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GNP-COPPER HEXACYANOFERRATE MODIFIED ELECTRODE FOR VOLTAMMETRIC CHARACTERIZATION AND BEHAVIOR IN THE PRESENCE OF BUTYLATED HYDROXYANISOLE

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ABSTRACT
The modification of a graphite electrode with copper hexacyanoferrate as mediator is discussed how to determine Butylated Hydroxyanisole (BHA). Copper hexacyanoferrate modified electrode was equipped by dipping of metal solution on a GNP-L-Cys-wax (gold nano particle- L-cysteine) composite graphite electrode. The chemically modified electrode's electrochemical behavior was assessed by FESEM, UV and electrochemical studies etc. The cyclic voltammogram obtained by applying the potential flanked by 200 mv and 900mv using 0.1 M KNO_3 solution showed a pair of redox peaks. BHA exists to undergo electrocatalytic oxidation at the working electrode. The feat of the working electrode in static and dynamic conditions to be appraised. The rectilinear current retort of the working electrode under both conditions for the determination of BHA confirms its utility as an amperometric sensor for batch and flow systems. The working electrode preserve is simply erect by a simple immobilization method and has the advantages of good dependableness, hasty retort and astonishing firmness. The use of the modified electrode as an amperometric sensor for determining BHA in commercial samples has been proposed.
Keywords: Copper hexacyanoferrate, BHA, GNP, Wax Composite Electrode and CME.
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INTRODUCTION

In recent years, the determination of compounds of biological interest at reduced overpotentials is the primary goal of scientists of electrochemistry. Chemically Modified Electrodes (CMEs) have gained importance as analytical probes for the amperometric detection of natally relevant chemicals. This is dependent on the intrinsic selectivity attained in CMEs as a result of an intentional change of the electrode surface.[1] The working electrodes accordingly offer a substitute to the conventional ones with the additional advantage of reducing the overpotential. Working electrodes be primarily based on the immobilization of redox species on the surface of the electrode. These redox sorts arbitrate the transmission of electrons from the electrode to the substrate, diminish the overpotential of intention substrates and improve sensitivity and exactitude.[2] Accessory of the electroactive sort to an electrode can be achieved by electrodeposition, surface assimilation, ensnare them hooked on polymer matrices and mechanically transferring them onto the solid electrode façade.[3] Diverse inorganic complexes dole out as modifying agents.[4] An imperative faction of inorganic complexes that have been used for electrode modification is the transition metal hexacyanoferrates. Owing just before their affluent electrochemical possessions, they provide a wide range of applications in assorted fields.[5-10] A numeral cramming established on working electrodes with metal hexacyanoferrates have earlier been reported for the determination of analytes of environmental and biological interests.[11-19]

BHA (Butylated hydroxyanisole) is a chemical antioxidant that is used as a preservative in food, food sleeves, animal feed, and medicinal products. Examine, foundation, into rubber followed by gasoline crop, it is also a preservative used for vitamin A. It has been accompanied by matured fats and fat content foods for its antioxidant assets. It is expansively worn because of its soaring thermal stability and ability

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to remain active in parched and frizzled meals, it is used in culinary meticulousness, belongings are not mislaid the whole time of catering. The instructive and resilience of BHA into an assortment of preparations suit indispensable in scrutiny of its magnitude inside varied perception. Foodstuffs into BHA to be studied consist of food, superficial, pharma[20], artificial[21], fat[22] moreover drugs.[23] Our preceding workings take account of the Silver, Manganese hexacyanoferrate electrode used for the amperometric determination of BHA[24-25] and cobalt hexacyanoferrates electrode.[26] Current exploration, a flow method has been developed for the electrocatalytic oxidation of the analyte with a CuHCF electrode for the detection of BHA The strategy has shown to be beneficial to the BHA's fortitude in commercial preparations.

EXPERIMENTAL

Chemicals and Reagents

Copper chloride and potassium hexacyanoferrate be purchased commencing Merck. Other chemicals are analytical rating and used as bought. To sustain the pH, KOH & HNO_3 are used for pH maintenance. Double distilled water be used all over the experiments for solution preparations.

Instrumentation

FE The nanoparticles were photographed using a scanning electron microscope (SEM) (S-3400N, Hitachi, Japan). A fiber optic spectrometer (Ocean Optics, USA) was used to record the UV-Vis spectra of the substrates, with a deuterium tungsten source light, range as of 200 – 800 nm. Voltammetric and amperometric studies were performed using an electrochemical workstation model CHI 660b coupled with a delicate system. A conventional three-electrode cell assembly was used and the experiments were performed at room warmth (25°C). Three electrode systems were used.

Synthesis of the Modified Electrode (GNP – L-Cys-CuHCF)

Route way of the working electrode was done by a six-step methodology.

1. Preparation of Gold Nanoparticle Using Citrate (GNP)

The initial stair involves the creation of tri sodium citrate capped colloid GNP by sodium borohydride reduction method, as reported.[27]

2. Gold Nanoparticles by Physical Adsorption (GNPs) onto Graphite Powder

GNPs were physically adsorbed onto graphite powder by swirling 1g of graphite smash in a 0.6 micrometer elucidation of hundred and twenty-five milli liter GNP into a sheltered beaker up to three hours. The graphite crush was centrifuged, filtered and dried at room temperature.

