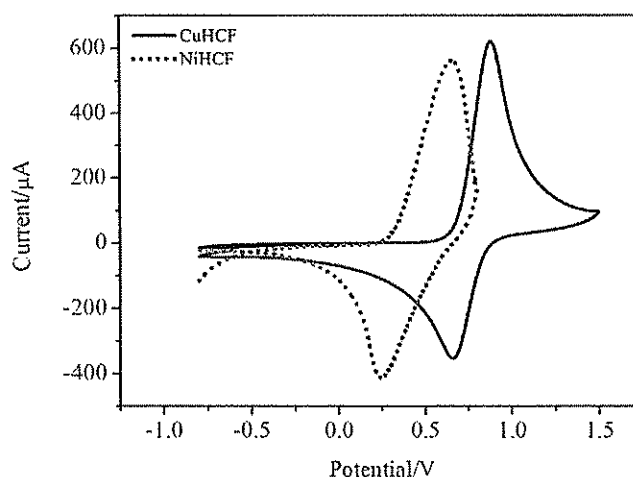


Figure 4. Cyclic voltammograms of modified NiHCF and CuHCF nanoparticles on GC electrodes in PBS buffer (pH 7.0)

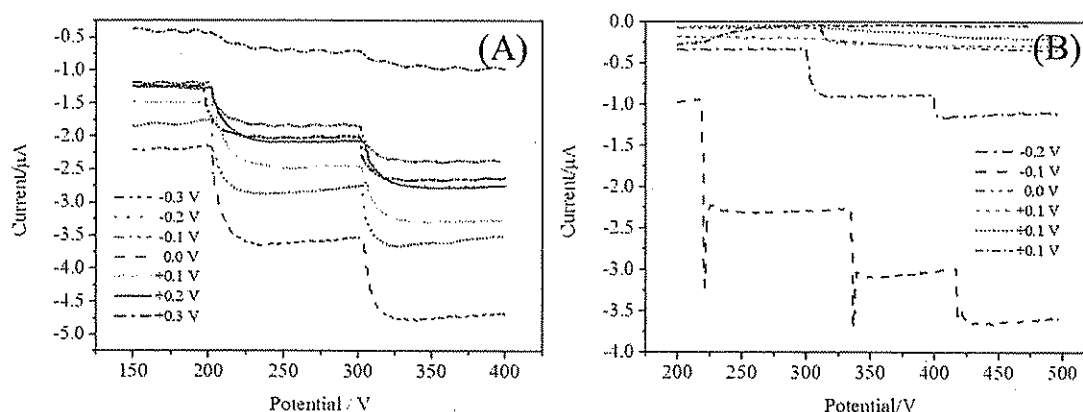


Figure 5. Amperograms of H_2O_2 in PBS buffer (pH 7.0) using NiHCF (A) and CuHCF (B) nanoparticles modified GC electrodes at various potentials.

