



Novel synthesis of nickel–iron hexacyanoferrate nanoparticles and its application in electrochemical sensing



P.C. Pandey*, Digvijay Panday

Department of Chemistry, Indian Institute of Technology (Banaras Hindu University), Varanasi 221005, India

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ABSTRACT

We report herein a novel method for the synthesis of nickel–iron hexacyanoferrate nanoparticles involving the participation of tetrahydrofuran and hydrogen peroxide. It has been found that desired ratio of potassium ferricyanide and nickel sulphate is converted into nickel–iron hexacyanoferrate nanoparticles in the presence of tetrahydrofuran and hydrogen peroxide within 30 min at 60 °C. The as synthesized nickel–iron hexacyanoferrate nanoparticles have been characterized by UV–Vis spectroscopy, Fourier transformation infrared spectroscopy (FT-IR), X-ray diffraction analysis (XRD), energy dispersive spectroscopy analysis (EDS), Transmission Electron Microscopy (TEM) and cyclic voltammetry. The average sizes of nanoparticles are found in the order of 31 nm. The nickel–iron molar ratio significantly influences the electrochemical behaviour of as synthesized nanoparticles modified electrode. Electrochemical sensing of hydrazine has been examined on mixed metal hexacyanoferrate modified electrode with analytical sensitivity in the order of $135.76 \mu\text{A mM}^{-1} \text{cm}^{-2}$ and lowest detection limit of 50 nM. Apart from its use in electrochemical sensing, mix metal nanoparticles are soluble in aqueous media and show excellent peroxidase mimetic activity with the Michaelis–Menten constant and V_{max} in the order of 1.5 mM and $3.06 \times 10^{-7} \text{m s}^{-1}$ respectively for H_2O_2 mediated oxidation of o-dianisidine thus representing novel material for both homogeneous and heterogeneous catalysis.

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1. Introduction

Transition metal hexacyanoferrate (MHCF) have gained a huge interest among scientist due to its unique physical and chemical properties which is exploited in the field of electrochemistry, environmental and medical science, electrode material in rechargeable batteries, molecular magnets and many more. When –CN group surrounds iron (Fe) metal centre then it is known as Prussian blue but when –CN group coordinates two metal centres and –Fe is one of the member then it is known as Prussian blue analogue [1]. Prussian blue and its analogues are generally synthesized either by co-precipitation or by electrodeposition method. Potential viability of mixed metal hexacyanoferrate have directed extensive investigation involving different combination of transition metals like Co–Fe [2], Ni–Co [3,4], Cu–Co [5], Ni–Pd [6] and Ni–Fe [7,8]. The electrocatalytic response and electrochemical behaviour of metal hexacyanoferrate analogues are relatively better as compared to that of single metal hexacyanoferrate due to the presence of another transition metal in the cubic structure along with iron and have been exploited in electrochemical energy storage [7], electrochemical sensing of glutathione and hydrogen peroxide [9, 10], dopamine [11] and many more. The practical applications of these metal hexacyanoferrate analogues are restricted due the insolubility

of the material for further processing in desired applications. In addition to that precise control over nanogeometry, crystallinity and catalytic activity of such material are another challenging task of research findings restricting the wider application of such materials. Therefore, there is a need of a novel synthetic approach that enable the synthesis of water soluble metal hexacyanoferrate analogues at room temperature in a rapid and cost effective manner. Meeting these requirements, we have recently investigated the synthesis of processable Prussian blue through the active participation of 3-aminopropyltrimethoxysilane (3-APTMS) and cyclohexanone [12] displaying excellent electrochemistry with the electron transfer rate constant in the order of 32s^{-1} . Similar reaction protocol has subsequently been exploited for the synthesis of super peroxidase mimetic mix metal analogue nanoparticles [9]. Although such method yielded the synthesis of processable mix metal hexacyanoferrate nanoparticles, the use of 3-APTMS enabled autohydrolysis, condensation and polycondensation leading to the formation of –Si–O–Si– linkage during prolonged operation of the materials. Fortunately, we succeeded in replacing the use of 3-APTMS by tetrahydrofuran hydroperoxide (THF-HP) that enabled controlled synthesis of water soluble Prussian blue nanoparticles (PBNPs) at room temperature [13]. Although the use of THF-HP allowed the synthesis of processable PBNPs, the synthesis of mix metal hexacyanoferrate remained in question due to relatively slow process of Prussian blue formation in the presence of THF-HP. In addition to that the availability of THF-HP for cost effective synthesis of such material has been another

