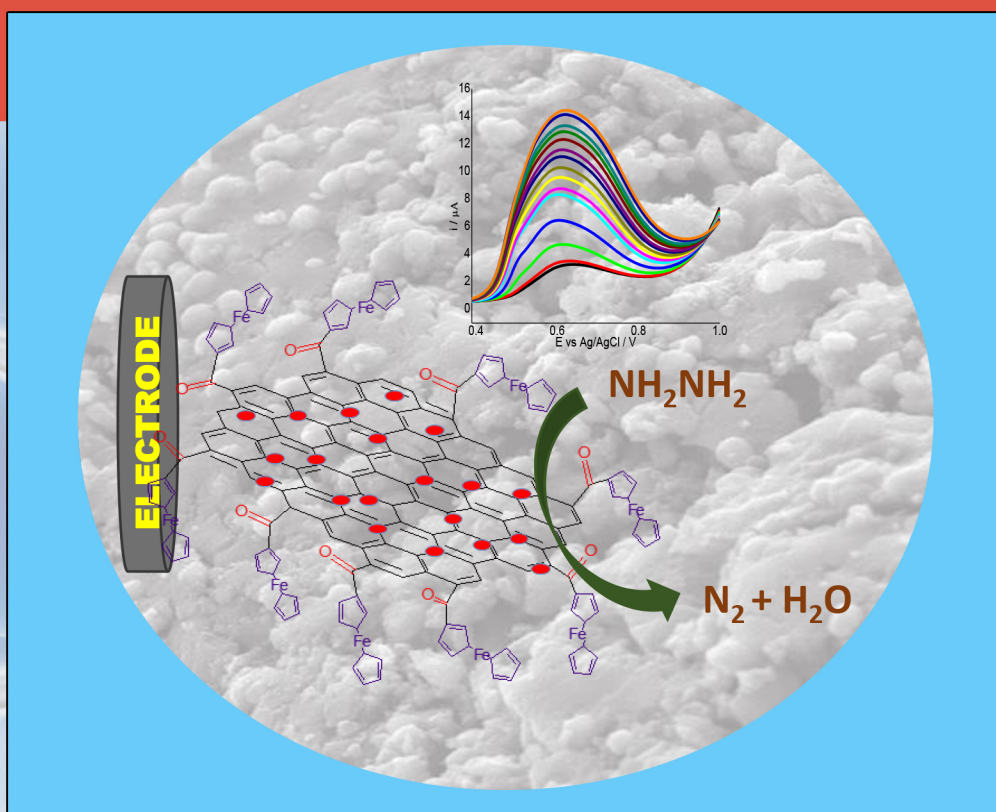


NANOCOMPOSITE MATERIALS FOR SENSORS



Editors:

Manorama Singh

Vijai K Rai

Ankita Rai

Bentham Books

**Current and Future
Developments in Nanomaterials
and Carbon Nanotubes**

(Volume 2)

***(Nanocomposite Materials for
Sensors)***

Edited by

Manorama Singh

*Department of Chemistry,
Guru Ghasidas Vishwavidyalaya,
Bilaspur, CG-495009
India*

Vijai K Rai

*Department of Chemistry,
Guru Ghasidas Vishwavidyalaya,
Bilaspur, CG-495009
India*

&

Ankita Rai

*School of Physical Sciences,
Jawaharlal Nehru University,
New Delhi-110067
India*

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Preface

Nanocomposites are rapidly emerging as novel materials for sensor technology; therefore, the scientific community has recently focused on the advancement in the development of innovative methods and materials relied upon efficient composites. The implementation of nanocomposite materials for the development of specific and sensitive sensing platforms receives good attention. This book focuses on the reviews of important reported literature for new approaches of nanocomposite material preparation and their applications in the development of physical, chemical, electrochemical, biological, and optical sensors, *etc.* These nanomaterials have been extensively used widely (to amplify the signal) in the detection of heavy metal ions, vital signs (*i.e.*, glucose, *etc.*), explosives, hydrazine, humidity, *etc.*

This book focuses on representing some state-of-the-art review chapters based on reported works in the last few decades, outlining the synthesis, role, and progress of nanocomposite materials in fabricating flexible and multifunctional sensing platforms in sensor technologies. The book is intended to prepare a highly compiled knowledge for designing novel nanocomposite materials to be used as sensing platforms in sensor technologies. A broad range of readers such as graduates and post-graduates, Ph.D. scholars, faculty members, professionals working in the area of material science, the healthcare industry, biological sciences, medical sciences, environmental science will be benefitted from the topics preferred in the proposed book.

Manorama Singh

Department of Chemistry
Guru Ghasidas Vishwavidyalaya
Bilaspur, CG-495009
India

Vijai K Rai

Department of Chemistry
Guru Ghasidas Vishwavidyalaya
Bilaspur, CG-495009
India

&

Ankita Rai

School of Physical Sciences
Jawaharlal Nehru University
New Delhi-110067
India

List of Contributors

A Rajapriya	Department of Nanoscience and Technology, Bharathiar University, Coimbatore, Tamilnadu, India
A Rebekah	Department of Nanoscience and Technology, Bharathiar University, Coimbatore, Tamilnadu, India
Anu Rose Chacko	Mahatma Gandhi University, Kottayam, Kerala 686560, India
Archana Aravind	NSS Hindu College, Changanashery, Kerala, India
Ashoka S	Dayananda Sagar University, Bangalore, India
Beena Mathew	Mahatma Gandhi University, Kottayam, Kerala, India
Chandra Shekhar Kushwaha	Department of Polymer Science, Bhaskaryacharya College of Applied Sciences, University of Delhi, Delhi, India
D Amilan Jose	Department of Chemistry, National Institute of Technology, Kurukshetra, India
D Navadeepty	Department of Nanoscience and Technology, Bharathiar University, Coimbatore Bharathiar University, Coimbatore, Tamilnadu, India
G Srividhya	Department of Nanoscience and Technology, Bharathiar University, Coimbatore, Tamilnadu, India
J Debbarma	Department of Chemistry, National Institute of Technology, Agartala, Tripura (West), India
Juhi Srivastava	Department of Chemistry, MMV, Banaras Hindu University, Varanasi, Uttar Pradesh, India
M Swapna Sai	Centre for Applied Research, Chennai Institute of Technology, Chennai, India
M Varsha Shree	Centre for Applied Research, Chennai Institute of Technology, Chennai, India
Manju Srivastava	Dayalbagh Educational Institute, Agra, Uttar Pradesh, India
Meenakshi Singh	Department of Chemistry, MMV, Banaras Hindu University, Varanasi Uttar Pradesh, Uttar Pradesh, India
Mitali Saha	Department of Chemistry, National Institute of Technology, Agartala, Tripura (West), India
N Ponpandian	Department of Nanoscience and Technology, Bharathiar University, Coimbatore, Tamilnadu, India
Nancy Sharma	Department of Chemistry, National Institute of Technology, Kurukshetra, India
Nazia Siddiqui	Dayalbagh Educational Institute, Agra, Uttar Pradesh, India
Prashanth S. Adarakatti	SVM Arts, Science and Commerce College, Karnataka, India
Pratibha Singh	Department of Polymer Science, Bhaskaryacharya College of Applied Sciences, University of Delhi, Delhi, India
S Keerthana	Department of Nanoscience and Technology, Bharathiar University, Coimbatore, Tamilnadu, India

Sachin Saxena	Dayalbagh Educational Institute, Agra, Uttar Pradesh, India
Sajini T	St Berchmans Autonomous College (Affiliated to Mahatma Gandhi University), Kottayam, India
Saroj Kr Shukla	Department of Polymer Science, Bhaskaryacharya College of Applied Sciences, University of Delhi, Delhi, India
Srushti Gadiyaram	Department of Chemistry, National Institute of Technology, Kurukshetra, India
Suma B. Patri	Department of Chemistry, Bangalore University, Central College Campus, Bengaluru, India
V Dhinakaran	Centre for Applied Research, Chennai Institute of Technology, Chennai, India

CHAPTER 1**Nanocomposites: Introduction, Structure, Properties and Preparation Methods****V Dhinakaran^{1,*}, M Swapna Sai¹ and M Varsha Shree¹**¹ Centre for Applied Research, Chennai Institute of Technology, Chennai-600069, India

Abstract: The production of composites and materials based on nanocellulose has attracted considerable attention in the last few decades since their abundance, renewability, high strength and rigidity, environmental friendliness, and low weight are all unmissable and potentially useful. This analysis deals with crucial factors in the manufacture of nanocellulose composites and presents and explores different composite processing techniques. Rare combinations of features and new design opportunities are seen in high-performance nanocomposites. Their potential is so high that their utility in different fields, ranging from packaging to biomedicine, with an annual growth rate projected at around 25% and a standardized summary emphasizes the need for such products, their methods of fabrication, and several recent studies on structure, properties and potential applications. There is a focus on the possible use of naturally occurring materials like clay-based minerals, chrysotile and lignocellulose fibers. In this chapter, an overview of nanocomposites is deliberated in detail and the nanocomposite applications provide new technology and business options for different industries in the aerospace, vehicle, electronics, electrical and biomedical engineering sector as they are naturally friendly.

Keywords: Carbon nanotubes, Nanocomposites, Nanometers, Polymer matrix, Scanning tunnel microscope, Sensors.

INTRODUCTION

At the atomic or molecular level, nanotechnology engineering is the collective term for a wide variety of processing technologies and measurements involving the smallest scale handling of matter from 1 to 100 nanometers. The processing of particles and materials at nanoscale dimensions is concerned with nanotechnology [1]. Nanocomposites are composites in which the nanometer range of at least one of the phases is $1 \text{ nm} = 10^{-9} \text{ m}$. Because of their outstanding Properties, nanocomposites are potential alternatives to micro composites and monolithic and consist of two or more distinct constituents or phases of physical and chemical

* Corresponding author V. Dhinakaran: Centre for Applied Research, Chennai, Institute of Technology, Chennai-600069, India; Email: dhinakaranv@citchennai.net

properties, which are separated by a separate interface [2]. However, nanocomposite preparation techniques face challenges due to the regulation in the nanophase of elemental composition and stoichiometry. The constituent, which is normally more current, is known as the matrix. In order to improve the mechanical characteristics of nanocomposites, the component is called reinforcement in the matrix material or nanomaterials [3]. Strengthening is normally made of nanosized fillers. In general, anisotropic nanocomposites occur because of the distinct properties of constituents depending on the direction and because the reinforcement is inhomogeneous [4]. In addition, as dimensions reach the level of nanometers, interactions at interfaces are much better and appropriate for improving the material's properties. In these cases, the surface area or volume ratio of the materials used for the preparation of nanocomposites is essential for understanding the structure-property links [5]. Furthermore, the discovery and subsequent use of Carbon Nanotubes (CNTs) for the manufacturing of composites showing some of the special mechanical, thermal, and electric characteristics of CNT introduced a new and fascinating dimension [6]. Further advances in the production and application of CNT-containing nanomaterials were rendered by the possibility of spinning CNTs into composite products and textiles [7]. In addition to being environmentally sustainable, nanocomposites now deliver new technologies and market opportunities for all industries. A large range of materials, where one of the dimensions belongs to a nano range, is defined by nanocomposite. In certain cases, nanocomposites are stronger than typical composites [8]. Because of their excellent properties, nanocomposites are extremely good alternatives to traditional composite materials and are used in many areas [9].

STRUCTURE OF NANOCOMPOSITES

Nanocomposite architecture usually comprises a matrix of particle, whisker, fiber and nanotube nanosized reinforcement components. Several researchers have used various equipment and techniques for characterizing nanocomposites, including microscopy Atomic Force Microscopy (AFM), Scanning Tunnel Microscopy (STM), Fourier Transformed Infrared Spectroscopy (FTIR), X-ray photoelectron, Nuclear Magnet Resonance (NMR), Differential Calorimetry Scan (DSC), and scanning and transmission of microelectrons [10]. The AFM is a powerful method to research the surface up to the nanometer level. Simultaneous experiments have been used on quantitative characterizations of nano-structuring and crystallite structures of some nanocomposites at Small Angles of X-Ray Dispersion (SAXS) and X-Ray Diffractometry (XRD). Furthermore, theoretical calculations and simulations were developed to predict the force properties, including stress and strain curves [11]. A brief description of the CNTs will be given here due to their distinctive properties, compared with other refurbishments, before the structure

and properties of nanostructures such as CNTs are discussed [12]. In short, SWCNTs have a metal density of less than one-sixth, while MWCNT is roughly half the metal density. Tensile strengths of SWCNT and MWCNT have been stated to be considerably higher than steel with high resistance, while the values of Young's diamond module are comparable [13]. They show exceptional resilience because the deformations of plastic metal and carbon fiber fractures are different from bowing and rejuvenating without damage. Also, thermal and electrical theoretical conduciveness with an almost zero coefficient of thermal expansion is equal to diamond [14]. In contrast to lower metal wires in microchips and high magnetic parallel perpendicular susceptibility, they ensure good thermal stability in both air and vacuum. Theoretically, these materials have surface values of 3000 m²/g, although the calculated gas value is different [15].