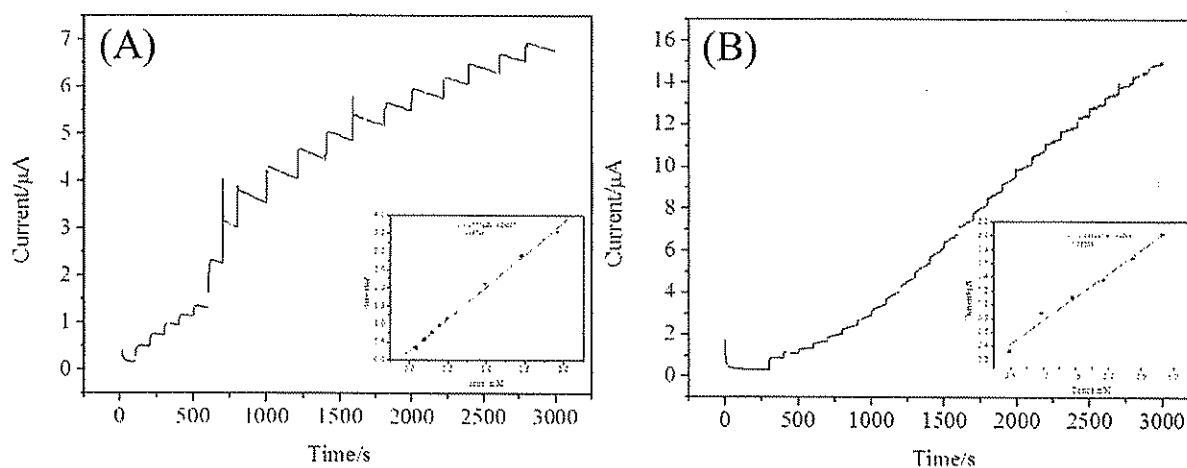


Figure 6 Amperograms of successive addition of H_2O_2 in 0.05 M PBS buffer (pH 7.0) using NiHCF (A) and CuHCF (B) nanoparticles modified GC electrodes at the potential 0.0 and -0.1 V, respectively. The inset showed linearity of calibration graphs.

In conclusion, the fabrication of NiHCF and CuHCF nanoparticle modified GC electrode was studied for H_2O_2 determination. This H_2O_2 sensor provided the good analytical performance such as low detection limit, high sensitivity, and wide linear range.

References:

1. Baioni A. P., Vidotti M., Fiorito P. A., and Torresi S. I. C., J Electroanal Chem 2008;622:219.
2. Yang M., Jiang J., Lu Y., He Y., Shen G., and Yu R., Biomaterials 2007;28:3408.
3. Yang M., Yang Y., Qu F., Lu Y., Shen G., and Yu R., Anal Chim Acta 2006;571:11.

Acknowledgements: Anchana Preechaworapun acknowledges The Research and Development Institute, Faculty of Science and Technology of Pibulsongkram Rajabhat University.

Keywords: hydrogen peroxide, NiHCF, CuHCF, amperometry



STT39-0620



Science Society of Thailand Under the Royal Patronage of His Majesty the King, Faculty of Science, Chulalongkorn
University, Phya Thai Rd., Bangkok 10330
Tel: 0-2218-5245 Fax. 0-2252-4516
E-mail: stt39@scisoc.or.th

Acknowledgement

Dear ANURAK CHITBUENGPHRAO,

The secretariat committee of STT39 would like to inform you that we have received your STT39 registration information for ID: 0620 and e-mail address: zoom_kkk@hotmail.com

The secretariat committee of STT39 have received your registration as: Participant with paper submission

We recieved the payment as -

with amount 1,500.00 baht (หนึ่งพันห้าร้อยบาทถ้วน)

Your submitted paper entitled SYNTHESIS AND CHARACTERIZATION OF Ni AND Cu HEXACYANOFERRATE NANOPARTICLES was reviewed from academic subcommittee of STT with the result : Poster

Sincerely yours,

Associate Professor Dr. Thararat Supasiri

Welcome to STT39

[Registration](#)[Scientific Sections](#)[Important dates](#)[Paper Guideline](#)[Download](#)[The Venue](#)[Accommodation](#)[Committee](#)[Exhibition](#)[Future Congresses](#)[Sponsors](#)

Program

[Overview](#)

Welcome to The 39th Congress on Science and Technology of Thailand

"Innovative Sciences for a Better Life"

October 21 - 23, 2013 at BITEC, Bangkok, Thailand.

The Science Society of Thailand under the Patronage of His Majesty the King in association with the Faculty of Science, King Mongkut's University of Technology Thonburi, Thailand are pleased and privileged to invite you to join the 39th Congress on Science and Technology of Thailand (STT39), which will be held in Bangkok from 21 to 23 October, 2013.

The Congress on Science and Technology of Thailand (STT) is an important national event which is organized annually. It is the biggest science and technology conference in Thailand with more than two thousands of participants from Thailand and abroad, attending the conference each year. The aim of the congress is to bring together national and international scientists and technologists as well as young Thai scientists from vast areas of science and technology and to give them an opportunity to share and exchange their experiences.

Typically, the congress Premier Keynote Lecture is from a

Latest News

[Keynote Speaker Announcement](#)[Register Announcement](#)[Registration Manual and Example Template](#)

Premier Sponsors

