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List of Publications

Research Publications:

1. **Vineet Kumar Mall**, Ravi Prakash Ojha, Preeti Tiwari, Rajiv Prakash, “Immunosuppressive drug sensor based on MoS₂-polycarboxyindole modified electrodes”, *Results in Chemistry*, 4 (2022) 100345, DOI: 10.1016/j.rechem.2022.100345.
2. Monika Srivastava, Preeti Tiwari, **Vineet Kumar Mall**, Sanjay Kumar Srivastava, Rajiv Prakash, “Voltammetric determination of the antimalarial drug chloroquine using a glassy carbon electrode modified with reduced graphene oxide on WS₂ quantum dots”, *Microchimica Acta*, 186(7) (2019) 1, DOI: 10.1007/s00604-019-3525-3.
3. **Vineet Kumar Mall**, Ravi Prakash Ojha, Priya Singh, Rajiv Prakash, “Gold nano rods decorated MoS₂ nanosheets modified electrodes for the voltammetric estimation of anti malarial drug” (Communicated).
4. **Vineet Kumar Mall**, Preeti Tiwari, Madhu Tiwari, Rajiv Prakash, “Synthesis of Poly (indole-5-carboxylic acid) using Microwave Irradiation” (Communicated).
5. **Vineet Kumar Mall**, Preeti Tiwari, Rajiv Prakash, “2D material modified electrode towards detection of anti-cancerous drug” (Communicated).

Symposium and Conferences

1. The 4th International Conference on Advances in Materials and Materials Processing on 5th-7th Nov 2016 in IIT Kharagpur (Oral Presentation)
 2. Global Initiative for Academic Networks (GIAN) Workshop on Nanochemistry: From Preorganised Molecular Architectures to Functional Materials on 19th-23rd Dec 2016 in IIT (BHU) Varanasi (Attended)
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List of Publications

3. 20th CRSI National Symposium in Chemistry on 3rd-5th Feb 2017 in Gauhati University (Poster presentation)
 4. National Conference on Energy, Environment and its Impact on Society (NCEEIS-2017) on 19th-20th Jan 2017 in K.N.Govt. P.G. College, Gyanpur, Bhadohi (Poster presentation)
 5. Institute day of IIT (BHU) on 25th-26th Feb 2017 in IIT (BHU) Varanasi (Poster presentation)
 6. 45th National Seminar on Crystallography (NSC 45) on 9th-12 July 2017 in IIT (BHU) Varanasi (Volunteered)
 7. 4th International Conference on Nanoscience and Nanotechnology (ICONN 2017) on 9th-11th August 2017 in SRM University Chennai (Poster presentation)
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Voltammetric determination of the antimalarial drug chloroquine using a glassy carbon electrode modified with reduced graphene oxide on WS₂ quantum dots

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Abstract

A voltammetric method is described for the determination of chloroquine (CQ) and validated simultaneously by two techniques and in three different conditions. The WS₂ quantum dots (WS₂ QDs) were synthesized by a hydrothermal method and then placed on reduced graphene oxide (rGO) sheets. The resulting composite material was then deposited on a glassy carbon electrode (GCE) where it showed excellent electroactivity. The modified GCE responds to chloroquine at a typical potential maximum of 1.2 V (vs. AgCl/Ag). Techniques including cyclic voltammetry and differential pulse voltammetry were tested. Features of merit include (a) a wide linear response (in the 0.5 μM to 82 μM CQ concentration range), (b) an electrochemical sensitivity of 0.143–0.90 μA·μM⁻¹·cm⁻², and a 40–120 nM limit of detection (at S/N = 3). The sensor has excellent selectivity even in the presence of potentially interfering biological compounds. Responses were tested in phosphate buffer, human serum and pharmaceutical formulations, and no cross reactivity or matrix effects were found. In all the three cases, quite satisfactory recoveries were obtained.

Keywords Chloroquine phosphate · Anti-malarial drug · Reduced graphene oxide · WS₂ quantum dots · Synergistic effect

Introduction

Chloroquine (CQ) is extensively used in the treatment of sudden attacks of malaria and elimination of porphyria for many decades. This drug is widely used for curing rheumatoid arthritis and similar kinds of collagen diseases. Chloroquine is also used for amoebic hepatitis treatment [1–3]. Various strains of *Plasmodium falciparum* have developed resistance against CQ [4]. But due to its low cost this drug is still widely used in developing countries. It is

also recommended as a strong anti-cancerous agent [5, 6]. High doses of this drug may cause several side effects like heart attack [7]. There are various analytical methods reported in the literature to monitor the concentration of drug like spectrophotometric method [8], high performance liquid chromatography with fluorescence detection [9, 10], chemiluminescence including radiostorage- and photostorage-chemiluminescence (CL) [11] and voltammetric methods [12]. Among all methods voltammetric methods got immense attention due to its fast response, less volume consumption of samples, high selectivity and sensitivity.

Graphene is an atom-thick two dimensional crystalline carbon film with sp² hybrid carbon atoms. It reveals the unprecedented amalgamation of beneficial properties such as remarkable mechanical strength, large surface area, high thermal conductivity and extraordinary electrical properties like tremendous charge carrier mobility and conductivity [13, 14]. Due to these peculiar material properties, graphene have grabbed significant attention and proved their applicability in the fields of sensors [15, 16], catalysis [17], energy-storage [18], electronics [19] and optoelectronics [20]. The graphene oxide (GO) possesses various hydrophilic groups like hydroxyl carboxyl, and epoxy and

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Immunosuppressive drug sensor based on MoS₂-polycarboxyindole modified electrodes

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ABSTRACT

Azathioprine (Azp) is the principal immunosuppressive drug used in organ transplantation autoimmune diseases by improving therapeutic management and suppressing the immune system. Any discrepancy of Azp in human blood may lead to some serious side effects that obligate its quantitative estimation. In this work, the molybdenum disulfide (MoS₂) nanosheets modified with poly(5-carboxyindole) (CPIn) is reported for quantitative estimation of Azp. The synthesis of MoS₂ nanosheets is reported through single-step one-pot hydrothermal reaction and exfoliation of sheets is obtained in an aqueous dispersion of 5-carboxyindole (5CIn) through probe ultra-sonication. The nanosheets are further utilized as a template for the growth of the poly(5-carboxyindole) to obtain CPIn stabilized MoS₂ nanosheets (MoS₂-CPIn). The synthesis of MoS₂-CPIn nano hybrid is further validated via different characterization techniques including FTIR, XRD, SEM, TEM, and XPS. MoS₂-CPIn with magnificent electrocatalytic property is further exploited for the electroanalytical sensing of Azp via Differential Pulse Voltammetry (DPV) in phosphate buffer solution at biological pH (pH = 7.4) as well as human blood serum at room temperature. The proposed sensor shows a linear dependence of current with Azp concentration. The developed sensor demonstrates an extensively wide linear range of 3.49–284.44 μM possessing a low limit of detection of 74.65 nM (S/N = 3). The developed sensor shows excellent stability, sensitivity, and good selectivity in physiological pH. The achieved analytical parameters of the proposed sensor for easy quantification of Azp are found to be comparable or better than the previously reported Azp sensors and it is proved to be an excellent contestant for the trace level estimation of Azp in human blood serum.

1. Introduction

Azathioprine (Azp) is associated with a chemical category of purine analogues named 6-[(1-methyl-4-nitroimidazole-5-yl) thio] purine. Azp is an immunosuppressant and antileukemic drug that finds contemporary applications in various disorders such as ulcerative colitis, rheumatoid arthritis, Crohn's disease, and pemphigus [1,2]. Mercaptopurine is the active form of Azp which acts by decreasing the reactions of T and B cells by inhibiting their proliferation [3]. The continued exposure of Azp for a long time may lead to the development of specific types of cancer like skin cancer, blood cancer, and lymphoma and with some serious side effects including hair loss, nausea, fatigue, and rash. In order to counter these issues, it is crucial to evolve an accurate and sensitive method for the routine and careful monitoring of Azp in clinical operations [4,5]. In past, various techniques have been applied, for instance, high performance liquid chromatography (HPLC) [6,7], UV-Visible spectrophotometry [8,9], ¹H NMR spectroscopy [10],

chemiluminescence [11,12], ultra-performance liquid chromatography [13], high performance thin layer chromatography [14], flow injection analysis [11], surface-enhanced Raman spectroscopy [15], capillary zone electrophoresis [16], titrimetry [17] for the determination of Azp in human blood. Although all these techniques are accurate, some of them suffer from various demerits like low sensitivity and selectivity, expensive instrumentation, high cost, time-consuming operation, cumbersome extraction procedure with complex and tedious sample pretreatment that makes them inappropriate for regular analysis in many cases. Analytical and electrochemical measurement techniques play a critical role in biological recognitions, environmental monitoring, and drugs estimation in human body fluids as well as pharmaceutical formulations. The modern electrochemical methods have an important role in these fields because they have come up with sensational advantages like broad linear dynamic range, high sensitivity, and selectivity, simplicity, short analysis time, cost-effectiveness with real-time monitoring [18–20]. Cyclic Voltammetry (CV) [21] and Differential Pulse

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