Graphene Oxide Decorated Tin Sulphide Quantum Dots for Electrochemical Detection of Dopamine and Tyrosine

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Abstract
The current study highlights the design and construction of a sensitive and selective sensor for detection of dopamine and tyrosine using a GO-SnS2 quantum dots by a drop casting method on glassy carbon electrode. Highly porous nanocrystalline GO-SnS2 quantum dots were synthesized by using ultrasonication followed by hydrothermal method in a facile manner. XRD, SEM, XPS, TEM, and pore size distribution techniques were used to characterize the quantum dots that were produced. The newly fabricated electrode was evaluated for EIS (Electrochemical impedance spectroscopy), CV (cyclic voltammetry) and chronoamperometric methods. The observed limit of detection of dopamine was observed to be 26 nM. High selectivity and sensitivity were observed for electrochemical detection of dopamine and tyrosine.

Keywords GO-SnS2 quantum dots · Dopamine · Tyrosine · Graphene oxide · Electrochemical

1 Introduction

Dopamine and tyrosine are essential biomolecules which play a key role in human metabolism. Tyrosine is an important precursor of thyroid hormones, dopamine, adrenaline, and other hormones that are used to establish and maintain a proper balance in humans [1]. Hypothyroidism, hypochondria, and dementia are all caused by a lack of tyrosine. Dopamine belongs to the catecholamine family and is formed in the brain by dopaminergic neurons [2]. Dopamine is a key signal-transmission component between neurons because it is linked to the majority of important human body functions like motor control, reward, motivation, and cognition [3–6]. Low levels of dopamine and tyrosine in the blood, as well as the death of dopaminergic neurons in the brain, have been linked to a range of significant neurological illnesses, including Parkinson’s disease, psychosis, and attention deficit hyperactivity disorder (ADHD) drug addiction [6, 7]. To solve this problem, several studies have described novel approaches for detecting dopamine and tyrosine in a highly sensitive and selective manner, which might be utilized to identify dopamine and tyrosine-related neurological illnesses promptly [8–10].

Because of its short detection time and cost efficiency, the electrochemical sensing approach is recognized as one of the most effective approaches for dopamine and tyrosine detection among the different existing methods such as ELISA, colorimetric methods, Raman, and HPLC. [11–14]. Dopamine and tyrosine are redox-active chemicals that may be reduced or oxidized at different potentials, and their electrical characteristics can be used to detect their presence in a sample (usually human blood). The use of an electrochemical dopamine and tyrosine detection approach is challenging due to signal interference from other biological molecules.
(e.g., uric acid (UA), ascorbic acid (AA), and catecholamine molecules). Signal interference might greatly limit the sensitivity of dopamine detection since the reduction and oxidation potentials of these biological substances allegedly coincide with those of dopamine [15–17]. Furthermore, the electrochemical sensitivity of dopamine is still lower than that of other traditional techniques like HPLC and ELISA, which is a substantial hurdle to overcome before this approach can be utilized to detect accurate levels of dopamine [18]. By functionalizing electrode surfaces or introducing other types of conductive materials, several attempts have been made to overcome the issues of selectivity and sensitivity.

Graphene, a two-dimensional (2D) honeycomb structure made up of pure carbon molecules, has been widely exploited in different scientific fields, including batteries, display panels, solar cells, and even biological applications [19–22]. Furthermore, graphene derivatives have been shown to exhibit notable dopamine-detection properties [23], which are principally owing to π-π and electrostatic interactions between the graphene oxides’ surfaces. Various graphene-derivative-modified electrodes have been created to increase the performance of dopamine biosensors, including graphene/glassy carbon electrode (GCE), graphene–gold nanoparticles/GCE, TiO2–graphene/GCE, and GO/GCE electrodes [24–27]. One of the most intriguing carbonaceous compounds is graphene, a one-layer thick sheet with exceptional optical, thermal, and electrical characteristics. The discovery of porous graphene oxide (PGO), a type of graphene-oxide sheet with numerous hydroxyl groups and a porous surface [28], has the potential to improve the electrostatic interaction between the PGO and the analytes while also facilitating electron transfer between the molecules and the underlying electrode substrates [29].

Generally graphene oxide functionalized metals oxides or sulphides shows high affinity towards sensing of the bio molecules [30–32]. Richard et al. reported ZnO–ZnFe2O4/Fe3O4/carbon nanocomposite with ultrasensitive and selective electrode for detection of dopamine[33]. SnO2 nano wires were used for selective detection of riboflavin [31]. Apart from this many of the sensors were fabricated for real time monitoring [34].

Among the various binary compounds of tin chalcogenides, tin sulphides are well studied or explored owing to adaptable chemical nature and can be fabricated into hybrids, composites, non-toxic nature etc. hence they are widely used in energy storage devices, solar cells and optoelectronic devices. Despite this, the electrochemical procedure produces substantial capacity fading in tin sulphides due to the high-volume change. [14–16]. Because these matrices can greatly promote electron/ion transfer and effectively accommodate cycle-induced stress/strain of SnS, the electrochemical performance of tin sulphide has recently been improved by grafting nanosized tin sulphide into various types of carbon matrices (e.g., carbon spheres, amorphous carbon, macroporous carbon, carbon nanotubes, or graphene) [17–23]. Despite considerable gains in gravimetric capacity and cycle performance, the nanostructure of these composites, in combination with the low tap density of carbon matrix, can restrict volumetric capacity [24, 25]. Furthermore, the creation of these composites typically involves severe conditions or sophisticated synthesis, both of which are costly to industry. As a result, achieving a simple, scalable synthesis of tin sulphide-based graphene materials with superior volumetric storage remains a major challenge.

We used a facile hydrothermal method to create a novel graphene Oxide/ SnS2 (GO-SnS2) composite. SnS2 quantum dots are tightly supported on porous graphene oxide (PGO) in the composite, forming a primary microstructure and then assembling into a secondary nanostructure. The tap density of the nanostructured SnS2 and PGO hybrid is very high. The combination of SnS2 quantum dots and PGO nanosheets inside nanosized building blocks can not only improve electron/ion transport, but also efficiently insert SnS2 volume change and provide strong structural stability to the composite. As a result, the tightly compacted GO-SnS2 quantum dots show high, fast, and stable dopamine electrochemical detection. Thus, the prepared GO-SnS2 quantum dots were found to be exhibiting superior electrochemical performance, combined with its simple scalable synthesis, makes it a promising material for practical application.

2 Experimental section

2.1 Reagents and chemicals

Stannous dichloride (SnCl2.4H2O), Sodium Sulphide flakes (make: SD fine chemicals, INDIA), Dopamine (make: Aldrich), Tyrosine from Fischer Scientific Ltd., Potassium ferrocyanide and Potassium Chloride from SD Fine. Chemicals used in this work are of AR grade or analytical grade and used as received.

2.2 Synthesis of SnS2 nanoparticles using plant extract

The synthesis of SnS2 quantum dots was carried out as per our earlier work in slightly modified two step manner[35] Fresh Syzigium cumini (S. cumini) leaves (100 g) were collected and rinsed several times with distilled water to remove foreign particles before being ground using a mortar and
2. Fabrication of electrode

Before being utilised to form the working electrode, the glassy carbon electrode (GCE) was thoroughly washed with deionized water and polished with an alumina polishing pad. The electrode was sonicated for 15 min after being distributed in 1 mL DMF. To make a thin layer, the resultant mixture was drop casted over the surface of GCE with a micropipette and air dried overnight at room temperature. The modified electrode after casting was analysed for SEM analysis in order to check the uniformity of the material on the surface of the electrode (Fig. 4e). The SEM images clearly show the uniformity of the material that is coated.

2.1 Synthesis of Graphene oxide/SnS$_2$ nanocomposites (NCs)

Ultra-sonication followed by the hydrothermal method is simple and fast for synthesis of graphene oxide/ SnS$_2$ nanocomposite materials as reported in our earlier work [35]. In a beaker about 500 mg of graphene oxide in 100 mL water are homogenized by using an ultrasonic bath. The homogeneous graphene solution in the beaker were mixed with preformed SnS$_2$ synthesized, followed by hydrothermal treatment for 24 h at 100$^\circ$ C without adding any precipitating agent. The resultant colloidal solution was rinsed with ethanol and water, and then aged for roughly 12 h in beaker. Finally, the resultant combination solutions were dried in an oven at 65 $^\circ$C for 24 h, yield GO-SnS$_2$ quantum dots.

3 Material characterization

The produced GO-SnS$_2$ quantum dots were examined using a various of characterization methods. The size of GO-SnS$_2$ quantum dot phase purity and crystalline nature were examined using the X’pert Pro X-ray diffractometer with Ni filtered Cu Kα radiation ($\lambda=1.5406\AA$, 2θ=0–60). SEM (ZEISS EVO 18 model) was used to record the morphology of the GO-SnS$_2$ quantum dots. The GO-SnS$_2$ quantum dots were photographed and their selected area electron diffraction (SAED) patterns were obtained using an FEI TECHNAI G2 transmission electron microscope (TEM). A UV-1800 pc Shimadzu spectrophotometer was used to detect colloidal dispersions of GO-SnS$_2$ quantum dots in 200 to 1100 nm range. X-ray photoemission spectra were obtained on a KRATOS AXIS 165 with Mg Kα radiation (1253.6 eV) at 75 W. The C 1s line at 284.6 eV was utilized.
as an internal reference. Asymmetric gaussian forms were adopted in each situation. Binding energies of similar samples were typically constant within 0.1 eV.

4 Results and discussion

4.1 Material characterization

![Fig. 2 XPS spectra of the GO-SnS$_2$ quantum dots high resolution spectra of (a) Survey spectrum (b) Sn 3d$^{5/2}$ and Sn 3d$^{3/2}$ (c) S 2p spectra (d) C 1s spectra](image)

![Fig. 3 Pore sized distribution of SnS$_2$-GO quantum dots (a) Pore diameter (b) Surface area and (c) Pore volume](image)
4.1.1 XRD

The crystal structure of the as synthesized GO-SnS$_2$ quantum dots was given in Fig. 1.

The diffraction peaks at $2\theta = 26.0$ and also a hump around 25 shows the sp$^2$ graphene carbon. The peaks observed at $2\theta = 27.0$ (100), 34.09 (101), 42.58 (102), 51.8 (111), and 59.14 (200) and its corresponding planes is attributed to the hexagonal phase of SnS$_2$ [36]. The diffraction patterns agree well with the JCPDS card No. 23-0677. The obtained peaks are sharp with no impurities. The particle size calculated based on Scherrer’s equation at $2\theta = 26.0$, 34.09 and 42.58.
42.58 are estimated to be 5.27 nm, 9.19 nm and 7.74 nm respectively.

### 4.1.3 Pore size distribution

Figure 3 (a), (b) and (c) depicts the N$_2$ adsorption and desorption isotherms of GO-SnS$_2$ quantum dots. As observed from the Fig. 3c, the adsorption isotherms are similar to that of type (IV) isotherms with prominent hysteresis loop in the P/P$_0$ range of 0.5-1 reflecting the presence of mesopores. The synthesized GO-SnS$_2$ quantum dots showing the mesoporosity and is further confirmed by corresponding pore size distribution [38]. The SnS$_2$-GO quantum dots have a surface area of 13.865 m$^2$/g, an average pore size of 15.24 nm, and a measured pore volume of 0.066 cc/g. The findings are consistent with previous reported results [38].

### 4.1.4 SEM and TEM

Figure 4(a) and (b) shows the SEM and TEM images of synthesized GO-SnS$_2$ quantum dots. The SEM pictures disclose that the particles are agglomerated with sheet like
morphology of the graphene on which the SnS$_2$ particles are decorated and the sheets are attached to each other due to tiny dimensions and large surface energy. Each nano-cluster size ranges from, 31.40–57.83 nm, as indicated from SEM images. The SnS$_2$ particles were seem to embedded in the graphene sheets [39]. The EDAX patterns are given in Fig. 4(c) and the surface composition of C, O, Sn and S are in a specific area are 82.05, 15.78, 1.84 and 0.32 (atomic weight\%) respectively. 

The TEM images of GO-SnS$_2$ in Fig. 4b, demonstrates the quantum dots of size around 3 nm. The quantum dots are showing good crystallinity and the SAED pattern (Fig. 4d) infringes with hexagonal phase of tin sulphide and SnS$_2$ phases were decorated on graphene oxide, which is well in accordance with that of XRD.

4.2 Electrochemical performance of dopamine and tyrosine on the GO-SnS$_2$ nanocomposite modified electrode

Dopamine and tyrosine coexist in blood and many biological fluids and interfere with each other in the detection and moreover the concentration of tyrosine is generally low. High concentration of DA may interfere in determining the tyrosine. Hence, simultaneous determination of DA and Tyrosine is highly essential in electrochemical analytical research.

Three electrode voltammetry was carried out for electrochemical characterization and sensing. The primary event that reveals the existence of dopamine is the oxidation of dopamine on the surface of GCE. Dopamine molecules are linked to the surface of modified GCE electrode, releasing 2 H$^+$ ions to generate dopamine-o-quinone. These ions are then identified on the GCE, resulting in a conspicuous anodic peak on the voltammogram. Similarly, tyrosine molecules also attached to the electrode surface and releases 1 H$^+$ ion producing the ketone derivative of tyrosine and these ions are detected and analysed by CV studies. Scheme. 1 depicts a graphical illustration of this phenomenon.

4.2.1 EIS

Electrochemical impedance experiments in 0.1 M KCl at its formal potential in the frequency range 100 kHz to 100 mHz with a 10mV amplitude were performed to examine the electrical characteristics of the prepared electrodes. A typical EIS response of bare GCE and GO-SnS$_2$ quantum dots/GCE are shown in Fig. 5. At the bare GCE, a partial semicircle with a virtually straight tail indicates electron transport resistance to the redox probe. On the GO-SnS$_2$ quantum dot/GCE, the semicircle does not appear, suggesting a lower barrier to electron transmission. This is due to the high conductivity of the graphene oxide-SnS$_2$ formed on the surface. As indicated by the enhanced electrode’s impedance behaviour, GO-SnS$_2$ has been effectively adsorbed on the GCE surface. The modified electrode’s resistance is lower than the bare graphite electrodes, which could be due to improved conductivity of the modified electrode [40, 41]. The impedance charts match the behaviour of the CV.
### 4.2.2 Simultaneous detection of dopamine and tyrosine on GO-SnS₂ modified electrode

Simultaneous detection studies of dopamine and tyrosine were given in Fig. 6. There are no peaks observed in bare glassy carbon electrode as seen in Fig. 6(a) and moreover in case of GO-SnS₂/GCE with analyte only tyrosine is showing Ip (current) 0.0187 mA at Epa (voltage) 0.782 V which is given in Fig. 6(f). And in the third case simultaneous detection of tyrosine and dopamine was carried out using 100 µM Dopamine and 500µM of tyrosine in PBS buffer solution at pH 7 and is given in Fig. 6(e). The oxidation peaks of dopamine and tyrosine are very well separated and peaks appeared at 0.202 V with current 0.0257962 mA, and the peak at 0.7952 V with current 0.02579 mA correspond to dopamine and tyrosine respectively. Thus, the CV studies clearly showing the modified GO-SnS₂/GCE was successful in separating and distinguishing the analytes dopamine and tyrosine.

With precise redox behaviour of dopamine, GO-SnS₂/GCE demonstrated a three-fold increase in anodic peak current of 0.0362 mA. (Fig. 6d). These GO-SnS₂ dots, which boosted conductivity and surface area, are responsible for the better electrochemical current responsiveness.

To prove the surface area of GCE increases with modification with GO-SnS₂, the electroactive area of bare GCE, GO-SnS₂/GCE were determined and compared using CV technique as per the Randles-Sevik equation [42].

\[
i_p = \left(2.69 \times 10^5 \right)^{3/2} D^{1/2} v^{1/2} A_c.
\]

\[i_p = nFQυ/4RT \]

Fig. 6 Simultaneous detection studies of Dopamine and tyrosine on GO-SnS₂/GCE: a) bare GCE without any analyte, b) bare GCE with dopamine, c) GCE coated with GO-SnS₂ without analyte, d) GCE coated with GO-SnS₂ with dopamine, e) GO-SnS₂/GCE with dopamine 100µM + tyrosine 500 µM, f) GO-SnS₂/GCE with 500 µM tyrosine at scan rate of 50mV/s

The electrochemical areas calculated by using the equation are 0.112 cm² and 0.226 cm² for base GCE and GO-SnS₂/GCE respectively. As seen from the values the electroactive surface area increases nearly by 50% compared to that of bare GCE.

### 4.2.3 Effect of scan rate on peak current of dopamine and tyrosine

Using cyclic voltammetry, the impact of changing the sweep rate for 100 M dopamine in 0.1 M PBS at pH 7 was examined (Fig. 7A). Different scan speeds ranging from 50 to 400 mV/s were used to record the CV profiles. Peak current rose with a minor positive shift in peak potential in the region of 50 to 400 mV/s, as seen in the graph. Ip vs. potential and Ip vs. square root of scan rate demonstrate a linear relationship with zero intercept as seen in the figure inset. The regression equation is expressed as Ip = 2.9852 x + 0.03638 \((R^2 = 0.99418)\). The Ip increased linearly with scan rate and the corresponding regression equation is obtained as Ip = 0.082 x + 0.01431 \((R^2 = 0.99172)\). All these results confirm the diffusion-controlled process controlling the overall kinetics.

The number of electrons ‘n’ was estimated using Laviron’s equation, which is expressed as below [43].

\[\text{Ip} = nFQυ/4RT\]

In the above equation, Ip represents the anodic peak current (A), Q represents the charge associated with oxidation (C), υ is the scan rate (V s⁻¹), R represents the gas constant.
reduction peak was observed at – 0.445 mV potential. Apart from the oxidation and reduction peaks of dopamine, the third peak due to leucodopaminechrome observed due to ring closure of dopamine-o-quinone [44, 45].

Effect of varying concentration of tyrosine at modified GO-GO-SnS$_2$/GCE is shown in Fig. 8(B) and a similar trend as seen in dopamine were observed. As prominent oxidation peak at 500µM is seen in tyrosine studies, this concentration was chosen for all the comparative studies.

4.2.5 Effect of pH on dopamine and tyrosine studies

CV tests were performed to assess the influence of pH on oxidation of dopamine at PBS solutions with varying pH ranging from 4 to 12 at a scan rate of 50 mV/s to enhance the electrochemical responsiveness of the GO-SnS$_2$/GCE towards the electrochemical oxidation of DA (Fig. 9 A). The peak potential shifts to the negative side when pH rises from 4 to 11, owing to enhanced reversibility of the oxidation which involves deprotonation at elevated pH ranges (Fig. 9 A). Furthermore, pH = 7 PBS had a superior electrochemical response in sensor applications. As a result, pH = 7 PBS was discovered to be optimal electrolyte for electrochemical research.

Effect of pH on tyrosine oxidation in PBS solutions with varying pH from 4 to 12 at a scan rate of 50 mV/s was given
Fig. 9 Cyclic voltagrams of (A) 100µM dopamine and (B) 500µM tyrosine with varying pH using 0.1 M PBS, scan rate 50mV: (a-i); a)4, b)5, c)6, d)7, e)8, f)9, g)10, h)11, i)12

Fig. 10 Amperometry response of GO-SnS$_2$/GCE for each addition of (a) 50 µM DA (b) interferents (AA and UA) at constant applied potential of +0.25 V in PBS containing 0.1 M KCl (pH 7.0)
of GO-SnS$_2$/GCE electrode. In the concentration range of $2.5 \times 10^{-6}$ to $250 \times 10^{-6}$ M, a linear connection between peak current and DA concentration was observed, with the lowest detection limit being 26 nM. In order to understand improvement of the modified sensor the calculated limit of detection and sensitivity were compared with earlier reported dopamine sensors and is given in Table 1.

### 4.2.7 Interference studies

The interference studies on GO-SnS$_2$/GCE was carried out by taking the common interfering biomolecules like 50 µM uric acid (UA), 50 µM ascorbic acid (AA) in phosphate buffer at pH 7 by using the chronoamperometry and the results are given in Fig. 10(b). The synthesized sensor system shows insignificant current intensity changes with respect to dopamine as demonstrated in Fig. 11.

### 4.2.8 Stability and repeatability of GO-SnS$_2$/GCE electrode

The electrode’s stability was tested by immersing it for three weeks in a phosphate buffer solution with a pH of 7.0. Every week, CVs were collected and compared to the ones received on the first day. The oxidation peak current was found to be somewhat lower than anticipated. The current reduction was just 10% after three weeks as demonstrated in Fig. 11(a) indicating that the modified electrode is highly stable.

The modified electrode’s repeatability was tested ten times with 100 µM DA. After each measurement, the modified electrode was rinsed with buffer solution and evaluated in Fig. 9(B). With increase in concentration there is increase in anodic peak current but the relative response was low when compared to dopamine. There is shift in negative peak potentials due to increase in reversibility of the oxidation at elevated pH of 7 and concentration of 500 µM was found to be optimum for tyrosine studies.

### Table 1

Comparison of analytical performance at GO-SnS$_2$/GCE with modified electrode with previously reported dopamine sensors

| S. No. | Modified Electrode | LOD(µM) | Linear range(µM) | Ref |
|--------|--------------------|---------|------------------|-----|
| 1      | Indium Tin oxide   | 0.001   | -                | [46]|
| 2      | GC/G-SnO$_2$       | 2       | 0–100            | [47]|
| 3      | Bi-Sn NP/CGA/SPCE  | 0.0046  | 0.02 – 97.59     | [48]|
| 4      | Pd NPs/GC         | 8       | 8.0–88           | [49]|
| 5      | CTAB/rGO/Zn       | 0.5     | 1.0–500          | [50]|
| 6      | GCE/CQDs/CuO      | 2.4     | 1–180            | [51]|
| 7      | N-Graphediyne quantum dots (GDQDs) | 0.14 | 0.32–500 | [52]|
| 8      | GO-SnS$_2$        | 0.029   | 2.5–250          | Current work |

Fig. 11 (a) Repeatability and (b) Stability studies of GO-SnS$_2$/GCE in Phosphate buffer at pH 7 containing 50 µM dopamine concentration @50 mV/s scan rate
5 Conclusions

Finally, using a facile ultrasonication and hydrothermal method, the quantum dots of SnS$_2$-carbon composites were synthesized. The as synthesized materials were characterized by using various techniques like XRD, SEM, TEM, EDX and elemental mapping, XPS, and pore size distribution. These dots were used to construct a modified glassy carbon electrode for dopamine and tyrosine detection. For EIS, CV and chronoamperometric studies, the electrocatalytic activity of modified electrodes is investigated. Intriguingly, chronoamperometric studies discloses a LOD of 26 nM for dopamine detection. Compared to dopamine the relative response of tyrosine is less. The modified electrode has excellent stability, selectivity, sensitivity, and reproducibility, according to our research.

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