3. GNP Preparation Adsorbed Graphite Wax Composite Electrode

Using GNP adsorbed graphite crush, a paraffin wax graphite composite electrode was created. The ratio of GNP capped graphite to wax was optimized to 4:1 because the aforementioned ratio was shown to be effective in both conduction and solidity. Together, the electrode's crack ends are refined and electrical contact is made with one end while the other serves as the functioning exterior.

4. GNPs Graphite Wax Composite Electrode with Adsorbed L- Cys

Equipped GNPs capped wax graphite mixed electrode surface was dipped in 20mM of L- Cys solution for two hours then taken out and washed.

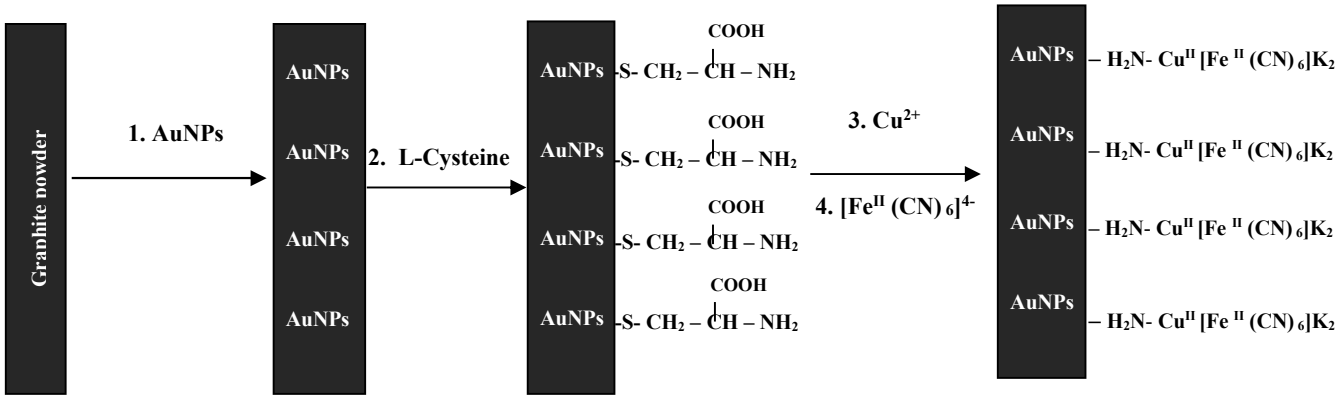
5. Co-ordination of Cu^{2+} on the Pre-mixed Electrode

After dipping the electrode façade in 0.01M ethanolic $CuCl_2$ for around six minutes, the Cu^{2+} ion was coordinated to the cysteine amine present on the graphite wax composite electrode.

6. Derivation of Hexacyanoferrate From Divalent Metal Ions

The Cu^{2+} ions were subsequently derivatized by cycling the electrode for 20 cycles between -200mv and 1000 V in 0.02 M $K_4[Fe(CN)_6]$ solution through 0.1 M KNO_3 while serving as an electrolyte, resulting in

a stable thin coating of CuHCF on the outside. The electrode should be cleaned with DDW after cycling (double distilled water). Graphical depiction of the intact falsehood approach of the three different CuHCF film modified electrode is shown in Scheme-1.



Scheme-1

Real Sample Analysis

Butylated Hydroxy Anisole (BHA)

A commercial food sample (biscuits) was used for investigation. The biscuit sample was scrutinized to contain no preservatives. As a result, they have a spike among BHA intended for scrutiny. The sample was purchased in a confined superstore. The practice of groundwork of taster is as follows. Concerning 500 mg of biscuit, be taken as a crush. BHA was added to the sample at a known amount. The substrate was extracted by three-five milli liter portions of ethyl acetate. 3 haul out to be varied and spotless throughout a watt man twist paper. In the crate, the scum was not obvious, CH_3OH was mixed, then the dig out was shaken and clean again. A known aliquot was taken from this for analysis.

RESULTS AND DISCUSSION

UV Analysis

The AuNPs turned into characterized the usage of UV-Vis spectroscopy and the most absorption turned into discovered about 530 nm Fig.-1 corresponds to the particle length of much less than 25 nm [28]. The FESEM photo of AuNPs as proven in Fig.-2 additionally confirms the particle length turned withinside the variety of 10 – 25 nm.

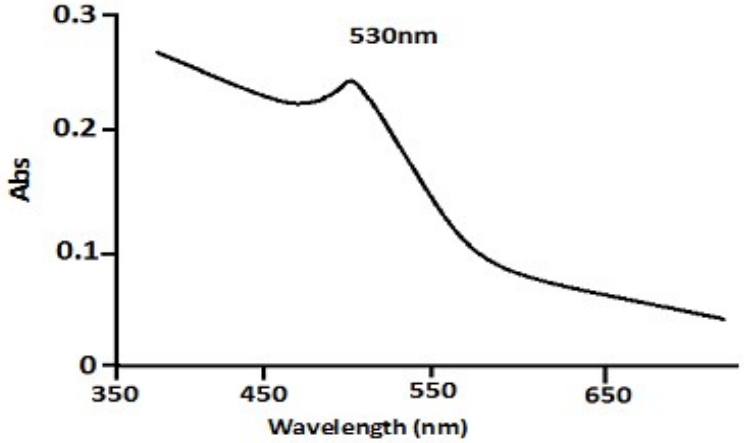


Fig.-1: UV-Visible of Citrate Capped AuNPs

FESEM Studies

The FESEM photographs supplied in Fig.-2a confirm the formation of CuHCF debris inside the length tiers of a hundred to one hundred fifty nm. The presence of AuNPs at the electrode with inner lengths ranging between 35 and 40 nm is seen in Fig.-2b for the AuNPs adsorbed GW composite floor. and the naked electrode picture is proven with inside the Fig.-2c.

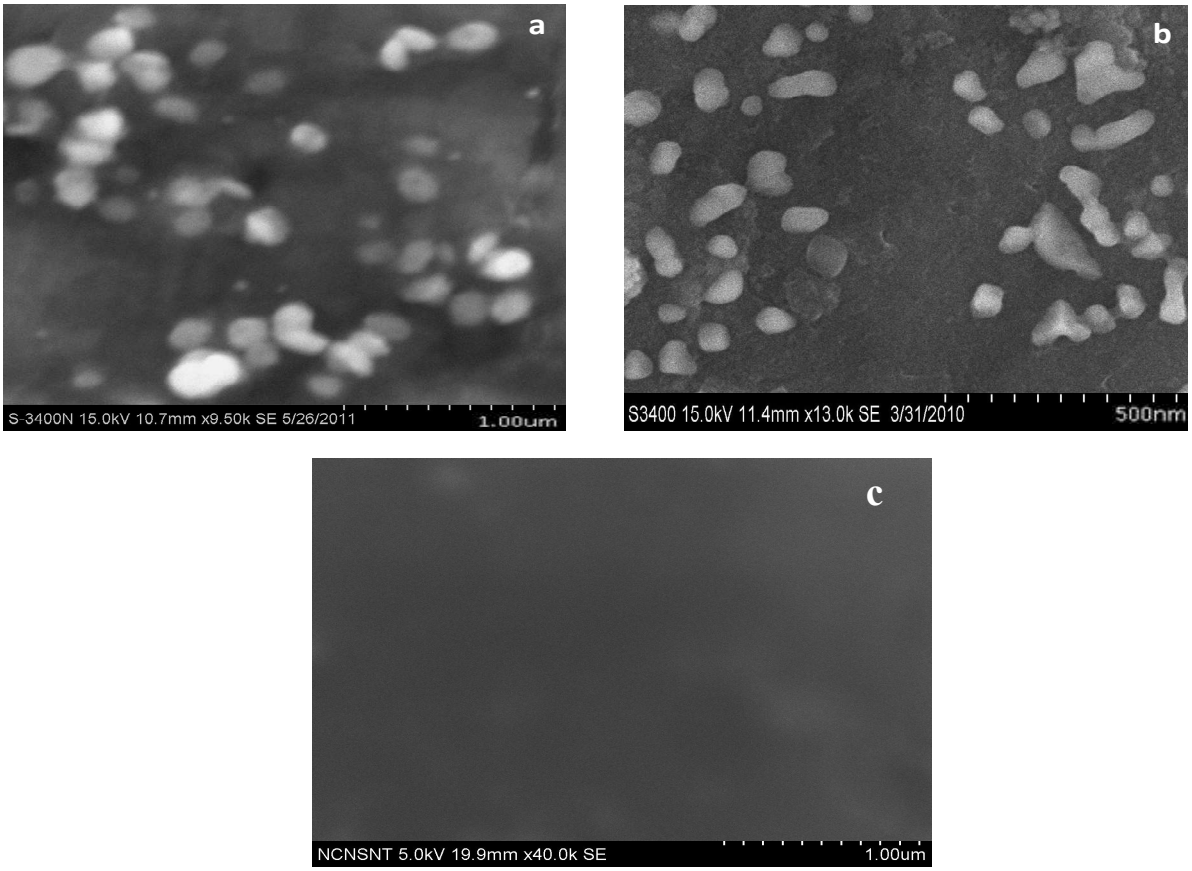


Fig.-2: FESEM Images of (a) CuHCF Film Modified Electrode (b) Citrate Capped AuNPs (c) Bare Graphite Wax Composite Electrode

Electrochemical Characterization of the CuHCF Electrode

Cyclic Voltammetric Studies of CuHCF Electrode

Electrochemical behavior of the CuHCF film electrode (That is CuHCF film on L- Cys functionalized gold nanoparticles graphite wax composite The performance of cyclic voltammetry was investigated. Exploration of the CuHCF film electrode using CV (Cyclic Voltammetry) exhibits a couple of peaks similar to the CuHCF film whilst the potential changed into swept with inside the array starting 2 hundred mV to a thousand mV at a test pace of 20 mV/s at KNO_3 (0.1 M) (Fig.-3,curve c). The naked electrode's (That is graphite wax composite electrode) reaction below comparable condition does now no longer display any feature wave (Fig.-3, curve a). In toting up, The CuHCF film modified electrode with AuNPs was found to have a greater peak current when compared to the CuHCF film modified electrode without AuNPs, as shown in curve b of Fig.-3. CuHCF electrode shows a cathodic peak at 560mV and anodic peak at 610mV. The prescribed potential $E^{0'}$ [$E^{0'}=(E_{pa}+E_{pc})/2$] was intended to exist 585mV, wherever E_{pa} is the oxidation crest potential and E_{pc} is the cathodic peak potential. The exterior exposure of CuHCF film (Γ) was premeditated to be 9.48 x 10^{-9} mol cm^{-2}.

Effect of Supporting Electrolyte

The recital of the working electrode was a veteran in a special supporting solution. The extent of the contradict cation down through The CuHCF electrode's voltammetric response is mostly determined by its mobility. Figure-4 juxtaposes the CuHCF electrode's cyclic voltammetric responses by placing them inside the corresponding 0.1 M lithium, sodium, potassium, ammonium and barium nitrate solutions. From the figure, it's far clear that the decreased formal capacity at the side of better top currents for CuHCF will be performed in the presence of K^+ ions. The top currents are considerably terrible for the

redox method of (Fe^{2+}/ Fe^{3+}) in the presence of Na^+, Li^+, and NH^{4+} ions of CuHCF. These ions, Na^+, Li^+, and NH^{4+}, have a blocking effect on the CuHCF lattice channels. Ba^{2+} ion being superior in length makes it undeserving to enter the crystal lattice. Hence 0.1 M KNO_3 becomes selected because of the best electrolyte for the proposed sensor.

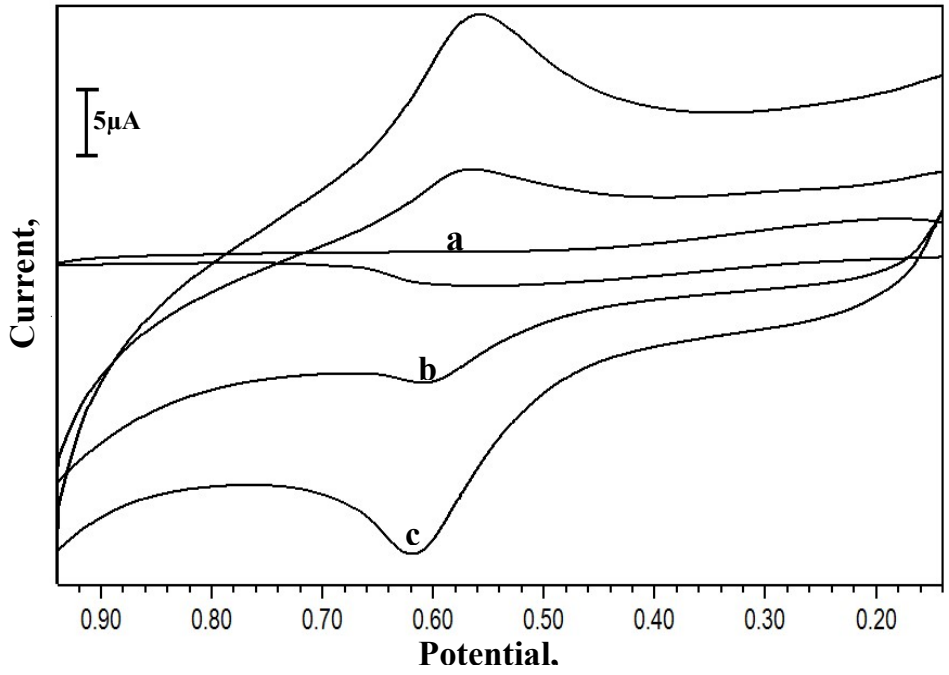


Fig.-3: (a)Cys-AuNPs-GW Voltammograms (b) CuHCF Film Absence of GNPs and (c) CuHCF film with GNP-Cys in 0.1 M KNO_3 @ 20 mV/s.

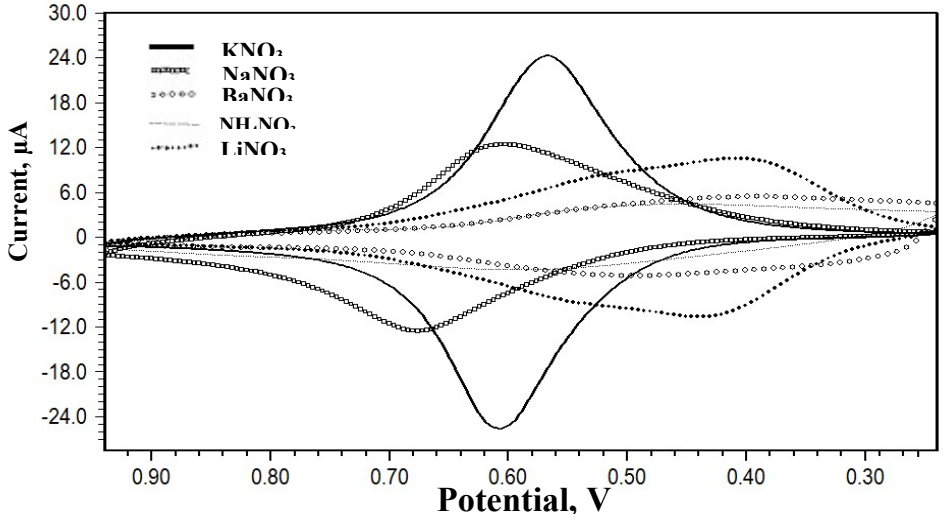


Fig.-4: At A Scan Rate of 20 mVs^{-1}, Cyclic Voltammograms of a Modified Electrode in the Presence of Nitrate Salts of Ba^{2+}, K^+, Li^+, Na^+, and NH^{4+} Cations

Scanning Rates and its Impact

The electrochemical deeds of the CuHCF film electrode in 100mM KNO3 were predicted at varied scan rates from 10 to 0.5 Vs-1 in a potential range of 200mV to 1000 mV (Fig. 5 A). As seen in (Fig.-5A), increasing the scan rates increased both the anodic and cathodic peak currents. Both the anodic and cathodic peak currents were proportional to the square root of scan rate for sweep tariffs ranging from 0.01 to 0.2 mVs-1, showing that the redox process is diffusion regulated at lower sweep tariffs (Fig.-5B).

The kinetics of electron transport at a CuHCF film modified electrode has also been studied. Starting with Laviron's conjecture, monitoring the variations of the height potentials as a characteristic of the test rate may be a promising way to discover the heterogeneous electron switch rate constant (ks). In Fig.-5C, the importance of height potentials, Ep, as opposed to the logarithm of capacity, is demonstrated. For test fees greater than 200 mVs^{-1}, the Ep values are found to be proportional to the logarithm of the test rate. The electron switch rate constant (ks) was calculated to be 0.89 s^{-1}, and the cost of change into calculated to be 0.68.

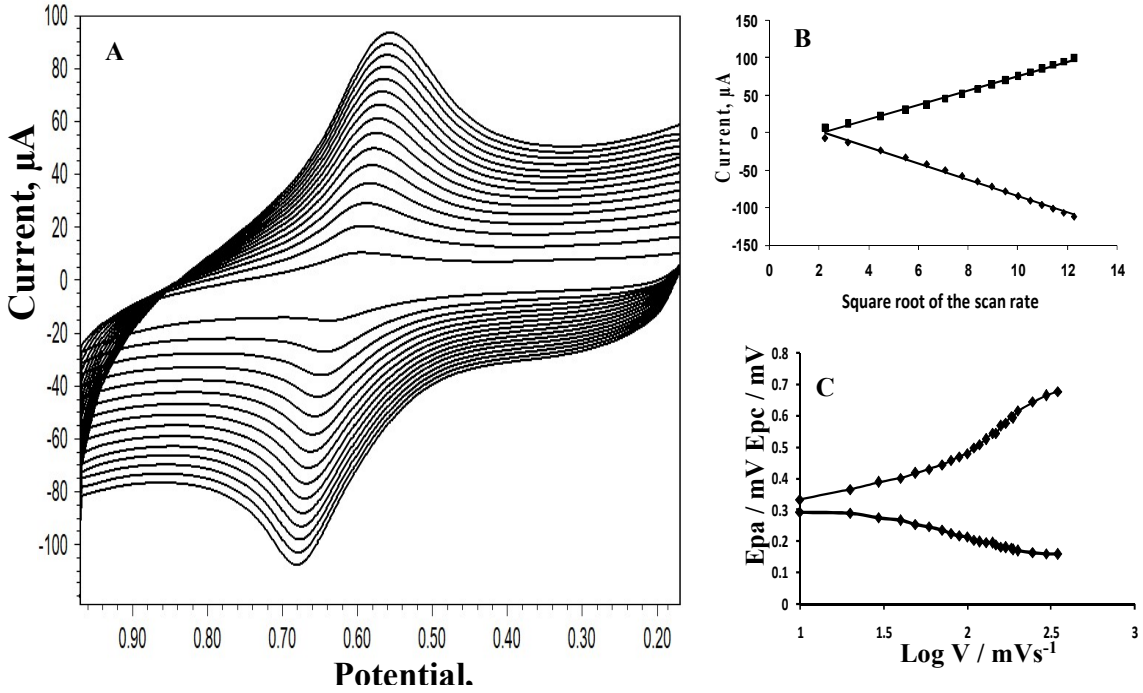


Fig.-5: (A) CV of at Several Scans, the CuHCF Film transformed the Electrode. Rates, from inside to outer are 10–140 mVs^{-1} with increments of 10 mVs^{-1} in 0.1 M KNO_3. (B) Dependence of Peak Current I_{pa} and I_{pc} on Square Root of Scan Rate (ν). (C) Variation of Peak Potential Vs. logarithm of Scan Rates (log ν).

Effect of pH on Performance of CuHCF Film Electrode

The performance of a CuHCF film modified electrode under various supporting electrolyte pH conditions was studied in the pH assortment of 2-9. It shows in Fig.-6, oxidation peak current was relatively privileged on pH 7.00 in the sort deliberate. Thus, in order to obtain the highest sensitivity, pH of the standard was maintain at seven for the subsequent experiments. A supplementary augment The crest current was reduced as the pH increased, which could be due to the hydroxylation of CuHCF in an alkaline media.

Stability of the CuHCF Film

CuHCF film exhibited incredible stability. The concert of the working electrode was examined by conducting continuous cyclic voltammograms in the potential assortment of 200mV to 1000mV for approximately two months and the retort pragmatic is revealed in Fig.-7. In cooperation, the current retort and the prescribed potential remained constant on repetitive scanning due to the covalent coupling of the mediator to the graphite surface, preventing the leaching of the CuHCF into the solution during experiments.

Electrocatalytic Oxidation and Amperometric Determination of BHA

CV was recorded in the presence and absence of BHA in 0.1 M KNO3 to assess the electrocatalytic effect of the CuHCF modified composite electrode closer to the oxidation of BHA (0.05 M phosphate buffer; pH 7). As shown in Fig.-8, when 2.1 x 10-4 M BHA was accumulated, the anodic peak current at the

modified electrode (Curve d) increased dramatically, but a minor increase in the anodic current was practical at the naked electrode (Curve b). Furthermore, the anodic potential favoring BHA oxidation at the CuHCF modified is 650 mV, whereas BHA oxidation occurs at 820 mV at the bare electrode. The decrease in overpotential and increase in anodic peak current for BHA oxidation show that the CuHCF modified electrode has a good electrocatalytic impact.

The Scheme-2 depicts the mechanism for electrocatalytic oxidation of BHA using a CuHCF film modified electrode.

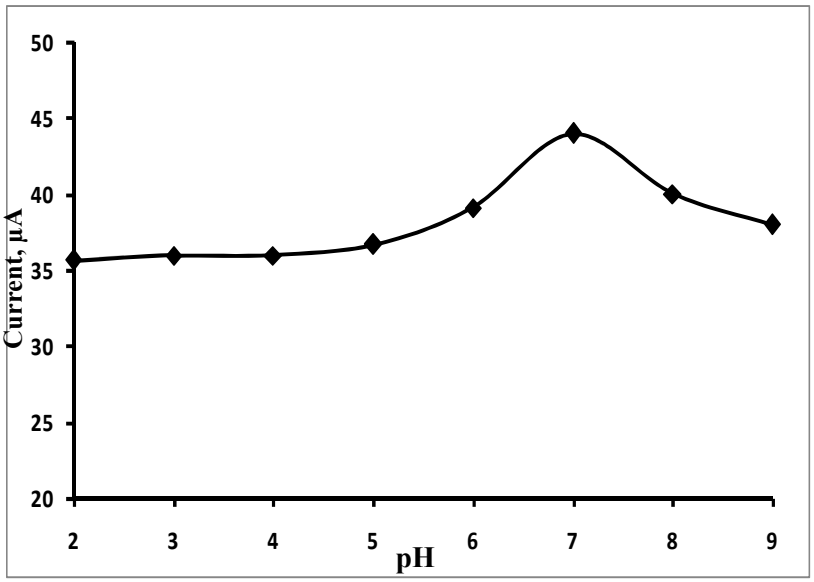


Fig.-6: The Effect of pH on the Peak Current Response of a CuHCF Film Modified The electrode in 0.1 M KNO3 at a Scan Rate of 20 mV/s.

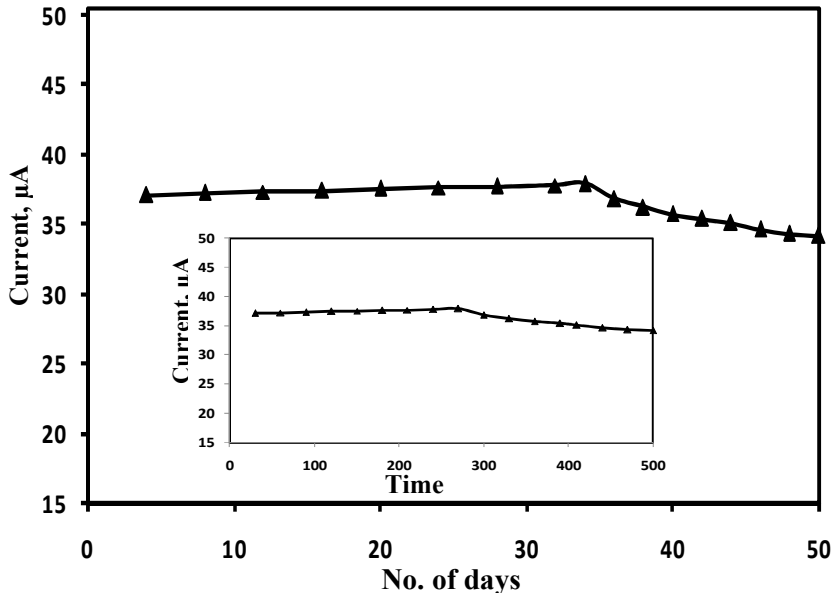


Fig.-7: The Peak Current of a CuHCF Film Modified Electrode Versus Time in Days. The Plot of Peak Current Versus Time in Minutes is Shown in the Inset

Hydrodynamic and Chronoamperometric Studies

To investigate the electrocatalytic response of a CuHCF modified composite electrode under dynamic conditions for the oxidation of BHA and to optimize the operating potential for amperometric capacity, hydrodynamic voltammetry (HDV) was used. In HDV, the working electrode's potential was gradually

increased in a stirred solution, and the resulting steady-state current was measured and plotted alongside the practical potential. Figure-9 shows the relationship between current and applied voltage in the presence of 3.6 x 10-4 M BHA at the unmodified composite (Curve a) and modified (Curve b) electrodes. With the stripped electrode, there is a negligible snap, however with the functioning electrode, there is a considerable increase in current. The current rises from 0.4V to 650 mV, where it reaches its highest significance. As a result, for electrocatalytic oxidation of BHA in flow systems, a voltage greater than or equal to 700 mV can be used.

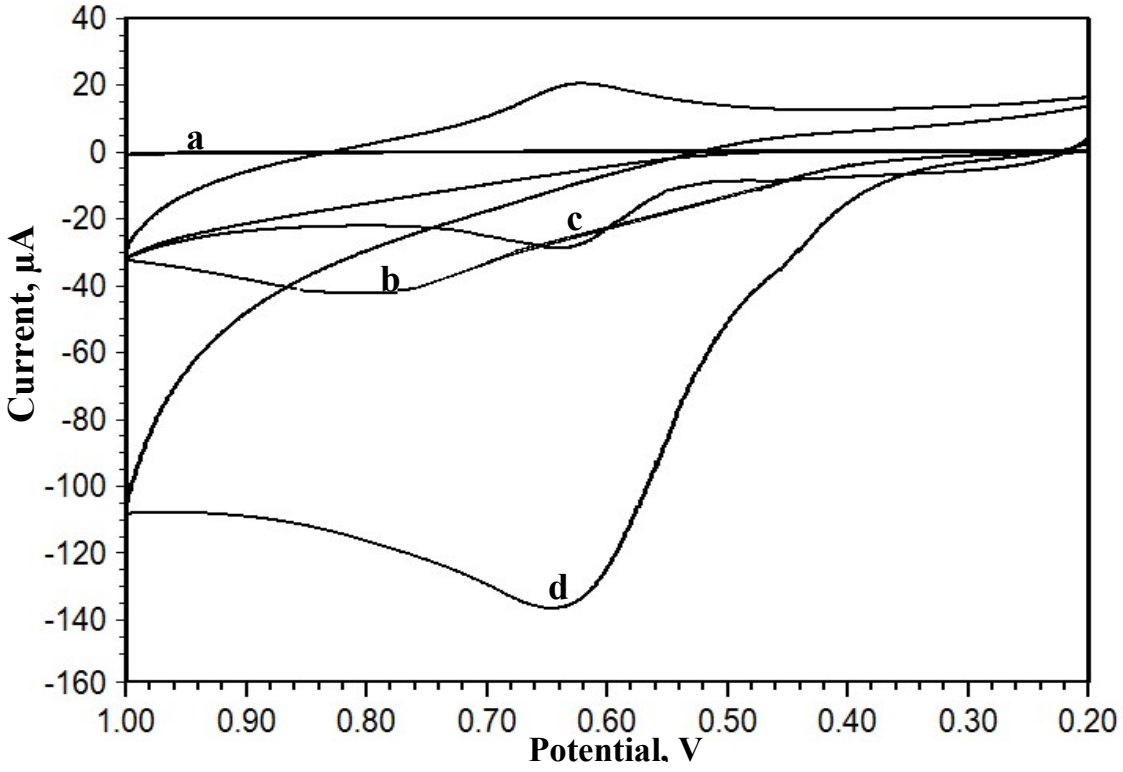
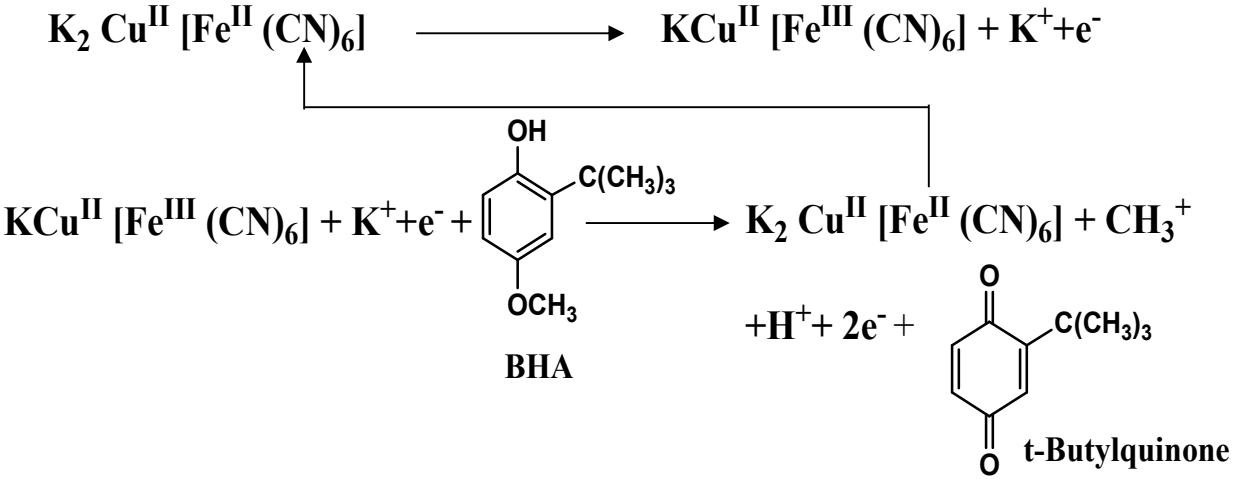


Fig.-8: Cyclic Voltammograms of (a, c) Bare Electrode and CuHCF Film Modified Electrode in the absence of BHA and (b, d) 1.1 x 10^{-4} M BHA in 0.1 M KNO_3 (pH 7) at 20 mV/s in the presence of BHA.



Scheme-2: Mechanism for Electrocatalytic Oxidation of BHA using CuHCF Film Modified Electrode

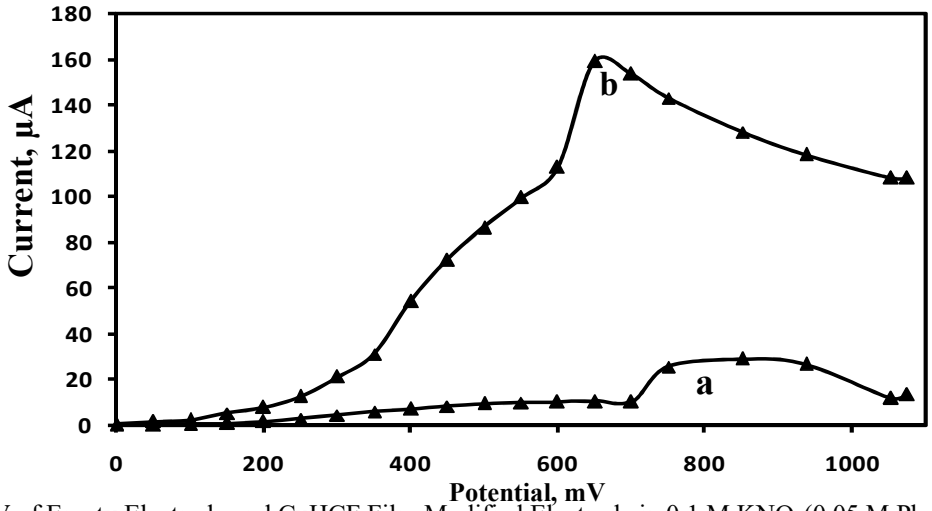


Fig.-9: HDV of Empty Electrode and CuHCF Film Modified Electrode in 0.1 M KNO_3(0.05 M Phosphate Buffer; pH 7) at 20 mV/s, agitated 300 rpm in the presence (a,b) of 1.7 x 10-4 M of BHA, respectively

For the electrocatalytic oxidation of BHA, the amperometric response of the CuHCF film modified electrode was predetermined. Figure-10 shows the current-time retort obtained for the CuHCF modified electrode in a stirred solution (300 rpm) for successive increases of 0.2 ml of 0.01 M BHA in 70ml of 0.1M KNO_3 (0.05 M phosphate buffer; pH 7) for successive increases of 0.2 ml of 0.01M BHA in 70ml of 0.1M KNO_3 (0.05 M phosphate buffer; pH 7) The current crisis is being exacerbated by the growing popularity of BHA. Figure-10 shows the relationship between catalytic current and BHA concentration.

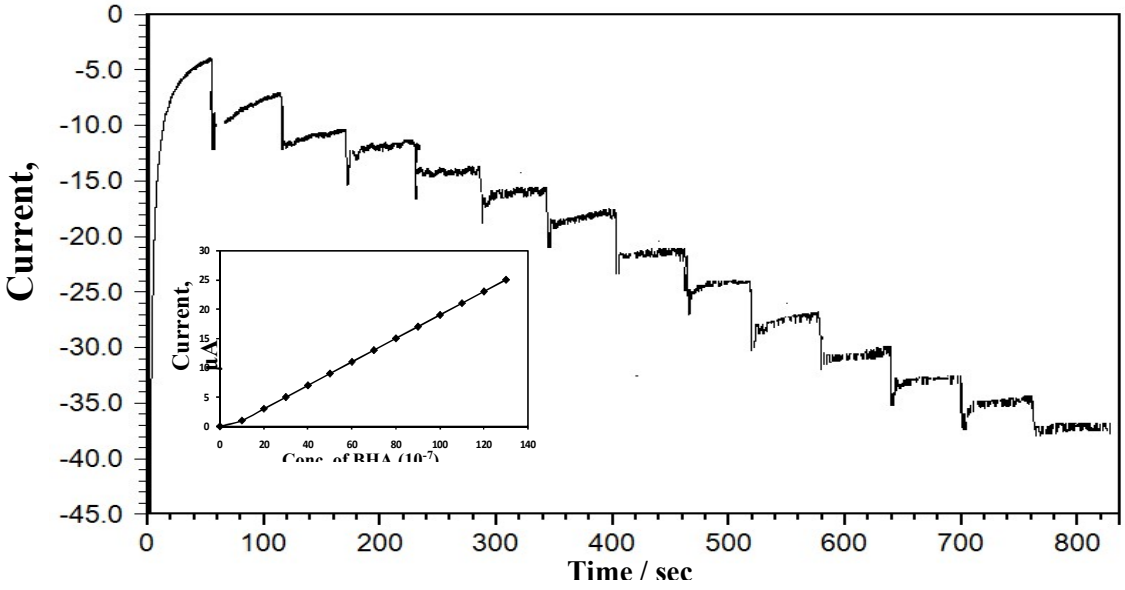


Fig.-10: Chronoamperometry Response of CuHCF Film Modified Electrode for 0.2 ml of 0.01 M BHA in 70ml of 0.1 M KNO_3 (0.05 M phosphate buffer; pH 7) at 20 mV/s; 300 rpm at 700 mV. The calibration plot is shown in the inset.

Analysis of BHA in Food Samples

To demonstrate the applicability of the proposed electrode, we used it to measure BHA fortitude in two distinct biscuit samples spiked manually through BHA, with the results shown in Table-1. The ideals achieved are typical of five replicate dimensions, and the statistical treatment is arithmetical, indicating that this procedure is precise and consistent.

Table-1: Real Sample Analysis of BHA

Samples	The Concentration of BHA (μM)		Recovery (%)
	Added	Found[a]	
Sample I	30	29.70± 0.70	99.0
	40	39.01± 0.49	97.5
	50	48.99± 0.60	97.9
Sample II	30	29.23 ± 0.50	97.4
	40	38.99 ± 0.48	97.5
	50	49.43 ± 0.58	98.9

[a]Our duplicate studies on average R.S.D

CONCLUSION

A copper hexacyanoferrate amperometric sensor is primarily based totally on the bespoke electrode. The electrode reveals an advanced electrochemical reaction in terms of solidity, reproducibility and sensitivity, revealing the deserves of low cost and easy grounding. The sensor can electro catalyze the oxidation of BHA. The changed electrode was dispensed to be an amperometric sensor for the willpower of BHA and used to decide the BHA in actual samples.

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