* Corresponding author.

E-mail address: pcpandey.apc@iitbhu.ac.in (P.C. Pandey).

Table 1
Synthesis of Ni–FeHCF as a function of variable composition of potassium ferricyanide and nickel sulphate.

Vial	Potassium ferricyanide (mol l ⁻¹)	Tetrahydrofuran (mol l ⁻¹)	H ₂ O ₂ (mol l ⁻¹)	Nickel sulphate (mol l ⁻¹)	Ni–FeHCF formation
A	0.035	1.20	0.7	–	(Deep blue) PB formation
B	0.035	1.20	0.7	0.001	Blue
C	0.035	1.20	0.7	0.003	Blue
D	0.035	1.20	0.7	0.006	Light blue
E	0.035	1.20	0.7	0.009	Light blue
F	0.035	1.20	0.7	0.013	Green

issue since commercial availability of the same is rare and laboratory preparation of material is also tedious. Accordingly, we attempted to find out the way for controlling the synthesis of water soluble mix metal hexacyanoferrate nanoparticles. We found that the use of tetrahydrofuran and hydrogen peroxide, both of them are common laboratory reagents, efficiently convert the synthesis of nickel–iron hexacyanoferrate within <30 min at 60 °C displaying excellent nanogeometry, crystallinity and processability for practical applications. Accordingly, the findings on: (i) tetrahydrofuran, hydrogen peroxide, and nickel sulphate based conversion of potassium ferricyanide into its nickel–iron hexacyanoferrate (hereinafter Ni–FeHCF), (ii) structural and elemental characterization of as synthesized nickel–iron hexacyanoferrate, (iii) homogeneous catalysis of as synthesized nickel–iron hexacyanoferrate towards hydrogen peroxide determination, and (iv) electrocatalytic ability of as synthesized nickel–iron hexacyanoferrate modified electrode in hydrazine sensing are studied and reported in this manuscript.

2. Experimental

2.1. Material

All the reagents were of analytical grade and used without further purification. Potassium ferricyanide [K₃Fe(CN)₆], hydrogen peroxide (H₂O₂), and nickel sulphate (NiSO₄) were purchased from Merck, India. o-dianisidine, graphite powder (particle size 1–2 μm), Nujol oil (density 0.838 g ml⁻¹), and Hydrazine hydrate were obtained from Sigma Aldrich Chemical Co. India. Tetrahydrofuran (THF) was received from Alfa Aesar, India. The water used in all the experiments was double distilled water (Alga water purification system).

2.2. Synthesis of Ni–FeHCF

Synthesis of Ni–FeHCF involves mixing of optimized concentration of potassium ferricyanide, tetrahydrofuran, hydrogen peroxide and