PROPERTIES OF NANOCOMPOSITES

The properties of nanocomposites depend not on the characteristics of each component but on the parameters (processes used in the development of nanocomposites) (type and orientation of filling materials, improvement of mechanical efficiency of the parental material, improved clarity due to small scale, small, high looks and therefore large area particles) [16]. Particles should adequately be dispersed and spread into matrix material in order to attain the improved nanocomposite characteristics, as otherwise the particulate matter will agglomerate and the nanocomposite characteristics will deteriorate. Particles must be properly distributed and distributed to the matrix [17]. The nanocomposite's most common feature is the layering of the interface between the matrix and the filler material. The interface properties, composition and microstructure of the filler vary from the interface matrix [18]. The interface between the nanofillers and the polymer matrix optimizes the interactors and can therefore be adapted to fit the superficial bonding surface, so the overall properties of the nanofillers are quite profound [19]. The interface region is highly interconnected with the matrix and filler. In relation to the relationships, the surface energy filling and matrix ratio are calculated [20]. The properties of nanocomposite depend on their microstructure. The relation between the structural characteristics of nanocomposite's polymer nanoplatelets defines the morphological nature of the composite system [21]. A good nanomaterial dispersion is hard to achieve, especially for non-polar polymers, but a consistent distribution of nanoplatelets guarantees good nanomaterial quality [22].

CLASSIFICATION OF NANOCOMPOSITES

Nanocomposites are graded in accordance with the forms of material reinforcement and matrix used in their construction [23]. Nanocomposites are

CHAPTER 2

Nanocomposites: A Boon To Material Sciences**Sachin Saxena^{1,*}, Nazia Siddiqui¹ and Manju Srivastava¹**¹ *Dayalbagh Educational Institute, Dayalbagh-282005, India*

Abstract: The imperfections of microstructures and monolithic in different realms of material sciences have been completely engulfed by the improved characteristics and excellent properties of nanocomposites. Their multiphase components with nano dimensions provide these structures with much superiority over conventional composites. This paper is a brief review about nanocomposites and their classification. Based on the dimensionality of particle size these can be grouped into one-two- and three-dimensional nanomaterial derived composites while if the number of components form the basis, they can be classified into binary, ternary and quaternary nanocomposites. This work also discusses and focuses on the computational analysis of nanocomposites with designing and energy calculation studies, based on DFT, and other mathematical tools and models. The role of metal organic framework-based nanocomposites in sensor fabrication and quantification of different redox system have also been listed.

Keywords: Nanocomposites, Binary, Ternary, Quaternary, Metal organic frameworks.

INTRODUCTION

This decade has observed a detailed review and research reports in the field of nanocomposites, either from prosthetics [1, 2], food and health corporations [3 - 5], chemical industries [6], or in highly sensitive and specific electrochemical applications [7 - 10]. The composites and in case, one or more phases are in nanoscale, the nanocomposites, have many superior properties than their precursors.

This blending of properties of single components provides extended range of high mechanical strength, chemical inertness, thermal stability, surface to volume ratio and better optical properties with much insignificant wear and tear losses *etc* [11]. It can be stated, that the reinforced material (phase) distribution or dispersion in the continuous phase or the matrix determine the performance of the nanocompos-

* **Corresponding Author S. Saxena:** Dayalbagh Educational Institute, Dayalbagh-282005, India; Email: sachinusic@gmail.com

ite [12]. In other words, the homogenous distribution of the particles in the matrix is the key to the properties found in the nanocomposite. If the distribution of particles is homogenous, a sufficient amount of reinforcement will occur, this will lead to better interfacial interactions, but this is rarely found. Due to this, inferior properties of the nanocomposite may also be observed.

The properties of the nanocomposites are governed by essential factors that are needed to be optimized. These factors are mainly

- characteristic of matrix
- structure
- composition (the filler content), and
- interfacial interactions.

High aspect ratio, adhesion and reinforcement component makes the composite with enhanced principal properties. These properties rely on the structure of nanocomposite which is dependent upon the surface to volume ratio of the reinforced phase. Further, these peculiar properties of the nanocomposite are the outcome of the different interfacial interactions [13].

The nanomaterials used are basically nanofillers, nanoclays and nanoparticles. The characteristics of matrix and fillers highly influence the properties of nanocomposites. Literature reports the decrease in matrix stiffness leads to an increase in reinforcement property while the purity of filler material affects the nanocomposite applicability, for *e.g.*, in glassy polymer the matrix stiffness and reinforcement observed is moderate, while in elastomers very large increase is observed, in case of impure filler nanomaterial decrease in stability and even discoloration is observed. Heavy metal contamination and impurity affect the exfoliation and deteriorate the properties of the nanocomposite [14].

CLASSIFICATION

As per the studied classifications, nanocomposites are classified on the basis of polymer and non-polymer nanocomposites [15]. Further, if the criteria basis is reinforcement material, they can be classified into carbon nanotubes, polymer, noble metal and metal oxide-based nanocomposites. If matrix material is concerned, the nanocomposites may further be grouped into ceramic, polymer and metal matrix nanocomposites, respectively [16]. Detailed work has been accounted comprehensively in books and research articles, regarding the classification of nanocomposites. Here we can further classify these nanocomposites on the basis of number of components:

- Binary nanocomposites
- Ternary nanocomposites; and
- Quaternary nanocomposites

If the dispersion of nanofillers is heterogeneous, then according to the dimensionality and particle size, nanocomposites can be divided into 3 groups:

- One dimensional
- Two dimensional; and
- Three dimensional

When the size in all three dimensions is in nanoscale (<100nm), such particles include TiO₂, CaCO₃, SiO₂, oligomeric silsesquioxane *etc.*

Nanofibers and nanotubes lie in the two-dimension range. It is also found in the micrometer range.

Layered silicates have larger dimensions but one of its dimensions is found to be almost 1nm thick [13].

Based on the components and their properties, nanocomposites can be summarized as below:

Binary Nanocomposite

Production of advanced nanocomposites nowadays, has resolved many complex problems in real-life applications. One such application is decontamination of wastewater (removal of inorganic contaminants, degradation of dyes, *etc.*) using spinel ferrite MFe₂O₄ based binary nanocomposite [17]. Further, the metal oxides binary nanocomposites have been found to detect complex organic compounds and serve as better catalytic nanocomposites. They enhance the electron transfer mechanism by serving acidic and basic components in a single entity, due to which a high adsorption phenomenon occurs [18 - 20]. The adsorption rate is enhanced by the presence of nanofillers or nanomaterials used during its preparation. Recently, Hareesh et al. 2020 have summarized in a review that the graphitic carbon nitride binary nanocomposites can be used as energy storage materials, owing to high supercapacitor performance [21]. Ce and Cd binary metal oxide nanocomposites exhibited good antibacterial, antimicrobial activity and have reported absorption spectra of binary metal oxide nanocomposite [22].

Bimetallic-Carbon Based Composites for Electrochemical Sensors

S. Keerthana¹, A. Rajapriya¹ and N. Ponpandian^{1,*}

¹ Department of Nanoscience & Technology, Bharathiar University, Coimbatore 641 046, Tamilnadu, India

Abstract: The robust, sensitive, and selective finding of various biomolecules and environmental factors by potential nanostructures holds much promise for accurate electrochemical sensors. However, to be competitive, present electrochemical sensor technologies need noteworthy developments, particularly in specificity output rate, and long-lasting steadiness in complex biological environments. Bimetallic carbon nanocomposites are newly emerging materials with fascinating physicochemical properties and are very prospective in the innovative point-of-care study of various healthcare issues. Particularly, the multidimensionality of bimetallic carbon composites and their structural, optical, electronic, and electrocatalytic properties are suitable for the design of various electrochemical sensing devices. This chapter summarizes the sensing applications of bimetallic @C and its modern advances in the detection of different analytes. The chapter begins with a brief introduction to the advancement of bimetallic @C based electrochemical sensors followed by the discussion of the structure and properties of the bimetallic @C nanocomposites. We also discuss in detail the utilization of these bimetallic @C nanocomposites with graphene, MWCNTs, CQDs, and g-C₃N₄ for their worthwhile application in electrochemical sensors. Finally, the chapter concludes with a positive outlook on the use of bimetallic @C nanocomposites for day-to-day life and clinical applications based on the present growth.

Keywords: Bimetallic, Biomolecules, Biosensors, Carbon nanomaterials, Electrochemical sensors, Graphene, Sensitivity, Selectivity.

INTRODUCTION

Due to the present advancement and demands in the field of medical diagnosis and several environmental applications, the development of fast, sensitive, and selective analytical techniques are required for the sensing of health factors as the clinical investigations in laboratories are expensive and time-consuming processes in the hospital point-of-care settings [1]. A variety of detection techniques are

* Corresponding Author N. Ponpandian: Department of Nanoscience & Technology, Bharathiar University, Coimbatore 641 046, Tamilnadu, India; Email: ponpandian@buc.edu.in

available such as chromatography, chemiluminescence, and spectroscopic techniques [2, 3]. Among them, the technique of electrochemical analytical techniques has many outstanding properties like accuracy, reliability, sensitivity, and specificity over the other methods. Moreover, electrochemical sensors have gained massive attention in the field of pharmaceutical, biological, environmental, and food industries.

These electrochemical devices should also have additional features like high flexibility, non-invasive detection, wearability, biocompatibility, lightweight, ease of fabrication, and cost-effectiveness [4, 5]. The analytical techniques to study the electrochemical characteristics include cyclic voltammetry, differential pulse voltammetry, and square-wave amperometric techniques. All of them are effective techniques to study and determine the electrochemical behavior of prominent electrodes developed for electrochemical sensors. These electrochemical processes can be influenced by many factors, such as the nature of the analyte, structure of the electrode material, type of the electrode, and the choice of the electrolyte [6]. Among all factors, the electrode material plays a crucial part and the researchers are particularly driven to prepare an effective electrode material for high-quality electrochemical sensors. Generally, numerous nanomaterials, including metal-nanoparticles (NPs), metal oxides, polymer nanocomposites, various types of 2D nanomaterials transition metal dichalcogenides, black phosphorous, layered double hydroxides, and graphene, *etc.*) and the carbon derivatives are widely exploited and investigated as the electrode material for electrochemical detection. This chapter comprises an analysis of electrochemical sensors employing the composites of bimetallic NPs and carbon-based materials, which can improve both the sensitivity and selectivity of the sensors. Carbon nanomaterials and their allotropes like carbon nanotubes (CNTs), graphene oxide (GO), carbon-based quantum dots (CQDs), crystalline diamond, and fullerenes [7] exhibit outstanding electrochemical properties, which have resulted in their employment in a wide range of applications. Currently several bottom-up and top-down techniques are available to prepare carbon structures with tuned uniformity, size, and morphology by varying reaction parameters. Hence, the above-mentioned diverse carbon nanomaterials are deemed to be wonder materials in the electrochemical field due to their excellent properties like electrical conductivity, high mechanical strength, higher electron transfer kinetics, and large surface area [8]. NPs of noble metals and non-noble metals have been studied for several decades and the precious metals are shown great interest owing to their greater surface area and number of corner atoms and edges, which greatly enhance their catalytic activity [9]. Bimetallic NPs are significantly unique from their monometallic counterparts and have attained great attention from both technical as well as scientific points of view owing to their distinctive catalytic properties [10]. By choosing the suitable combination of metal NPs and their sizes, we can

tune the properties and hence, the performance of the nanostructures. However, pure metals (mainly Pt or Au) have poor sensitivity and selectivity. Bimetallic NPs composed of two dissimilar metals consume greater attention than monometallic NPs from both technical as well as scientific points of view. Some approaches have been adopted for the synthesis of metal NPs like chemical reduction using reducing agents like sodium borohydride, citrate, hydrazine, and glucose [11]. The synergy of carbon materials with the appropriate addition of metal NPs can inhibit the aggregation of composites and so, improves the catalytic activity because of the interaction and surface reactivity between the support and the supported electrocatalysts. Therefore, many pieces of research have been accomplished on the preparation of electrochemical sensors with highly efficient bimetallic-carbon nanocomposites [12]. As a result, it is deemed that combining the carbon materials and bimetallic NPs to construct an effective electrochemical sensing platform is a noble alternative to detect complex systems with interfering compounds.

STRUCTURE AND PROPERTIES OF BIMETALLIC NPS AND CARBON NANOMATERIALS

Carbon-based materials such as derivatives of graphene, carbon nanotubes, and carbon quantum dots are shown in Fig. (1a). Graphene, the greatly explored carbon material is a layered sheet of sp^2 -bonded carbon atoms. Graphene is synthesized by various top-down and bottom-up approaches [13]. The family of graphene-related materials includes structural or chemical derivatives of graphene, such as a few layers of graphene. Another derivative is graphene oxide (GO), which is an oxygenated derivative of 2D graphene, and reduced graphene oxide (rGO), a product of the reduction of GO. CNT is an allotrope of carbon exhibiting a narrow cylindrical structure, composed of sp^2 hybridized carbon hexagon network rolled up with at least one end-capped with fullerene molecule. The sp^2 hybridization builds a stacking of layers with van der Waals force combined with weak out-of-plane and strong in-plane force. The general classification of CNTs includes single-walled carbon nanotube (SWCNT) and multi-walled carbon nanotube (MWCNT). SWCNT is known to be a single layer of rolled carbon network with a diameter ranging from 1-2 nm and MWCNTs can be presumed as a few graphene sheets rolled into hollow cylindrical tubes with a high aspect ratio. MWCNTs have numerous advantages, such as excellent thermal conductivity, higher electrical conductivity, and chemical stability. Due to their excellent physicochemical characteristics, MWCNTs can effectively serve as electrodes for electrochemical analysis of different chemicals, food preservatives, clinical and environmental agents. MWCNTs based electrochemical sensors show enhanced sensitivity, a lower limit of detection because of their high aspect ratio and rapid electrode kinetics [14]. GQDs, a novel class of carbon nanomaterials,

CHAPTER 4**Two-dimensional Graphene-based Nanocomposites for Electrochemical Sensor****J. Debbarma¹ and M. Saha^{1,*}**¹ *Department of Chemistry, National Institute of Technology Agartala, 799046, Tripura, India*

Abstract: This chapter reviews the usage of graphene nanostructures in the fabrication of electrochemical sensors. In current ages, graphene derivatives have attracted a great deal of attention due to their outstanding electrical, mechanical, and thermal properties, making them one of the most popular choices to develop the electrodes of a sensor. In addition, the high effective surface area, electrocatalytic activity, excellent electrical conductivity, high porosity and adsorption capability, fascinating their electrochemical properties, which turn them as potential candidates for electrochemical applications, particularly sensing. This chapter deals with an overview of the work done on graphene-based nanocomposites in very recent years. It explains the properties of graphene-based nanocomposites for their usage as electrochemical sensors.

Keywords: Electrochemical sensor, Graphene, Nanocomposite.

INTRODUCTION

The remarkable properties of carbon family members, including fullerenes, carbon nanotubes and graphene, have stimulated a broad range of researches in various applications. All the carbon nanomaterials, especially graphene is the latest family member, have attracted considerable attention, because of their unique structure and properties. Moreover, the large surface area, high electrical and electrochemical activities make them suitable candidates for developing energy-related devices (supercapacitors and batteries) as well as sensors. Electrochemical sensors have proved to be powerful analytical tools which have great potential to improve both selectivity and sensitivity through tuned signal amplifications. This technique also possesses outstanding properties such as low cost, fast response time, instrumental simplicity, the possibility of miniaturization, and integration in portable devices.

* **Corresponding Author Mitali Saha:** Department of Chemistry, National Institute of Technology Agartala, 799046, Tripura, India; E-mail: mitalichem71@gmail.com

In addition, another distinguishing feature of electrochemical sensors is their capability of detecting a wide range of compounds from neutral molecules, organic, inorganic, ionic and metal ions. The combination of these fascinating properties with graphene and its derivatives has been emerged in powerful electrochemical sensing platforms based on so called “graphene modified electrodes” which are being used for the electroanalysis of a large range of analyte species. During the past decade, the modified electrodes based on graphene were applied for the determination of various compounds and have been extensively applied as the modifier electrodes because of their chemical stability, antifouling resistance as well as large adsorption ability towards various analytes. This chapter represents an overview of the current state of researches on the application of electrochemical sensors with various electrode modification approaches using graphene. The electrochemical sensors based on graphene and its composites for the determination of diverse target analytes such as drugs, pharmaceutical formulations, biological species, neurotransmitters, biomarkers and heavy metals, in very recent years have been discussed in detail.

ELECTROCHEMICAL SENSING OF VARIOUS ANALYTES USING GRAPHENE BASED NANOCOMPOSITES

In recent years, graphene and its derivatives have provided a most promising platform for the sensing of various electrochemical analytes. More recently, FGP/AuNC nanocomposite was prepared *via* ultrasonic method based on the fact that AuNC facilitated to reduce the agglomeration of graphene materials and also improves the catalytic activity of nanocomposite as the synergistic effect of the AuNC and derived graphene materials enhances their physical and chemical properties, thus providing a novel technology for real-time monitoring of heavy metal pollution in food [1]. The electrochemical detection of amikacin sulphate was examined by utilizing a nanocomposite of silver nanoparticles and reduced graphene oxide (AgNPs/rGO) on a nickel foam (NiF) electrode. The practical viability of the developed sensor was evaluated in spiked human urine samples under validated conditions and the recovery rate was found to be in the range of 99%–102%, which indicates the sensor’s effectiveness in the detection of amikacin sulphate [2]. Fe_2O_3 NPs is a promising catalyst for rapid electron transfer between the electrode and the active site of the redox system. Hence, a one-pot hydrothermal method was carried out for the green synthesis of $\gamma\text{-Fe}_2\text{O}_3$ (~40 nm) decorated with N-rGO hydrogel which exhibits electrochemical detection performance of 4-Nitrophenol under neutral pH [3]. It is well known that; metal or metal oxide nanoparticles exhibit potential catalytic performances for sensing various analytes. Among various metal/metal oxide nanoparticles, ZnO acts as a promising candidate for the electrochemical detection of bio analytes due to its significant features such as large loading capacity, easy

fabrication, biocompatibility, highly physicochemical stability and electrocatalytic activity. Owing to these properties, (rGO)-g-CN/ZnO nanocomposites were developed for the electrochemical analysis of sunset yellow in real samples [4]. Conductive polymers possess unique physical and chemical properties, which enhance their electrocatalytic activities towards dopamine and ascorbic acid [5]. Polymers such as polyaniline-co-thionine based GO/P(ANI-co-THI) composite modified electrode was successfully prepared *via* a simple one-step electrochemical deposition method. A nanocomposite consisting of polyaniline-GO-iron tungsten nitride was developed and then applied for accurate detection of 4-NP in the aqueous samples through a real-time and repeatable approach, making it a fantastic alternative for the development of precise sensors and electronic devices [6]. A sensor was fabricated based on a modified screen-printed carbon electrode (SPCE) for Rohypnol measurements. The surface of SPCE was modified with amine-functionalized graphene oxide sheets reinforced through Cu nanoparticles (SPCE/AGO-Cu). It provides an accurate and precise assay pathway of Rohypnol in the fruit juice [7]. Biochar and reduced graphene oxide (rGO) were prepared as a modifier electrode for the construction of an electrochemical sensor for sensing carbendazim (CBZ, methyl benzimidazol-2-yl carbamate) in different samples. The modified electrode showed to be able to preconcentrate CBZ and presented the highest analytical response in comparison to the unmodified electrode [8]. In 2020, graphene oxide based CaTiO₃NPs nanocomposite was prepared by an *in-situ* sonochemical process, which applied towards electrocatalytic and electrochemical sensing of chemotherapeutic drug flutamide. This bimetal (CaTiO₃) enhances the electrocatalytic performance towards detection of flutamide drug [9]. A simple modified glassy carbon electrode (PVP-GR/GCE) based on the polyvinylpyrrolidone (PVP)-graphene film was also reported in the same year. This modified electrode was used for the determination of dopamine, uric acid, and ascorbic acid in real samples. It was established that PVP was allowed to make the carbon atoms less prone to aggregation in graphene nanosheets. Hence, the dispersion and stability of PVP-GR nanocomposite improved compared with pure GR [10]. An electrochemical sensor was developed with a metal-organic gel (MOG) consisting of Fe₃Cl(H₂O)₂O clusters interconnected with 5-aminoisophthalic acid (NH₂-ip) in the presence of isophthalate functionalized graphene (IG) for sensing of dopamine. Here, IG acted as a structure directing templating agent which also enhanced conductivity of the material [11]. Electrochemical sensor based on rGO-Ag@SiO₂ nanocomposite was established to analysis etidronic acid (EA) using the differential pulse voltammetric (DPV) technique. The real-time application of the rGO-Ag@SiO₂/Au PCB for EA detection was investigated using EA-based pharmaceutical samples. Recovery percentages were found to be 96.2% to 102.9% [12]. It was found that, copper has high electrochemical activity, low

Conducting Polymer Based Nanocomposites for Sensing

D. Navadeepthy¹, G. Srividhya¹ and N. Ponpandian^{1,*}

¹ Department of Nanoscience and Technology, Bharathiar University, Coimbatore 641 046, India

Abstract: The enormous development in the industrial and ecological base has led to increasing concern over the advent of new materials with implicit properties. A considerable interest has grown in conducting polymer nanocomposites as they are widely attracted for diverse applications due to the combinatory effect of conducting polymers and electrical and chemical properties of inorganic nanoparticles. The result was incredible with unique functionality, conductivity, structure, reactivity, processability (colloidal stability or mechanical strength), and sensitivity with biodegradable and bio-compatible nature. The unique features of conducting polymer nanocomposites have gained attention in multiple applications. Recently, conducting polymer based nanocomposites are widely utilized as nanosensors in detecting temperature, stress, toxic gases and bio-elements. The present chapter deeply discusses the types of conducting polymer nanocomposites, as novel hybrid materials for sensor applications.

Keywords: Conducting polymers, Hybrid materials, Nanocomposites, Oxidation, Polymerization, Reduction, Sensors.

INTRODUCTION

The advancement in the field of material science has led to the development of new materials with unique properties and revolutionary combinations. One such novel invention is conducting polymers. Polymers are large compounds that are made of smaller subunits (monomers) in various ways or patterns to form a chain. Basically, polymers are insulators commonly used as coatings on wires and cables for electrical applications. A new era has begun with the invention of conducting polymers in the year 1977 by Hideki Shirakawa, Alan G. MacDiarmid, and Alan J Heeger. The discovery won the polymers arises from the π - conjugated polymer network.

* Corresponding author N. Ponpandian: Department of Nanoscience and Technology, Bharathiar University, Coimbatore 641 046, India; Email: ponpandian@buc.edu.in

The Conducting polymers, for the past two decades, have been highly employed in a wide range of applications such as sensors, catalysis, energy storage, shielding electromagnetic radiations, corrosion resistance, medicinal device constructions, and electronic devices (displays, memory cells, transistors) [3]. Conducting polymers possess inimitable electrical, mechanical, optical, and conducting properties with high tunable capability, low cost, less weight, easy preparation methods and resistance to corrosion [4]. With the improvement in researches on nanoparticles and new functional materials, nanocomposites have evolved. Nanocomposites are the combination of two or more nanoparticles or nanoclusters in such a way that the properties of the parent constituents are not affected. By successful choice of combinations, highly potential nanocomposites applicable for a wide range of applications can be obtained [5].

Conducting polymer nanocomposites is the combination of conducting polymers (Polyaniline, polypyrrole, polyacetelene, polythiophene and PEDOT) and other inorganic nanoparticles or metal oxides to attain new characteristics. Among the various fields utilizing such improved physical and chemical properties, the conducting polymer nanocomposites are used to design sensors to detect various types of analytes such as solvent, vapor, strain/stress, pressure, and temperature [6, 7].

In this regard, the present chapter summarizes the introduction and types of conducting polymers and its nanocomposites, their preparation methods and applications in general. More specifically, the employment of conducting polymer nanocomposites for sensor applications is further discussed in detail.

CONDUCTING POLYMERS - AN INTRODUCTION

Conducting Polymers (CP) are novel materials with a combination of properties of organic polymers and semiconductors which are technically called as smart polymers. The electrical and optical properties of CPs are similar to a metal, whereas the mechanical properties of polymers are retained. For more than three decades, conducting polymers are dominating all fields of applications due to their intrinsic properties and flexibility. The high surface area of conducting polymers is highly advantageous for various types of applications, since the natural polymers generally do not possess these properties and do not come under the category of nanoparticles which are the most wanted and explored component of researchers. Conducting polymers are semi-conducting in nature in their pristine state, which can be tuned to become highly conducting by doping [8,9]. Conducting Polymers exert charge mobility along their p-electron back-bones, which are responsible for their unusual electronic properties. Conductivity of Cps thus exclusively depends on the delocalized π - electrons. On doping the oxidation

and reduction states of the π -conjugated chains are modified to form p- or n-doped conducting polymers. Based on the methods and chemical components used for doping, the oxidized or reduced forms of conducting polymers are generated. Mainly polymers exist as either cation or anion radical or di-cation, or di-anion species [2,9]. The conductivity of these doped polymers can be tuned by changing the nature of dopant, degree of doping, manipulating the polymer chain backbone or by blending with other polymers. The conducting polymers are polyacetylene (PA), polyaniline (PANI), polypyrrole (PPy), polythiophene (PTh), poly(para-phenylene) (PPP), poly(phenylenevinylene) (PPV), polyfuran (PF), etc. Among them, PANI, PPy, PEDOT and PTh possess some unmatched properties which are highly scrutinized for various applications [10].

CLASSIFICATIONS OF CONDUCTING POLYMERS

Conducting polymers can be classified based on their mode or nature of conduction. Initially the conducting polymers are broadly classified into two types

- i. Intrinsically conducting polymers
- ii. Extrinsically conducting polymers

In intrinsically conducting polymers, the conductivity depends either on the π -conjugated chains of the polymers or the dopants added to the polymers. They are highly advantageous, since they are easily tunable by ion exchanges and undergo oxidation or reduction easily. They have a high ability to store charges and are also optically efficient [11]. The classification of polymers is clearly shown in the chart in Fig. (1). In the latter type of polymers, conduction depends on the fillers in the polymers. The conductive fillers such as carbon black or fibers are added to the network to make them conductive. Extrinsically conducting polymers can also be formed by blending the insulators with conducting polymers. Such polymers possess bulk conductivity and are highly stable. They are cheap and easy processable. There is a third category of conducting polymers; they are inorganic conducting polymers which have charge transfer complexes [5]. The well-known CPs are polyaniline (PANI), polypyrrole (PPy), polythiophene (PTh), polyvinyl pyrrolidone (PVP), poly(3,4-ethylene dioxythiophene) (PEDOT), poly(m-phenylene diamine) (PMPD), polynaphthylamine (PNA), poly(p-phenylene sulfide) (PPS), poly(p-phenylene vinylene) (PPV), polyacetylene (PAC), polyfluorene, poly-phenylene and polypyrene [12]. Most of them are intrinsically conducting through their π -conjugated network, and their conductivity falls in the range 10^{-16} and 10^{-5} S/cm. Conducting polymers are either made directly by electro- or oxidative-polymerization or polymerized and then oxidized chemically or electrochemically. In the next section conducting polymers and their synthesis, structure, properties, and applications are briefly discussed.

CHAPTER 6

Nanostructured Molecularly Imprinted Polymers in Electrochemical Sensing

Sajini T¹ and Beena Mathew^{2,*}

¹ *Research & Post Graduate Department of Chemistry, St. Berchmans College (Autonomous), Affiliated to Mahatma Gandhi University, Changanassery-686101, Kerala, India*

² *School of Chemical Sciences, Mahatma Gandhi University, Priyadarsini Hills P O, Kottayam - 686560, Kerala, India*

Abstract: Molecular imprinted polymers (MIP) are one of the promising method in various research area in which artificial receptor sites of targeted molecule were fabricated on a polymer matrix. These polymers are analogues to naturally occurring antigen-antibody system. Due to its high recognition capability and structural specificity towards the target molecule, these kind of polymers exhibits wide variety of applications in various fields. Among the tremendous applications, MIPs in electrochemical sensing got much attention in recent years. Innovative developments in nanochemistry again improve its applications in electrochemical sensing. In this chapter, we detailed the significance of nanostructured MIP focusing on multiwalled carbon nanotubes as supporting material in electrochemical sensing applications. It presents recent progresses associated to molecularly imprinted electrochemical sensors based multiwalled carbon nanotubes.

Keywords: Applications, Electrochemical sensor, Molecularly imprinted polymer, MWCNTs, Nanostructured MIP.

INTRODUCTION

Molecular imprinting technology is the most developing area of research in which artificial binding sites of target or template molecule were created on a polymer network. A typical molecular imprinting process involves the fabrication of a pre-organized complex of chosen template molecule and its complementary functional monomer followed by a crosslinking polymerization in the addition of a suitable initiator [1]. Subsequently, the extraction of a target molecule using suitable eluents, complementary cavities of template molecule remains as such in the poly-

* **Corresponding author Beena Mathew:** School of Chemical Sciences, Mahatma Gandhi University, Priyadarsini Hills P O, Kottayam -686560, Kerala, India; Email: beenamsocs@gmail.com

mer matrix and the polymer composite formed were designated as molecularly imprinted polymer (MIP).

(Fig. 1) depicted the schematic representation of the formation of conventional MIP.

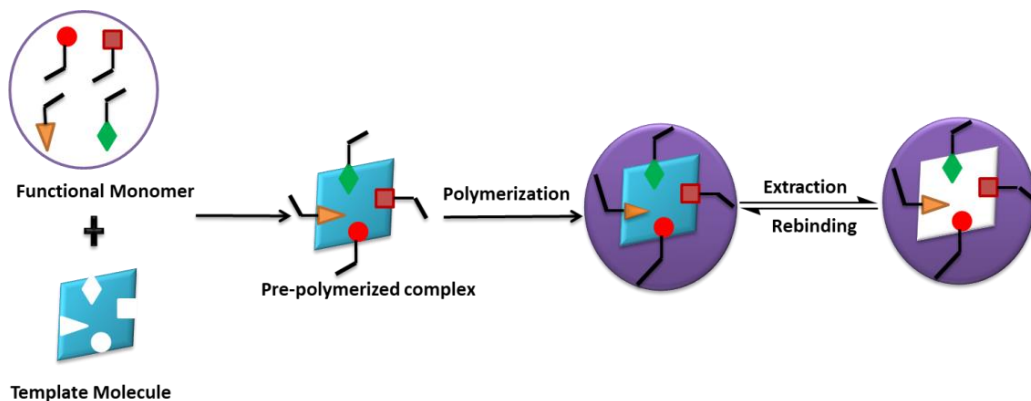


Fig. (1). Schematic representation of molecular imprinting process.

Electrochemical sensing mechanism involves the transformation of the interaction of the template molecule with a receptor on an electrode surface, which acts as a transducer, into a useful analytical signal. Mosbach and Haupt were the first to discover the electrochemical sensor with MIPs, which they used to create a MIPs-coated electrode known as a molecularly imprinted electrochemical sensor (MIECS) in 1999 [2]. The electrochemical sensor generated from MIPs possesses both the properties of detection and transduction. The advantage of MIECS includes high detection characteristics, low cost and ease of fabrication and simple automation. These MIECS have got a lot of coverage for their ability to track a wide range of molecules, including organic compounds, emerging pollutants, biomolecules, heavy metals and more.

Although biosensor technology has progressed dramatically [3], there are still some problems with biological materials used in biosensing, such as (i) they are less stable, (ii) expensive, and (iii) lack of receptor sites that are capable of differentiating certain template molecules. Due to its high stability, MIP can be ideal substitute for the biological receptors in sensors [4]. The common mechanism of MIP based electrochemical sensors comprises the utilization of MIP electropolymerized or immobilized onto the electrode surface in order to accomplish the different electrochemical analysis [5]. MIP has been established for sensing sugars, herbicides, derivatives of nucleic and amino acids, toxins, drugs and solvents [6]. MIP is used in other emerging fields such as mass

sensitive sensing devices, microbalance-based and fluorescence-based sensors, and in electrochemical methods includes, chemical luminance, surface plasmon resonance spectroscopy, and conductivity sensing devices [7 - 9].

In order to improve the high sensitivity and specific selectivity of the MIP based chemo-biosensors, nanomaterials, which have remarkable physicochemical properties and characteristics, were comprehensively introduced in recent years. Nanomaterials have got much attention as the fundamental materials during the MIP fabrication and in order to improve and strengthen the electrochemical signal, it acts as an electrode surface modifier. Nanostructured materials have the extremely large surface area and high electrical and thermal conductivity and hence exhibit an electrocatalytic property. The majority of literature reports that the MIP based electrochemical sensors are fabricated in association with nanomaterials, including graphene, magnetic nanoparticles, MWCNT, and gold nanoparticles which are illustrated in Fig. (2).

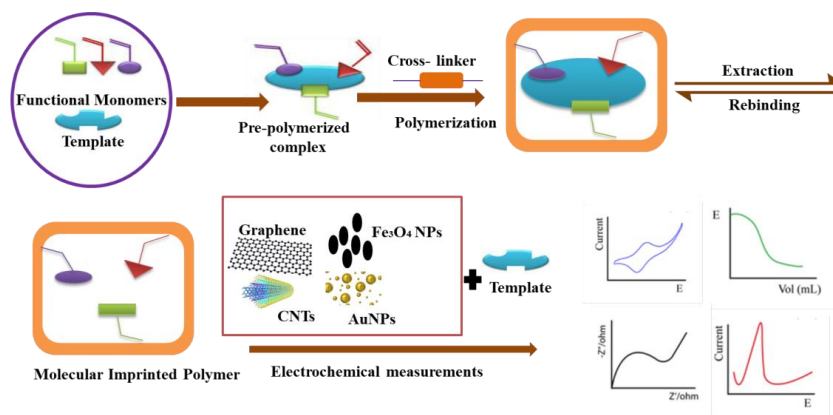


Fig. (2). Schematic representation of nanostructured MIP fabrication as electrochemical sensor.

NANOSTRUCTURED MOLECULARLY IMPRINTED POLYMER (MIP)

The process of molecular imprinting can generate complementary recognition sites with high selectivity and specificity of template molecule in a crosslinked polymeric matrix. The efficacy of MIP technique is directly dependent upon the bonding nature of template and functional monomer pre-polymerized complex [10 - 13], nature of the imprinted materials [14 - 17] and the flexibility of the polymeric network [18, 19]. Though the traditional imprinting protocol is effective and simple, it possesses major difficulties like (i) highly crosslinked bulky imprinted cavities are formed which exhibit uneven size and shape, (ii) the removal of target molecules from the bulk part of the polymeric network is somewhat challenging which diminishes the capacity of rebinding analytes [19, 20], (iii) rate of the binding reaction reduces [21, 22], (iv) high rigid polymeric

Multi-walled Carbon Nanotubes Based Molecular Imprinted Polymers for Sensing

Archana Aravind¹, Anu Rose Chacko¹ and Beena Mathew^{1,*}

¹ School of Chemical Sciences, Mahatma Gandhi University, Kottayam, Kerala 686560, India

Abstract: Molecular imprinting technique (MIT) has been commonly and effectively used to prepare polymers which have some unique features like structure predictability, recognition specificity, low cost, remarkable robustness, physical stability, and application universality compared to other reported recognition systems. The application of molecular imprinting technology to the surface of carbon nanotubes, resulting in MWCNT-MIPs. Surface imprinting polymers have a high mass transfer power, high sensitivity, and a fast response time since the imprinting sites are on or near the surface of the substrates. These materials spread out their applications in many fields such as biomaterials, chemo/biosensors, catalysis, molecular/ionic separation and drug delivery. Even though there are enormous applications of such nanomaterials in various fields, this chapter proceeds with the sensing applications.

Keywords: Multi-walled carbon nanotubes, Molecularly imprinted polymers, Sensor.

INTRODUCTION

The molecular recognition method has an infinite number of applications in nature; it is used in enzymatic catalysis, antibody-antigen recognition, cell communication, and other biological processes [1]. Molecularly imprinted polymers (MIPs) have been shown to be the most promising synthetic materials carrying selective molecular recognition sites, as they mimic the mechanism of biomolecular recognition while having high stability, ease of preparation, and low cost [2]. In the 1970s, Wulff *et al.* used a covalent approach to implement Molecular Imprinting Technology (MIT), which includes the creation of binding sites in a synthetic polymer matrix that are functionally and structurally complementary to the 'substrate' molecule. The analytes (or templates) were first compounded with the monomers, which were then polymerized [3].

* Corresponding Author Beena Mathew: School of Chemical Sciences, Mahatma Gandhi University, Kottayam, Kerala 686560, India; Email: beenamses@gmail.com

The polymers are hollowed out to create cavities that are sterically and chemically complementary to the templates. When the templates are removed, the precise binding cavities will tell the difference between the templates and their analogues [4].

In the molecular imprinting approach, functional and crosslinking monomers are copolymerized in the presence of template molecule (analyte) and a three-dimensional polymer network is created. The functional monomers primarily form a complex with the molecule to be imprinted and polymerized. The functional monomers are assumed to be in position by a highly crosslinked polymeric structure. The elimination of the template molecule discloses binding sites that are complementary in size and shape to the template. So a molecular memory is left into the polymer matrix which is now capable of rebinding the template with high specificity. The schematic representation of the general imprinting procedure is shown in Fig. (1).

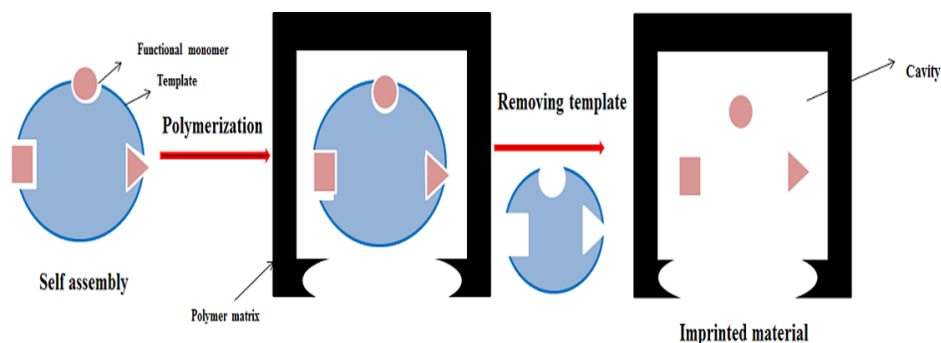


Fig. (1). Schematic representation of general procedure of imprinting.

The molecular imprinting methods can be divided into four categories according to the mechanism of interaction between the monomers and templates: (1) pre-organized approach (covalent bonding), (2) self-assembly approach (non-covalent interactions), (3) hybrid molecularly imprinting method, and (4) metal-chelating method [5]. They were confined to a small range of use due to the restrictive choices of templates. While conventionally prepared bulk MIPs have high selectivity, coarse post-treatment causes some inborn defects. Restricted mass transferability, another major flaw in bulk MIPs, has become a major problem in the macromolecule imprinting or biosensor fields [6, 7].

FACTORS AFFECTING THE IMPRINTING PROCESS

The methodology of molecular imprinting is effortless. The design of the imprinting process is very complicated since it depends upon the number of

variables such as templates, functional monomers, crosslinkers, solvents, initiators, temperature and pressure. Brief descriptions of some factors are described below.

Template

The template molecule should possess at least one functional group through which it can connect to the functional monomer as well as a part of the distinct three-dimensional structure. The kind of functional group directs the imprinting approach that can be utilized. Not all templates will form a covalent bond with a functional monomer that is effortlessly cleaved. On the other hand, the number of functional groups has an effect on the affinity of the template for the molecularly imprinted polymer.

As the number of interactions between the template and functional monomer increases, the affinity with which the molecularly imprinted polymer rebinds the template molecule also increases. Conversely, it also raises the non-specific binding of the template to the polymer. The removal of the template after polymerization is essential to reveal the imprinted cavities. If the template molecule remains, these can disclose while performing analysis on the polymers [8]. The compounds such as drugs, amino acids, proteins, carbohydrates, hormones, pesticides, and co-enzymes have been effectively used for the synthesis of the discerning recognition matrices. A few of the templates used in the molecular imprinting process are shown in Fig. (2).

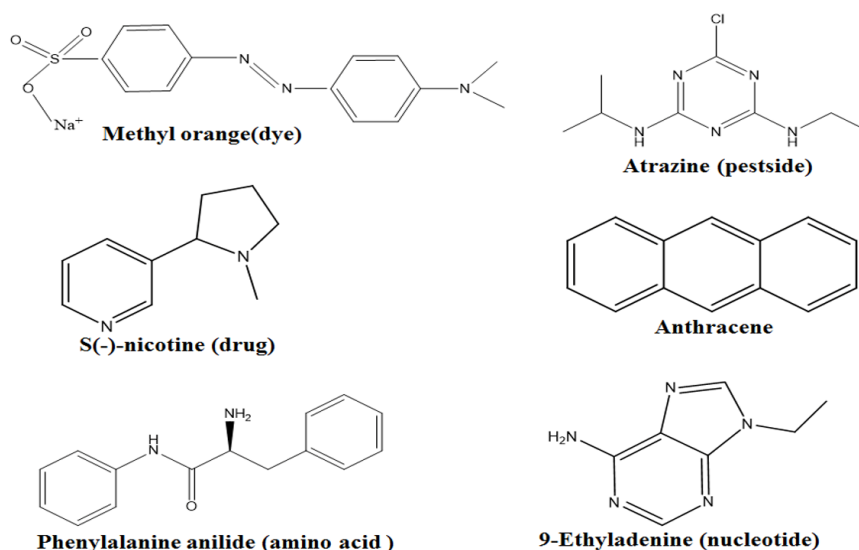


Fig. (2). Commonly used templates in imprinting process.

Molecularly Imprinted Polymer (MIP) Nanocomposites–based Sensors

Juhi Srivastava¹ and Meenakshi Singh^{1,*}

¹ Department of Chemistry, MMV, Banaras Hindu University, Varanasi, U.P., India

Abstract: Molecular recognition in biological systems drives and controls all the activities related to ‘Life.’ The accuracy, specificity, and selectivity of biological elements led to their use as biosensors for ‘sensing’. An ideal molecular recognition agent must comprise a stable, reproducible, reusable, robust, specific and preferably nonbiological material. Molecular imprinting has almost all attributes that qualify it to be an ‘ideal’ recognition agent. As a surrogate to biological receptors, synthetic MIPs have shown aspiring futuristic tools. Next-generation sensors could be visualized by a collaboration of synthetic polymers (MIPs) with innovative technologies replacing biosensors. Over the period of the last three decades, the introduction of specific binding sites within synthetic polymers by utilizing target-directed cross linking of functional monomers has attracted substantial consideration for the sake of the formation of molecularly imprinted polymer (MIP) based sensors. MIP seems like a reasonable tool for the creation of various sensors with broad practical relevance.

This chapter outlines the sensors prepared on nanocomposite as an imprinting matrix. Strategic planning in synthesizing these novel matrices is praiseworthy. Hopefully, such measures would bring down the economic burden by devising cheaper sensing tools, especially diagnostic kits in such pandemic times.

Keywords: Chitosan nanoparticle, Graphene, Molecularly imprinted polymer, Nanocomposite, Sensor, Starch Nanoparticle.

INTRODUCTION

In the last decade, engineered nanoparticles with diverse functionality and purpose received utmost attention from almost all affiliations of science. As the scientists worked upon them and came with novel feats and achievements regarding their design, innovative ideas popped up while working with them. Conventionally, metallic nanoparticles are favourites, followed by bimetallic, magnetic, organic, polymeric and biopolymeric particles, which have been

* **Corresponding author Meenakshi Singh:** Department of Chemistry, MMV, Banaras Hindu University, Varanasi, U.P., India; Email: meenakshi@bhu.ac.in

synthesized and explored well. Their exploration fetched varieties of characteristics, some of them unique to their class only. To employ them optimally for diverse needs, hybrid nanocomposites were also tested successfully. The typical definition of a composite is “a material which is produced from two or more constituent materials having dissimilar chemical or physical properties, merged to create a material with properties, unlike the individual elements.” So, for visualizing these ‘new’ characteristics, many attempts are made in almost all sections of science to prepare nanocomposites. They offer new applications with these hybrid materials expecting synergy between them.

In this chapter, an attempt is made to summarize the scope of molecularly imprinted polymers (MIP) – composites in sensing devices, especially to serve the healthcare sector of our society. To achieve this purpose, accurate detection and quantification of analytes are required in clinical analysis, biological and chemical security, environmental protection and food safety. These analyses involve a huge economic burden on our society [1 - 5]. Efforts are being made to reduce this burden by designing and fabricating cost-effective arrays of sensors, sensing devices and also smart devices. Tailoring such devices needs a combined effort of interdisciplinary research and their application in ‘real’ samples.

Sensors or sensing devices, whether chemosensors or biosensors, comprise of two main units; recognition unit and transduction unit. The recognition unit predicts selectivity *via* chemical interactions, whereas the transduction unit transduces these interactions into analytical signals [6, 7]. Among the approaches adopted for chemosensors, molecular imprinting is one of the most aspiring and realistic approaches for fabricating synthetic receptors, which can be used as a recognition unit in chemosensors [8 - 12]. Generally, MIPs in thin-film form with suitable transducers are used for the fabrication of such sensing devices for different analytes [8, 10, 13]. Commonly used transducers for the fabrication of such chemosensors are voltammetric, amperometric, piezoelectrogravimetric, electrochemical impedance spectroscopy (EIS) and surface plasmon resonance (SPR) spectroscopic techniques.

MIPs are commonly used ‘recognition sites’ for chemosensing devices. Molecular imprinting has come up as an almost foolproof method for generating artificial receptors or, in other words, as chemical sensors competing with the biological analogues - biosensors. The molecular imprinting approach is able to form cavities in polymer matrices, which are the exact mirror images of analytes; thus, it is able to induce the movement of analyte molecules only towards these imprinted cavities, rather than their analogues or other structurally similar molecules. These polymers have an affinity for the original target molecules and have been used for various purposes, such as chemical separation, molecular

sensors, biosensors, bioseparation and drug delivery, *etc.* [14, 15]. MIPs are known for their stability under ambient conditions, whether mechanical and/or chemical stability. Their purposive characteristics of selectivity, stability under ambient conditions, sturdiness and steadiness, reproducibility, reusability and specificity are instrumental for their wide applications in fields of biosensors, bioseparation, chromatography, molecular receptor and drug delivery, *etc.*

MIP synthesis includes the arrangement of a complex with template and useful monomers during self-organization, either by covalent or non-covalent bonds pursued by polymerization with cross linkers in an appropriate solvent (porogen). Upon complete extraction of template molecules, the specific imprints are made in the polymeric grid corresponding to format fit as a fiddle and contain properly arranged recognizing elements valuable in rebinding and consequently, after extraction of the analyte, the subsequent polymer can rebind analyte with high inclination and specificity. Rebinding of the template with imprinted polymer is made conceivable by the formation of shape-corresponding cavities inside the polymeric system. Fundamentally, an atomic memory is imprinted in polymer, or, in other words, the template is rebound specifically. A pictorial presentation of the development of MIP is shown in Fig. (1).

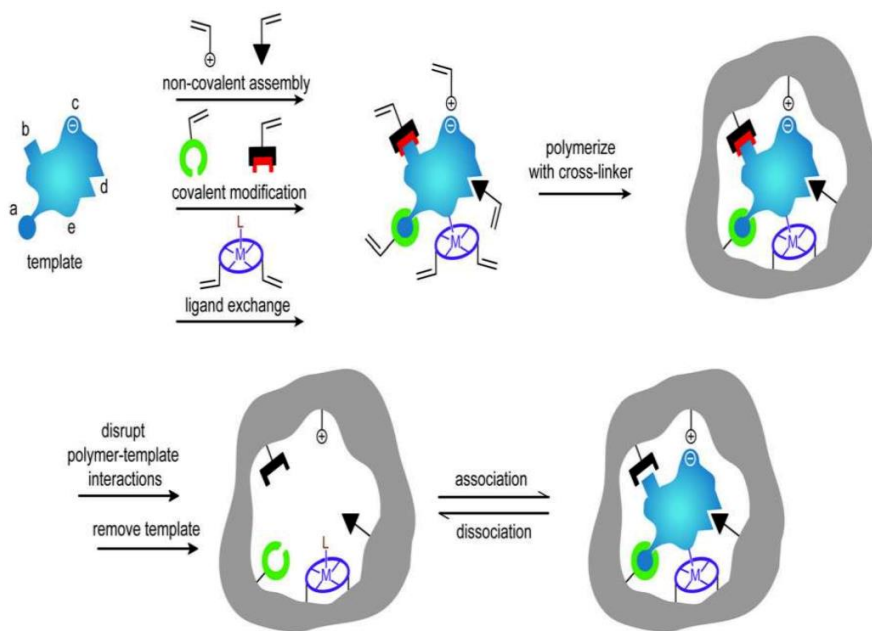


Fig. (1). Schematic representation for the synthesis of MIP [12].

Advancement in Nanocomposites for Explosive Sensing

V Dhinakaran^{1,*}, M Varsha Shree¹ and M Swapna Sai¹

¹ Centre for Applied Research, Chennai Institute of Technology, Chennai-600069, India

Abstract: In the research of nanocomposite, its selective and sensitive explosive detection is very critical. Due to a series of causes, including the vast collection of materials that can be used as explosives, lack of easy to detect signatures and broad ranges of means to deploy such weapons, and lack of affordable sensors of great sensibility and selectivity, explosive trace detection has been exceedingly difficult and costly. The fight against explosives needs a high resilience and selectivity coupled with the potential to lower deployment costs of sensors using mass processing. Nanosensors should fulfill the criteria of an efficient framework for explosive trace detection. In this research work, we confer about the ability of nanosensors to detect trace explosions, based upon high sensitivity and selectivity on nano mechanical sensors for both receiver and receptor-free sensing, which can be used because of their versatility and are incorporated into a multimodal sensor system.

Keywords: Explosives, Nanocomposites, Nanosensors, Polymeric Nanocomposites, Nitramines.

INTRODUCTION

Explosives usually need to be tracked through the processing or examination of vapours or particulate samples using a responsive sensor device. There are actually many approaches used for trace explosives identification [1]. Most generally, the spectrometry of ion mobility (IMS), mass spectrometry (MS) and gas chromatography (GC) are accompanied by responsive sensors identification. However, most of these instruments are very heavy, pricey and take time [2]. Owing to these drawbacks, systems like airports and government buildings are being sparsely built in sensitive areas. Complications not only occur at airports (where sensing and detecting environments are relatively controllable) but at practically unregulated points of entry to public areas, transit networks, infrastructures, or road networks with erratic cars and pedestrian traffic [3]. Ther-

* Corresponding author V. Dhinakaran, Centre for Applied Research, Chennai, Institute of Technology, Chennai-600069, India; Email: dhinakaranv@citchennai.net

efore, protection against explosives can only be accomplished by the use of miniaturized sensors in mass deployment that are adaptive, selective, and economical enough to manufacture in mass. For detection, it is important to detect chemicals using a selective agent and signal transducer. The signal is then sent to an electronic computer for reporting. High sensitivity, selectivity, reversibility and function in real time are the main features of sensors for trace level explosive detection [4]. Excellent sensitivity and low detection limits are needed for trace explosive detection due to the comparatively small number of molecules and vapour pressures. If false-positive rates are to be adequate, high selection is essential. To allow for continuous operation, the sensor should be reversible at room temperature. This sensor must also be easily detected and regenerated to function efficiently [5]. Finally, due to the vast number of terrorism attacks, including bombs, adequate sensors should be deployed. These rigid specifications cannot be met by currently available sensor platforms. Nevertheless, nanoscience-based sensors have a straightforward way to build trace explosive sensors that follow those requirements [6].

EXPLOSIVES

Explosives are chemical compounds that can immediately disperse heat and pressure. Explosives are known to be medium or high explosives because of their burn rates. The propellant, black powder, *etc.*, are low explosive burning (centimetres per second) [7]. In addition, high explosives, which rise at kilometres per second, are divided according to stability into main and secondary explosives. Core explosives such as plumb azides are very vulnerable to environmental stimuli, such as explosives and vibrations, thermal or electrical. Secondary explosives, such as 2,4,6 trinitrotoluene (TNT), hexogen (RDX), and other large explosives, are extremely stable [8].

Various typical explosives are organic compounds and can be classified according to their chemistry in six major classes:

- Acid salts (*e.g.*, Ammonium nitrate)
- Aliphatic nitro compound (*e.g.*, Nitromethane, Hydrazine nitrate)
- Nitramines or nitrosamines (*e.g.*, Octogen (HMX) or RDX)
- Nitrate esters (*e.g.*, Pentrite (PETN), Ethylene glycol dinitrate (EDGN), Nitroguanidine (NQ) and Nitroglycerine)
- Nitroaromatic compounds (*e.g.*, TNT, Dinitrobenzene (DNB), Hexanitrostilbene, Picric acid)
- Organic peroxides (*e.g.*, Triacetone triperoxide (TATP) and Hexamethylene triperoxide diamine (HMTD)).

Last-group explosives called household-produced explosive materials (HME), owing to the presence of volatile organic compounds (VOC), such as acetone, are at very high vapour pressure. HME-based terrorism has recently been quickly extended due to the ease with which it can be made [9]. However, HMEs are highly unstable and require careful treatment in order to prevent detonation. Most typical explosives have a very low ambient temperature vapour pressure. These molecules are very rigid and adsorbed very quickly by surfaces due to low vapour pressure. It is worth noting that explosive vapour pressure rises with temperature rapidly [10]. During sample heating, explosive heating steams quickly condense in colder locations. The adsorption will be stronger on elevated surfaces, such as metal oxides, *etc.*

Materials like polymers, fibres, *etc.*, are compared to low-power surfaces. Sometimes, since explosive molecules are sticky on surfaces at room temperature, they can be condensed into sensor structures by supply chains. Trace sampling of these families of very low vapour explosives is often difficult since the sampled volume includes such a minimal number of molecules [11].

II-ELECTRON RICH LUMINESCENT POLYMERIC NANOCOMPOSITES

In the modern scientific world, sensitive identification of trace-level explosive quantities is considered imperative because of safety issues and ecological contamination problems. By a single, phase-free radical polymerisation reaction, impregnated polymer vinyl alcohol and graft polymers for the detection of fluorescence compounds with carbon and silver nanoparticles are synthesised. The key function of the leading nanoparticles is to increase the luminous polymer's β -electron density [12]. The highly selective, ultrasensitive identification of nitro-aromatic explosives, based on a fluorescence scan process, by these electron-rich nanocomposites is successfully performed, as shown in Fig. (1) [13]. The quenching ability of carbon and silver nano-composites for strongly electron-deficient picric acid (PA) and 2, 4, 6-trinitrotoluene (TNT) was found to be 99 and 95%, respectively. Mechanical research has been studied in-depth and the combination of Forster Resonant Energy Transfer (FRET) and Phenogenic Electron Transfer (PET) in most analytes, like picric acid, results in fluorescence quenching. The analysis has been carried out in detail. On the other hand, PET is the only explanation behind fluorescence quenching in analytes such as 2, 4, 6-trinitrotoluen, 1,4-dinitrobenzene and nitrobenzene. Carbon and silver nanocomposite detection limits are located at the nM level [14]. Fast synthesis and scalable development in bulk volumes ensure their wide future application for real-time experience in defence and the industrial sectors for real-time explosive detection. Fluorescence Quenching Phenomena decides the excellent sensing

Nanocomposite Materials Interface for Heavy Metal Ions Detection

Prashanth Shivappa Adarakatti¹ and S Ashoka^{2,*}

¹ Department of Chemistry, SVM Arts, Science and Commerce College, ILKAL – 587125, India

² School of Science, Dayananda Sagar University, Kudlu Gate, Bengaluru – 560068, India

Abstract: The present chapter provides an overview of the sources, consequences, and quantification of heavy metal ions (HMIs) present in the water sample. Heavy metal ions are recognized as one of the major water pollutants. Long-term consumption of HMIs causes serious health hazards and is also a threat to the ecosystem. In this regard, the synthesis and use of nanocomposites for the selective quantification of HMIs have been discussed in detail.

Keywords: Electrochemical sensor, Heavy metal ions, Modified electrodes, Nanocomposite.

INTRODUCTION

The effluent released by several anthropogenic activities majorly contaminate natural resources [1, 2]. Long-term exposure to these HMIs causes adverse effects on living organisms. Consequently, even exposure to trace level concentrations of toxic heavy metal ions can lead to long-term disorders [3]. Therefore, the accurate and fast detection of HMIs is becoming a challenging issue for analytical chemists. Numerous detection methods have been proposed in the literature to monitor HMIs present in the water and food samples accurately [4, 4b, 5 - 7, 7b].

Chemically modified electrodes (CMEs) have been recognized as potential candidates in developing reliable electrodes to quantify HMIs. In the year 1970, Royce Murray first introduced the concept of CMEs where the SnO₂ electrode has been modified with amines [8].

The CMEs are formed when the modifier is covalently or non-covalently anchored to the surface of the substrate material with the help of a binder. Anchoring of the modifier molecule on the surface of the carbon substrate by

* Corresponding author Ashoka S: School of Science, Dayananda Sagar University, Kudlu Gate, Bengaluru, India; E-mail: ashok022@gmail.com

specific functional groups provides an excellent pathway for the interaction with the target analyte, such as HMIs.

IMPORTANCE OF CHEMICAL MODIFICATION

The electrochemical reactions occur at the interface of electrode and electrolyte solution. Hence, the surface structure of the electrode at the interface plays a vital role in promoting electrode kinetics [9]. The commonly used electrodes are carbon-based substrate materials. The carbon-based electrodes possess low background current, broad potential range, and chemical inertness and have low cost. These features made them a suitable candidate for various sensing applications [10]. However, these electrodes show some limitations over modified carbon electrodes in terms of sensitivity and selectivity. Hence, the modification of carbon-based electrodes with suitable modifiers having functional groups indeed enhances the electro-analytical signal intensity [11]. CMEs have been extensively employed to quantify HMIs owing to their tailor-made properties, such as selectivity and sensitivity, towards the target analytes. It can be illustrated with an example involving the electrochemical quantification of lead ions using graphene oxide (GO) modified electrode [12]. The presence of GO on the electrode surface will reduce the overpotential required for the redox process of lead ions. Similarly, mercury quantification can be achieved by modifying the carbon-based electrodes with thiol functional groups [13]. The modification of the substrate with suitable modifier molecules with specific functional groups can be achieved using several approaches like physical adsorption, chemical adsorption, covalent attachment, electrochemical, ball milling, and microwave-assisted covalent modification procedures [14, 15]. The carbon substrates like graphite, glassy carbon, carbon nanotubes, and screen-printed electrodes (SPEs) have been modified with specific modifier molecules.

TYPES OF ELECTRODES

Glassy Carbon Electrode (GCE)

The glassy carbon electrode is fabricated by using controlled charring of polymeric resin (phenol/formaldehyde or polyacrylonitrile) at a high temperature of about 1000 to 3000 °C. Glassy carbon has a ribbon-like structure with graphitic sheets that are cross linked (Fig. 1). Hence, it is stronger and more robust than graphite [16].

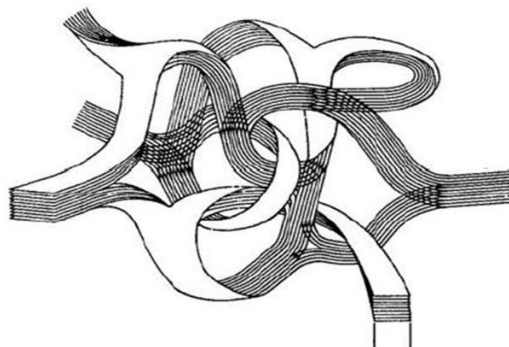


Fig. (1). Structure of glassy carbon [17].

The GCE has been extensively used in electroanalytical chemistry owing to its outstanding mechanical and conducting properties together with broad working potential. Generally, the surface of GCE is pretreated with alumina slurry of different particle sizes to get improved analytical response by means of enhanced electron transfer on the surface of GCE [18, 19]. This GCE is modified with a suitable modifier to enhance analytical signals.

Carbon Paste Electrode (CPE)

Carbon paste electrode is generally used as a working electrode for the measurement of target analytes in electro-analytical chemistry. It mainly consists of graphite powder and a binder like paraffin oil, Nujol, bromonaphthalene, and silicone grease. These electrodes offer easy surface renewability, low cost, and low background current. The fabrication of working electrodes can be made by mixing modifier molecules along with graphite powder in the presence of a binder. The potential drawback of this electrode includes continuous leaching of the modifier molecule from the surface of CPE along with the binder, which hinders electrocatalytic reaction.

Screen-printed Electrodes (SPEs)

Screen-printed electrodes (SPEs) have gained considerable attention in the electrochemical community in recent years [5]. The SPE contains all three electrodes (working, counter, and reference electrodes) in one strip and offers easy fabrication, on-site detection, low cost, broad potential range, and low sample volume. Being a disposable sensor, it requires no polishing or smoothening, but it can often be seen in the conventional solid electrodes [20]. The analytical signal could be improved by modifying suitable functional groups upon the carbon-based SPEs.

Nanocomposites as Electrochemical Sensing Platforms for Glucose Detection

Prashanth Shivappa Adarakatti^{1,*}, Suma B. Patri² and S Ashoka³

¹ Department of Chemistry, SVM Arts, Science, and Commerce College, ILKAL – 587125, India

² Department of Chemistry, Bangalore University, Central College Campus, Bengaluru – 560056, India

³ Department of Chemistry, School of Engineering, Dayananda Sagar University, Bengaluru –560068, India

Abstract: This chapter covers the advances in the development of a variety of composite materials ranging from noble metal/metal oxide nanoparticles, carbon composites, polymer composites and metal-organic framework-based composite materials specific to glucose. The advantages of nanocomposites as ‘electrode materials’ have been highlighted. The utilization of above-mentioned nanocomposites in non-enzymatic glucose sensors and their mechanism has been discussed. Further, special attention has been given to the MOF-based nanocomposites, which elaborates the applications of MOF-based materials in biosensing in recent years. This chapter gives an overall view of various nanocomposites used as electrochemical glucose sensors and opens up a new trend in materials science research to engineer advanced functional materials with tailor-made properties to suit relevant real-time applications within electroanalysis.

Keywords: Diabetes , Electrochemical Sensor , Glucose sensor, Nanocomposite , ModifiedElectrodes .

INTRODUCTION

Diabetes mellitus is a group of metabolic diseases characterized by variation in the blood glucose level, which results from defects in insulin secretion and its action. This increased glucose level in blood is associated with severe health effects like long-term damage to tissues, failure, and dysfunction of various organs, especially the nerves, kidneys, eyes, heart, and blood vessels [1]. Hence, regular monitoring of glucose levels in the blood has become mandatory in a large sector of people.

* Corresponding Author P. S. Adarakatti<Department of Chemistry, SVM Arts, Science and Commerce College, ILKAL – 587125, India; Email: prashanthsa143@gmail.com

Since the discovery of one-shot disposable biosensors, the development of glucose sensors have grown enormously in the past few decades. The non-enzymatic and enzymatic-based probes are two important protocols [2], which are used for glucose quantification. Fig. 1. shows the schematic representation of the classification of various types of glucose sensors. The development of the first enzyme-based glucose sensor led to the evolution of various glucose sensors to date.

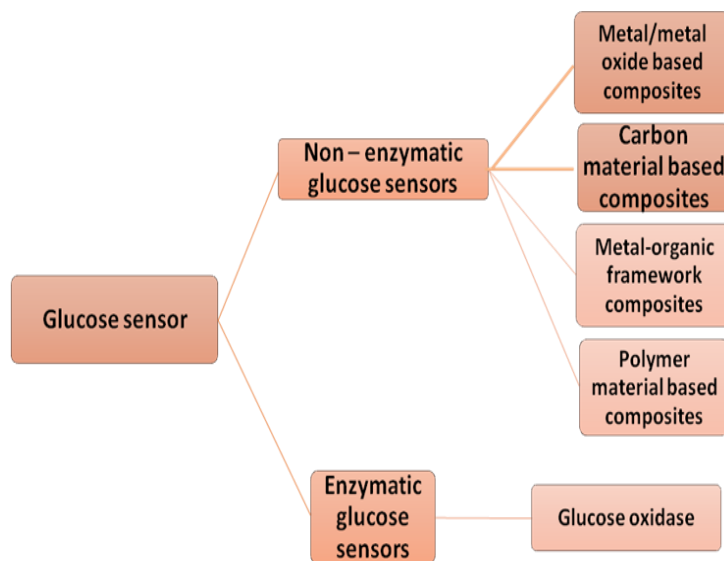


Fig. (1). Schematic representation of the classification of various types of glucose sensors.

NANOCOMPOSITES FOR ENZYMATIC GLUCOSE SENSORS

Enzymatic glucose sensors have gained much attention ever since the beginning of the first generation of glucose sensors by Clark and Lyons in 1962 [3]. The most important component of any blood glucose monitoring device is the detection technology or the sensing mechanism that quantifies the concentration of glucose selectively and sensitively. The detection mechanism evolved from first-generation blood glucose monitoring systems employs oxygen as the electron acceptor and determines the glucose concentration either by consuming the oxygen or by the liberation of hydrogen peroxide, whereas redox dyes and mediators have been utilized in the second generation glucose sensors. Furthermore, sensitive glucose sensors were commercialized, where the direct electron transfer has taken place upon the electrode interface. These types of sensors demonstrated better selectivity and anti-interfering properties in blood glucose monitoring in real samples [4].

Due to the high selectivity and sensitivity of enzymes towards glucose, glucose oxidases (GOxs) and glucose dehydrogenases (GDHs) are the two types of oxidoreductases utilized for glucose monitoring. Other types of enzymes immobilized are Pyrroloquinoline quinone (PQQ), Flavin-Adenine-Dinucleotide (FAD)-dependent Glucose dehydrogenases, Nicotine Adenine Dinucleotide (NADP) (Phosphate)-dependent Glucose Dehydrogenase, which varies immensely in terms of structure, substrate specificity, origin, primary electron acceptor and acceptable final electron acceptor. However, blood glucose monitors using GDH-PQQ can be affected by maltose or galactose, xylose, which is another form of sugar with similar activity, and interferes in glucose measurement at GDH-FAD modified electrodes [5].

The native GOx is incapable of transferring electrons to the electrode interface. Hence, huge works have been done to focus on connecting these enzyme's redox center to the electrode surface and non-biological electrode materials have been attracting significant attention as a substitute to biosensors. Most importantly, non-enzymatic sensors are free from oxygen limitations. Most of the enzyme electrodes with low or high oxygen concentrations may modify the sensor signals, causing divergence from analytical signals measured in the normal range of oxygen concentration. The main approach is to make use of various conducting molecular wires such as metal complex-linked polymers and macromolecules, synthetic co-factor derivatives and carbon nanomaterials. Alternatively, compositing carbon nanomaterials and metal oxide NPs can prevent agglomerating and restacking of metal NPs by steric hindrance and electrostatic attractions. Likewise, these composites can increase the electron transport rates between the electrolyte and modified electrode materials fabricated in an electrochemical device. In recent years, graphene, carbon nanotubes, conducting polymers, metal oxides/sulphides and noble nanoparticles and nanoparticles of transition metals were utilized for the measurement of the enzymatic glucose sensor. For instance, the immobilization of glucose oxidase on gold nanoparticles (AuNPs)-functionalized ZnO nanostructure on a glassy carbon electrode (GCE) is a facile approach to construct third-generation glucose sensors [6]. The non-enzymatic glucose sensing approach is a fascinating area that offers a number of opportunities to material chemists.

NANOCOMPOSITES FOR NON-ENZYMATIC GLUCOSE SENSORS

In recent times, enzymatic glucose sensors have been incorporated with nanomaterials to enhance conductivity, surface area, and electron transfer rates. These nanomaterials include the nanoparticles of noble and transition metals, the nanostructured metal-oxides or metal-sulfides, conductive polymers, carbon nanotubes, and graphene.

Tailored Nanocomposites for Hydrazine Electrochemical Sensors

A. Rebekah¹, G. Srividhya¹ and N. Ponpandian^{1,*}

¹ Department of Nanoscience and Technology, Bharathiar University, Coimbatore 641046, India

Abstract: In the field of nanotechnology, nanocomposites have gained increasing and significant attention owing to their unique physico-chemical characteristics. This outstanding characteristic makes them a suitable candidate in various fields of application, such as electronics, sensors, biotechnology and catalysis. The development of nanocomposites has proven to be a basis for the development of accurate electrochemical sensors with low limit of detection, high sensitivity and selectivity. The high performance electrochemical sensors have found their way in various application fields, such as biomedical, analysis of food products and other environmental pollutants present in the atmosphere. In this chapter, we present a survey of the application of various tailored nanocomposites as sensing platforms for hydrazine. Particularly, electrochemical sensors based on carbon-based nanomaterials, metallic nanomaterials, and related nanocomposites are given special attention.

Keywords: Composites, Hydrazine, Hybrid materials, Sensors, Tailored nanocomposites.

INTRODUCTION

The improved technological development in the past few decades has led to a colossal growth of industries.

This was combined with the economical growth of urban and semi-urban populations that has caused an upsurge in the productivity and consumption of industrial products in a huge scale. Most industrial products like automobiles, hardware, paints, domestic appliances, clothing and cosmetics are produced at the expense of many inorganic and organic chemical, some of which are highly dangerous for human health and environmental sustainability. Hence, screening of industrial surroundings, effluent and wastes are highly important to protect our environment by preventing hazardous chemicals from contaminating the biosys-

* Corresponding author N. Ponpandian: Department of Nanoscience and Technology, Bharathiar University, Coimbatore 641 046, India; E-mail: ponpandian@buc.edu.in

tems. Owing to the aforementioned reason, the development of gas and vapour sensors has received a massive research interest from the past two decades. A sensor is a device that can detect a physical component and produce a user understandable output signal corresponding to the quantity of the target component. There are different types of sensors that detect different physical entities by using different analytical methods. For example, based on the sensor target, they are classified into gas sensors, vapour sensors, temperature sensors, stress sensors, biosensors and so on. Similarly, based on the principle of operation, they are classified into chemi-resistive sensors, electrochemical sensors, optical sensors, piezoelectric sensors, chemi-luminescence sensors and so on. Among them, gas and vapour sensors functioning on the basis of chemi-resistive principle, optical and electrochemical techniques are widely developed to protect the working environment from various types of toxic and flammable gases like carbon monoxide, carbon di-oxide, methane and vapours of volatile organic compounds like ethanol, methanol, ammonia, chloroform, di- and trimethylamine, *etc.*

Hydrazine (N_2H_4) is an organic compound with two amino groups and is also dubbed as diamidogen. At normal conditions, it is a colourless liquid with a faint ammonia smell. It has strong basic nature and is a powerful reducing agent and so often used as an anti-oxidant, oxygen scavenger and corrosion inhibitor. It has high enthalpy of combustion and hence is used as a propellant in space vehicles. Apart from the aforementioned applications, hydrazine is frequently used as a chemical reactant in different industries like textile dyeing, pharmaceuticals, pesticides and fertilizers. Despite its numerous applications, hydrazine is highly toxic and inhaling hydrazine may cause a range of health problems. The health risk of exposure to hydrazine fumes, include mild symptoms like irritation in eyes, nose, and throat, dizziness, headache, nausea and also severe health deterioration like the damage of internal organs like liver, kidney and the central nervous system. Moreover, hydrazine easily dissolves in water and there is a high risk of drinking water contamination with hydrazine in industrial surroundings. The US Environmental Protection Agency identified hydrazine as a potential carcinogen and has defined a threshold value below 10 ppb in drinking water to prevent health complications.

Hence, it is a matter of urgency and necessary to design an efficient sensor that can detect even the small traces of hydrazine in drinking water and other consumable industrial products. Considering this, many researchers have aimed to fabricate sensors that can effectively detect hydrazine. Techniques like chromatography, chemi-luminescence, fluorescence and voltammetry have been employed to fabricate hydrazine sensors. Among them, the electrochemical sensors are highly desirable due to low cost, ease of fabrication, lower limit of

detection (LOD) and prospective commercialization. Nanocomposites possess the advantage and synergistic effect of the constituent nanomaterials and hence, nanocomposites based devices show better performance than those based on simple devices. Tailoring of composites for designing the hybrid materials of carbon derivatives and metal oxides is very important in the view of electrochemical based sensors. In this chapter, electrochemical sensors based on tailored nanocomposites for hydrazine detection are discussed.

COPPER BASED NANOCOMPOSITES FOR HYDRAZINE SENSOR

Zhao *et al.*, synthesized cupric oxide functionalized with carbon nanotube and reduced graphene oxide (CuO/CNTs-rGO) nanocomposites for sensitive hydrazine sensors. The nanocomposite exhibited a sensitivity of $4.28 \mu\text{A} \cdot \mu\text{M}^{-1} \cdot \text{cm}^{-2}$, a linear range of 1.2–430 μM , and detection limit of 0.2 μM towards oxidation of hydrazine. The efficiency of the sensor was attributed to the higher electron conductivity and large specific surface area of the hybrid structure, which would have enabled a promising nanocomposite for the effective determination of hydrazine. The fabricated CuO/CNTs-rGO/GCE sensor also retained about 90.5% of initial sensitivity even after 20 days of duration, demonstrating that the sensor possesses good stability [1]. Guo *et al.* demonstrated the electrochemical performance towards oxidation of hydrazine using hollow CuO nanospheres prepared using reduction reaction of copper ions on porous Si nanowires followed by calcination for uniformly anchoring on their surfaces. The sensor showed a better electrochemical performance with a rapid response time of below 3 s, a linear range from 1 to 5 mM, and a detection limit down of 0.25 mM [2]. Ramachandran *et al.*, fabricated graphene supported CuO nanorods for sensitive determination of hydrazine and the nanocomposite revealed a detection limit of 9.8 nM, linear response range from 0.1 to 400 μM and a sensitivity of $3.87 \mu\text{A}/\mu\text{M}/\text{cm}^2$. Moreover, the sensor disclosed good selectivity, reproducibility and long durability towards oxidation of hydrazine [3]. Teymoori *et al.* investigated the electrochemical oxidation of hydrazine using copper oxide nanoparticles/ionic liquid/carbon paste electrode (CuO NPs/IL/CPE) nanocomposite. The modified electrode exhibited sensitivity for hydrazine oxidation which has two linear ranges 0.1–15 and 15–150 μM , found to be 1.4741 and 0.4562 $\mu\text{A}/\mu\text{M}$. The stability of the modified electrode was investigated after it was stored at room temperature for 3 weeks. It was observed that the oxidation currents showed less than 2.35% decrease relative to the initial responses for hydrazine [4]. Zang *et al.* demonstrated hydrazine electrochemical sensor by painting Cu/Cu₂O@carbon on a glassy carbon electrode and immobilized using Nafion. The Cu/Cu₂O@carbon modified GCE was evaluated using cyclic voltammograms and amperometry technique, which exhibited high sensitivity of $2.37 \mu\text{A} \cdot \mu\text{M}^{-1} \cdot \text{cm}^{-2}$, linear concentration ranges from 0.25 to 800 μM , and a low

CHAPTER 13**Optical Detection Of Toxic Cations And Anions By Nanocomposite Materials****D. Amilan Jose^{1,*}, Nancy Sharma¹ and Srushti Gadiyaram¹**¹ *Department of Chemistry, National Institute of Technology (NIT) Kurukshetra, Kurukshetra-136119, Haryana, India*

Abstract: Nanocomposite materials have appeared as appropriate replacements to overcome the limitations of microcomposites and simple nanomaterials. There is an upsurge of interest in nanocomposite materials due to the significant applications and all the research areas related to chemical, physical and biological sciences. Making composites with the combination of nanomaterials could convert them into superior materials for the sensing analytes, such as anions, cations, biomolecules, explosives, toxic gases, food toxins and organic compounds. This chapter emphasizes only on the recent investigations of nanocomposite materials for the detection of toxic metals and anions by optical detection methods, such as fluorescent and colorimetric methods. Appropriately selected examples are discussed in detail.

Keywords: Anion sensor, Cation sensor, Colorimetric, Cyanide detection, Fluorescent, Mercury detection, Nanocomposites.

INTRODUCTION

Nanocomposite (NC) materials are composed of at least one phase having a nanometer size in scale. Nanocomposite materials are anticipated to show novel properties evolving from the mixture of individual phases. They could be considered as suitable substitutes to overcome the limitations of small molecules, nanomaterials, and microcomposites [1].

Currently, polymers are being used to encapsulate nanomaterials to develop new nanocomposites [2]. They have immense applications due to their extraordinary properties depending upon the combination of polymer and nanomaterials embedded within them. Nanocomposites have shown favorable applications in several fields, such as biomedical, food package, optics, electronics, ion separation, chemical/bio-sensor, catalysis, and protective coatings [1, 3, 4].

* **Corresponding Author D. Amilan Jose:** Department of Chemistry, National Institute of Technology (NIT), Kurukshetra-136119, Haryana, India. Email: amilanjoselit@nitkkr.ac.in

In the conventional methods, an optical sensor for anion and cation is prepared by employing receptors with signaling units, such as organic dyes, metal complexes, and nanomaterials. Choosing suitable materials to combine with the conventional sensors could enhance their sensitivity, selectivity, and water compatibility. Subsequently, several nanocomposite materials composed of inorganic nanoparticles, such as gold nanoparticles (AuNPs), MoS₂ nanosheets, Carbon nanotubes (CNTs), Carbon dots (CDs), and Quantum dots (QDs), have been developed. Nanocomposite materials composed of conjugated polymers (CP) and metal organic framework (MOF) with nanoparticles (NPs) have also been explored for various applications [2, 3, 5 - 10]. This chapter aims to describe the recent developments of the nanocomposite materials as optical (colorimetric and fluorescent) sensors to detect toxic cations and anions. Relevant selected crucial examples are discussed in detail.

DETECTION OF TOXIC HEAVY METALS

Mercury Detection

Mercury ions are toxic to the environment. They exist in the environment as inorganic ions within the variety of Hg(II) cations or monomethyl mercury as an alkylated form. Hg²⁺ is also known to bioaccumulate through the biomagnification process. It causes various health problems and diseases in humans and animals. Nanocomposites exhibit distinctive properties as sensing platforms for mercury detection. They have an advantage of simultaneously detecting and separating Hg²⁺ pollutants without causing secondary pollution.

Dithizone-based porous polymers are known for the elimination of cations from wastewater samples. Roya Sedghi *et al.* described a dithizone-based chromogenic nanocomposite probe to detect trace levels of lead and mercury ions in an aqueous solution [11]. In this sensor system, dithizone was anchored on TiO₂/poly (2-V-MBAm-AA) nanocomposite through π - π stacking interactions. The trace levels of ions exhibit a substantial colorimetric response from grey to violet for Hg²⁺ ions and red for Pb²⁺ ions. This nanocomposite sensor has the advantage of easy preparation, rapid response time, being cheap, highly selective and sensitive with LOD as low as 10 ppb.

Another group explored the polymeric TiO₂/poly(acrylamide-co-methylene bisacrylamide) nanocomposites as an ultra-trace level sensor to detect two heavy metals, lead and mercury ions, in the aqueous solution [12]. The LOD of the polymeric nanocomposites for the mercury and lead ions was found to be 1 and 10 μgL^{-1} , respectively. Dithizone is used as an indicator for sensitive sensing of heavy metal ions.

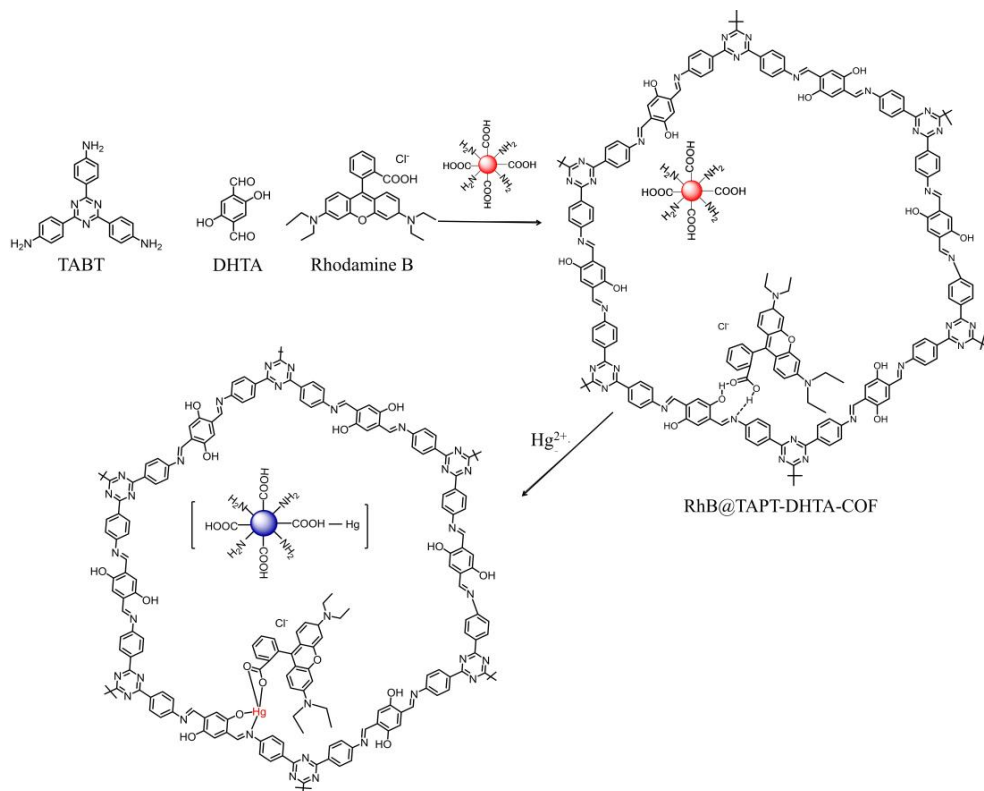


Fig. (1). Hg²⁺ detection mechanism and schematic representation for the synthesis of composite NCDs-RhB@COF.

Acrylamide is used as a monomer and methylene bisacrylamide as the cross-linker to arrange as a polymer on the TiO₂ nanoparticles' surface. The secondary interaction of dithizone with adsorbed target ions plays a significant role in detecting heavy metals on the hydrogel. At pH 6, the extreme adsorption of Pb²⁺ ion, and at pH 9, adsorption of Hg²⁺, were realized. The response time for both metal ions is less than 10 min. A prominent color change from violet to pink was observed for mercury ions and from red to light red for lead ions. The reversibility of the complex was verified by the addition of HNO₃. The selectivity of the metal ions can be derived from the HSAB principle. The adsorption of the target Pb²⁺ and Hg²⁺ is not interfered by other ions at optimum pH and sorption time. However, by changing the pH of the hydrogel, other ions could react with heteroatoms (O and N). The adsorption capacity of the new nanocomposite for Pb²⁺ and Hg²⁺ ions was determined as 96 mg/g and 87 mg/g, respectively. Up to 200 mL of sample volume can be used for the simultaneous recovery of mercury and lead ions by using the hydrogel.

Nanocomposites for Humidity Sensor: An Overview

Pratibha Singh¹, Chandra Shekhar Kushwaha¹ and Saroj Kr Shukla^{1,*}

¹ Department of Polymer Science, Bhaskaracharya College of Applied Sciences, University of Delhi, Delhi-110075, India

Abstract: The present chapter describes the synthesis and applications of different classes of nanocomposites in humidity sensing applications, along with their innovative surface and responsive properties. The uses of nanocomposite-based humidity sensors in different fields like environmental monitoring, packaging, and the medical field have been described with suitable examples and illustrations. Furthermore, the humidity sensing mechanism of nanocomposite based humidity sensors are explained with sensing parameters and with future requirements

Keywords: Humidity sensors, Hybrid materials, Sensing mechanism, and applications.

INTRODUCTION

The synergistic properties in hybrid materials with nano confinements have yielded several advantageous features for effective chemical and biochemical sensing with precise parameters. Some of the notable advantageous features of this hybrid, in general also called nanocomposite, are surface area, selective catalysis, porosity, responsive and induced physical properties. These properties are also frequently used in sensing humidity in gas, liquid, and solid substances as optical, electrical, and mechanical transducers [1, 2].

Although humidity sensing is the oldest analytical tool for monitoring atmospheric prediction since the 12th century BC by Shuang Ruller, in China, after measuring the change in mass change due to adsorption humidity on charcoal due to variation in humidity contents. However, in the current era, the importance of humidity sensors has drastically increased due to the importance of water molecules in several sustaining reactions of living and nonliving bodies [3, 4]. Thus, the importance of humidity sensors encouraged scientists to use a wide

* