ECS Advances, 2023 **2** 026503

One Pot Synthesis of $CuO\text{-}CuFe_2O_4$@rGO Nanostructure with Synergistic Effect for Efficient Electrochemical Sensing Application of Paracetamol

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In the present study, the facile and synergistic approach for electrochemical sensing of paracetamol (PA) drug was demonstrated by hydrothermally synthesized copper oxide-copper ferrite nanohybrid composite supported on reduced graphene oxide ($CuO\text{-}CuFe_2O_4$@rGO) glassy carbon electrode. The surface texture and structural information of the electrode material were examined by FE-SEM, HR-TEM, and X-ray diffraction techniques, whereas the electrochemical sensing application of paracetamol oxidation was investigated by amperometric method. The average crystallite size of $CuO\text{-}CuFe_2O_4$ was calculated from XRD data and found to be 35.45 nm. The fabricated sensor exhibited a higher sensitivity of 970.26 μA.mM ^{1}.cm ^{2} along with a lower limit of detection (LOD) and limit of quantification (LOQ) of 7.0 μM and 25 μM, respectively, with a linear dynamic range of 10–1200 μM. Furthermore, the $CuO\text{-}CuFe_2O_4$@rGO modified sensor showed excellent anti-interferents ability, long-term stability and reproducibility towards electro-oxidation of paracetamol drug. Moreover, it can be efficiently applied for the analysis of paracetamol in biological samples. Finally, the synthesized nanocomposite material was validated to be a competent electrocatalyst for electrochemical sensing application of paracetamol.

 [DOI: 10.1149/2754-2734/acd800]

Manuscript submitted February 6, 2023; revised manuscript received May 9, 2023. Published June 1, 2023.

Supplementary material for this article is available online

Drugs like paracetamol (PA) are used as both antipyretics and painkillers. It is frequently used to treat a variety of ailments, including coughing, migraines, headaches, different types of bodily aches, and menstruation discomfort. Generally speaking, it is prescribed in many nations throughout the world as an aspirin substitute.[1–4] At lesser doses, paracetamol normally has no harmful side effects since it is quickly absorbed in the stomach and completely used by the body. However, a high dosage can have a number of negative consequences, including discomfort in the pancreas, kidney and liver problems, hepatic insufficiency, skin irritation, and skin rashes.[5,6] Thus, fast, sensitive and specific determination of paracetamol is essential for further investigation.

Different analytical techniques such as, chemiluminescence,[7,8] spectrophotometry,[9] capillary electrophoresis,[10] chromatography,[11] flow-injection analysis,[12] titrimetry,[13] electroanalytical[14,15] etc., have been employed for paracetamol analysis. However, different disadvantages including high cost, lengthy analysis and complex procedures render some techniques unfavourable.[16] Accordingly, the electroanalytical method is frequently used due to its simplicity, rapid response, cost effectiveness, high accuracy, selectivity and sensitivity.[17–19]

Unlike bulk materials, nanomaterials exhibit improved catalytic performance in various applications owing to their unique structural characteristics. Specifically, nanomaterials are widely used in applications including chemical sensors and bio-sensors.[20] In the last decade, a wide variety of nanocomposite materials were widely studied as catalytic materials for paracetamol oxidation reactions. These include polyimide-multi-walled carbon nanotube (polyimide-MWCNTs),[3] AuPd/GN-CNTs-IL (GN is Graphene nanotube, CNTs is Carbon Nanotubes, and IL is Ionic Liquid),[6] MWCNTs/CTS-Cu (CTS is chitosan),[21] GO/carbon black/chitosan (GO is Graphene Oxide),[22] GO-XDA-Mn_2O_3 (XDA is Xylenediamine),[23] GC/CNT/PEDOT/NF/Crown ether (where PEDOT is poly (3–4-Ethylenedioxy Thiophene).[24] Other hybrid materials such as mesoporous chitosan/silica hybrid material,[25] rGO/carbon black/chitosan,[26] chitosan - carbon paste,[27] and MWCNT/HSA-oppy were used as electrode materials.[28] Additionally, biomolecules and polymeric materials such as poly (L-leucine),[29] cellulose and starch polymers-Co NPs,[16] nano Molecularly Imprinted Polymers (MIP),[1] MIP/PB,[30] and MIP micelle were used.[31] Moreover, noble metal-based composites such as Pd/MnO_2 NRs/graphene,[32] Pd nanoclusters/polyfuran,[33] Pd graphene oxide,[34] Pd nanorods @hollow N, S-doped C were employed.[35] However, the high cost, complex synthetic procedure and instability of biomolecules and polymers limit their applications in paracetamol detection.

Hence, to overcome these shortcomings, different transition metal oxides were investigated, and revealed fascinating properties and merits for sensing purposes including low cost, high surface-volume ratio, non-toxicity, greater conductivity and semiconducting nature, sensitivity and rapid response.[36–39] Recently, many transition metal oxides like NiO,[4] CuO,[19] MnO_2,[32] TiO_2,[20,40] ZnO,[41] and Fe_2O_3[42] with different support materials have been extensively employed for electrochemical sensing of paracetamol. Among these, magnetite (Fe_2O_3) was widely used for different applications like sensors, catalysis, solar cells, and energy renovation owing to its non-toxicity, low cost, high stability, bio-compatibility, enzyme mimetic action (peroxidase enzyme) and *n*-type semiconducting nature with a band gap of 2.0–2.2 eV suitable for visible light absorption. Additionally, it

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