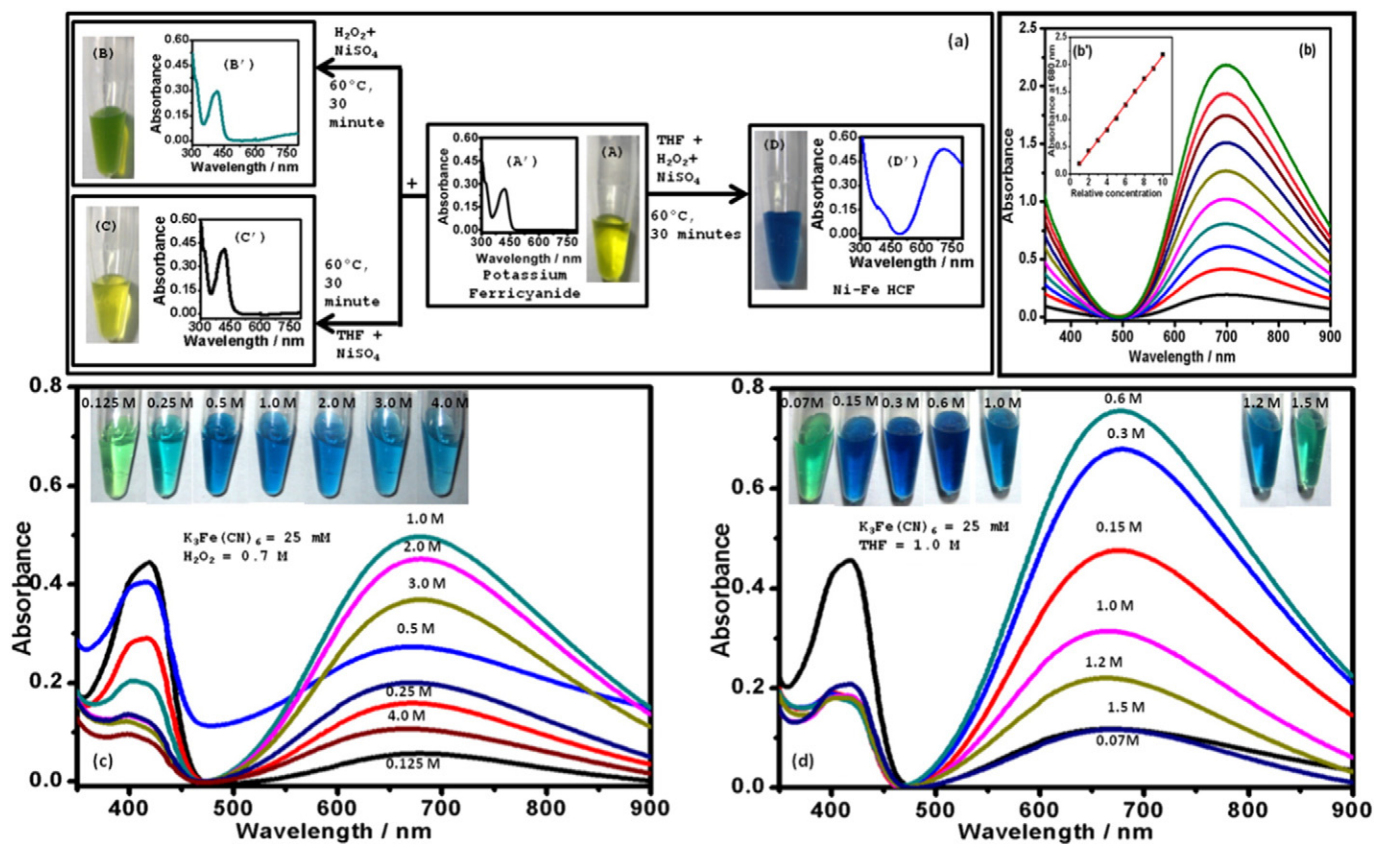


Fig. 1. (a) Schematic presentation of Ni–Fe hexacyanoferrate formation from tetrahydrofuran, hydrogen peroxide and potassium ferricyanide and nickel sulphate; (b) UV–Vis spectra of Ni–FeHCF in water, inset shows the dependence of absorption maxima (λ_{\max}) on relative concentration of Ni–FeHCF; (c) and (d) shows the effect of tetrahydrofuran and hydrogen peroxide on the formation of Ni–FeHCF respectively at constant molar ratio of Ni and Fe (1:5).

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