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Appendix-I

(A1) Cross linking mechanism of glutaraldehyde in PPy-MoS_2 system

The monomer unit of polypyrrole has a five-membered ring containing four carbon atoms and one nitrogen atom. It has conjugated double bond systems making it suitable for effective polymerization. On the other hand, glutaraldehyde (GA) consists of two reactive aldehydic groups, and widely used as a crosslinking agent for covalent coupling. The formation of covalent linkage in between the secondary amine (NH) group of the monomer unit with the GA molecule has been depicted below in the schematic chemical reaction. GA shows remarkable reactivity towards the amine groups, and it gets attached to the polypyrrole chain by removing its one oxygen atom from the aldehyde (-CH=O) part followed by binding with the nitrogen atom present in the monomer unit. The formation of C=C occurs due to the dislocation of the π-bond corresponding to C=O of the aldehydic group (as can be seen in the Figure A2 (a) below). To realize the interaction of GA with the polymer-TMDC based composite system, we have taken the FT-IR response of the PPy-MoS_2 after treating it with 4% glutaraldehyde for 1 h, and compared with the pristine PPy-MoS_2 system.

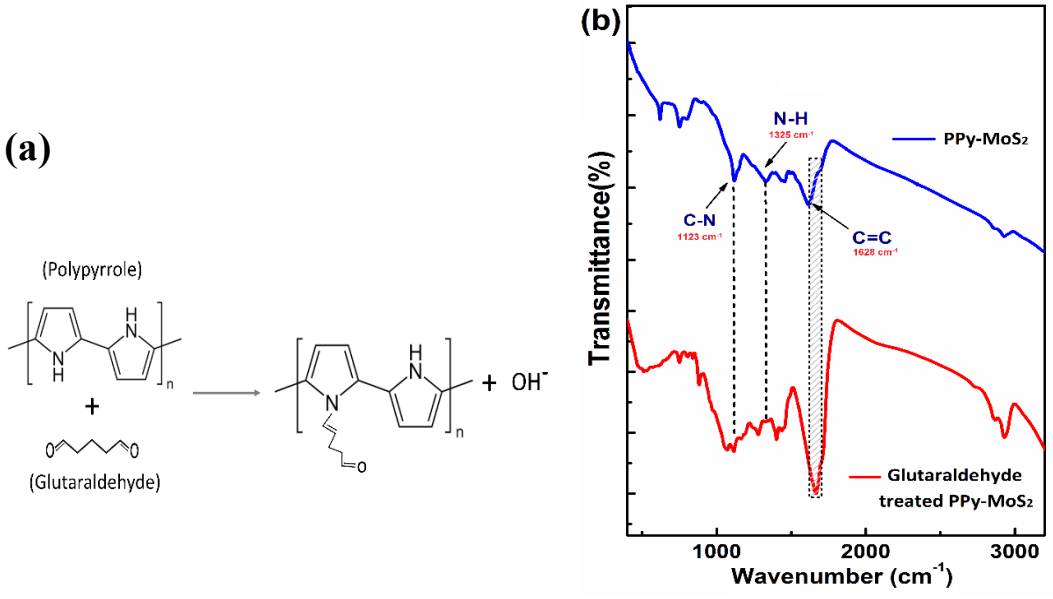

Figure A2: (a) Reaction mechanism of polypyrrole and GA, (b) FT-IR response of the pristine and GA treated PPy-MoS_2 electrode system

Here, we can see that the vibrational bands of the GA treated PPy-MoS_2 system has shifted little bit towards the lower wavenumber region. This decrease in the bond vibration energy indicates the generation of strain in the polymer chain due to the incidence of covalently attached GA molecules in polypyrrole system. In the FT-IR spectra of Figure A2 (b), one can see that intensity of N-H vibration mode (~1325 cm^{-1}) decreases as well as intensity of C-N mode (~1123 cm^{-1}) increases in case of the GA treated system, which is due to the replacement of N-H by C-N bond. Again, the sharp rise of the C=C vibrational mode (~1628 cm^{-1}) also indicates the appearance of additional C=C in the polymer structure after the covalent attachment of the GA. This suggests that the GA molecules interact with the surface of the polypyrrole-MoS_2 electrode system by forming a covalent linkage with the polypyrrole backbone.

Appendix-II

(A2) (a) Comparison of figure of merits of different immunosensors and detection methods

Table A1 compares the figure of merits for detection of immunoglobulin G molecules using different methods. Though the surface plasmon resonance (SPR) based detection methods are popular for their accuracy, the *LOD* value obtained from this method are comparatively large [1-3]. So, low concentration detection can't be effectively carried out by SPR based techniques. But electrochemical methods can overcome this drawback, offering very low *LOD* value, as can be seen in the table below [4-8]. Depending upon the detection protocols and types of transducer materials, the sensing parameters (*LOD*, sensitivity, detection range) of different electrochemical techniques varies accordingly. If we compare our works with the other groups, then we can see that the obtained values of *LOD* is relatively higher than few of the them, but the sensitivity is fairly high in our ion beam modified system indicating high accuracy. Moreover, most of the sensing protocol with low *LOD* have very narrow detection range (Table A1) which is the major drawback of such systems. While, a wide detection range of (9-363) ng/mL in case of AuNP/GO/ PEDOT-PSS based system stipulates better applicability of our immunosensor.

Table A1: Comparison of figure of merits of different immunosensor and detection methods.

System & detection method	Target	*LOD*	Sensitivity	Detection range	Ref.
Au-nano-shell, LRSPR	Human *IgG*	0.20 µg/mL	1.84 mM mL μg^{-1}	(1- 40) µg/mL	[1]
$MoSe_2$-Au based substrate, SPR	Goat-Anti-Rabbit IgG	0.33 µg/mL	--	--	[2]
Polydimethylsiloxane substrate, SPR	Human *IgG*	15 µg/mL	--	(15–225) µg/mL	[3]
Cu-MOF, DPV	Human *IgG*	3 pg/mL	--	(0.01-10) ng/mL	[4]

Ferrocenyl dendrimer/GCE, Amperometry	Goat *IgG*	2.0 ng/mL	0.020 $\mu A.mL.ng^{-1}$	(5-50) ng/mL	[5]
Modified Graphene, EIS	Rabit *IgG*	--	--	(0.3-7.0) µg/mL	[6]
Poly indol-6-carboxylic acid/GCE, SWV	Human *IgG*	0.8 ng/mL	--	(2-16) ng/mL	[7]
Core-shell SiO_2/Au (Using GOx as enhancer), CV	Human *IgG*	120 µg/mL	--	0.75 mg/mL to 0.14 gm/mL	[8]
AuNP/GO/ PEDOT-PSS, Impedimetric technique	Mouse *IgG*	49.2 nm/mL	--	9-363 ng/mL	Ch. 2 Our work
AuNP/PEDOT-MoS_2, Amperometry	Mouse *IgG*	12.22 ng/mL	1.845 µA ng^{-1} mL cm^{-2}	7.7–263 ng/mL	Ch. 3 Our work
Ion beam modified PPy-MoS_2, DPV	Mouse *IgG*	30.0 ng/mL	10.0 $\mu A.mL.ng^{-1}$	(5-190) ng/mL	Ch. 6 Our work

(A2) (b) Interpretation and comparison of non-enzymatic glucose sensor

We obtained electrochemical parameters for different investigated electrode specimens, as can be found in Table A2. In Chapter 4, a better electroactivity of CuO/PEDOT-MoS_2 system towards glucose sensing has been witnessed. A fairly low *LOD* value of 0.046 µM and a high sensitivity of 829 µA $mM^{-1}cm^{-2}$ would indicate a better redox activity in the conversion of glucose into gluconic acid. In the past, Kim *et. al*. designed a potentiometric glucose sensor by immobilizing Au-NPs over benzoic acid-functionalized poly-terthiophene (*p*-TBA) and could achieve a detection limit of 0.19 µM in the linear range of 0.32 μM^{-1} mM [10]. Other groups *viz.* Liu *et. al.* [12] and Wang *et. al.* [14] also designed CuNPs/ poly (*o* -phenylenediamine) and Ni/PANi based sensor electrode respectively and have achieved low *LOD* values and higher sensitivity over a broad linear range of glucose concentrations. However, the NiO-MoS_2 NS based electrocatalyst system in Chapter 5 exhibited a quite high sensitivity value for detection of glucose. This resembles high signal amplification of our layered NiO based modified transducer. A comparative view of sensing parameters, selectivity, *LOD*, etc. of other non-enzymatic sensors and this work can be found in Table A2.

Table A2: Comparison of reported non-enzymatic glucose sensors.

Substrate	*LOD* (µM)	Linearity	Sensitivity ($\mu A\ mM^{-1}cm^{-2}$)	Group
Ni Fe (NPs)-PANi	0.5	10µM-1.0 mM	1050	[9]
pTAB/AuNP/SPCS	0.19	0.32µM-1mM	----	[10]
$NiP_{0.1}$-SnO_x/PANi/ CuO/Cotton	0.13	1µM-10mM	1325	[11]
CuNPs/PoPD/GCE	0.25	5µM-1.6mM	----	[12]
Ni-Co-S NS/PPy NW core/shell structure	0.82	2 µM-0.14 mM	----	[13]
Ni/PANI coaxial nanowire	10	Upto 7mM	76.8	[14]
CuO/PEDOT-MoS_2/ITO	0.043	30µM-1.06 mM	830	Ch. 4 Our work
NiO-MoS_2	3.53	5-370 µM	1880	Ch. 5 Our work

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LIST OF PUBLICATIONS

Articles:

1) **A. Medhi**, S. Baruah, J. Singh, C.A. Betty, D. Mohanta, Au nanoparticle modified GO/PEDOT-PSS based immunosensor probes for sensitive and selective detection of serum Immunoglobulin G (IgG), Applied Surface Science. 575 (2022) 151775. (DOI: 10.1016/j.apsusc.2021.151775)
2) **A. Medhi**, D. Mohanta, Deciphering highly sensitive non-enzymatic glucose sensing mechanism based on CuO/PEDOT-MoS_2 electrodes measured in Chronoamperometry, ECS Advances, 1 (2022) 046504. 262-267. (DOI: 10.1149/2754-2734/ac9324)
3) **A. Medhi**, D. Mohanta, Development of highly sensitive electrochemical immunosensor using PPy-MoS_2 based nanocomposites modified with 90 MeV C^{6+} ion beams, Microchimica Acta, 191 (2024) 166. (DOI: https://doi.org/10.1007/s00604-024-06210-w)
4) D. Sarma, **A. Medhi,** D. Mohanta & P. Nath, Electrochemically deposited bimetallic SERS substrate for trace sensing of antibiotics, Microchimica Acta, 191(1) (2024) 14. (DOI: https://doi.org/10.1007/s00604-023-06075-5)
5) **A. Medhi**, M. K. Giri, D. Mohanta, A non-enzymatic approach of H_2O_2 and glucose sensing using NiO-MoS_2 derived electrochemical sensor. (In Press)
6) **A. Medhi**, D. Mohanta, Precise detection of *IgG* molecules using AuNP anchored, PEDOT-MoS_2 based electrochemical sensor. (Under review) (Preprint DOI: 10.21203/rs.3.rs-2865989/v1)

Conference Proceeding:

1) **A. Medhi**, M. K. Giri, D. Mohanta, Non-enzymatic electrochemical detection of H_2O_2 using $Ni(OH)_2$ nanoparticles, Materials Today: Proceedings, 68 (2022) 262-267. (DOI: 10.1016/j.matpr.2022.09.497)

Book Chapter:

1) **A. Medhi**, D. Mohanta, Non-enzymatic detection of glucose by electrochemically synthesized CuO nanoparticles (ISBN: 978-93-91953-55-3)

Papers presented in conferences

1) **Poster presentation in National Conference on Hard and Soft Condensed Matter Physics (NCHSCMP-2019),** Title: Development of Metal-Organic Framework (MOF) as visible light driven photocatalyst for Hydrogen production by water splitting, Authors: A. Medhi, A. Kumar.
2) **Oral presentation in CONDENSED MATTER DAYS 2020 (CMDAYS20),** Title: Conducting polymer based electrochemical sensor for efficient detection of Serum Immunoglobulin G (IgG), Authors: A. Medhi, D. Mohanta.
3) **Oral presentation in Advances in Physics and its Applications (APA-2021) conference,** Title: Non-enzymatic detection of glucose by electrochemically synthesized CuO nanoparticles, Authors: A. Medhi, D. Mohanta.
4) **Poster presentation in International Conference on Emerging trend in nanomaterials Science and technology (ICETNMST-2022),** Title: Non-enzymatic electrochemical detection of H_2O_2 using NiO nanoparticles, Authors: A. Medhi, M.K. Giri, D. Mohanta.
5) **Oral presentation in XIII Biennial National Conference of Physics Academy of North East (Pane), 2022,** Title: A non-enzymatic approach of H_2O_2 and glucose sensing using NiO-MoS_2 derived electrochemical sensor, Authors: A. Medhi, M.K. Giri, D. Mohanta.
6) **Oral presentation in National Conference on Emerging Trends in Material Science, 2022,** Title: Highly sensitive non-enzymatic glucose sensor based on CuO nanoparticles decorated over PEDOT-MoS_2 matrix, Authors: A. Medhi, D. Mohanta.

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Abstract

Immunoglobulin (*Ig*) molecules play most important role in body's immune system, serving as a defense line against the harmful pathogens. Among the other classes of *Ig*s, *Ig*G is the most abundant one found in the blood serum, making it a potential biomarker for several oncological and inflammatory diseases. This leads to an utmost importance of examining the *Ig*G level in blood for diagnosis purpose. Apart from this, monitoring the glucose and H_2O_2 level in body fluids can also provide valuable insight regarding blood sugar regulation, whole body oxidative stress and metabolism system. Out of other conventional methods, electrochemical detection technique has been extensively used due to its simplicity, high accuracy and point-of-care detection strategies. The detection mechanism of *Ig*G includes the proper immobilization of the antibody molecules over the electroactive specimen called transducer, followed by quantification of the target *Ig*G molecules *via* electrochemical methods. On the other hand, the redox activity of some metal oxide nanoparticles towards glucose and hydrogen peroxide (H_2O_2) have opened up a convenient approach for non-enzymatic detection of such simple analytes. To design a high-performance electrochemical sensor, meticulous selection and designing of the transducer material is very crucial. In this regard, conducting polymers (*eg*. polyaniline, polypyrrole, PEODT etc.) are widely used as an electroactive material that can offer high surface area, good conductivity and better stability. It has been reported that composite of 2D layered nanostructures (graphene and its derivative, TMDCs, MXenes, g-C_3N_4, hBN etc.) with conducting polymer holds high possibility for synergic tuning of several physico-chemical properties of the composite system, thereby expanding their range of functionalities for sensing applications. Apart from this, swift heavy ion (SHI) irradiation-based material modification can effectively modify the structural, morphological, electrical, optical and other spectroscopic behaviour of the target material. In polymer system, the SHI can introduce cross-linking, chain scissioning depending upon the energy, type and fluence of the ion beam used. So, it is anticipated that the electrochemical performance of the polymer-based nanosystem can be significantly improved through ion beam modifications.

In the present thesis, fabrication of both enzymatic and non-enzymatic electrochemical sensors has been carried out for sensing of biological analytes. In the first and second work, enzymatic detection of goat anti-mouse *Ig*G was performed by using conducting polymer (CP) and 2D layered material based composite substrate decorated by electrodeposited gold nanoparticles

DEVELOPMENT OF CONDUCTING POLYMER, 2D LAYERED MATERIAL NANOCOMPOSITE BASED ENZYMATIC AND NON-ENZYMATIC ELECTROCHEMICAL SENSORS, AND ION IRRADIATION EFFECTS

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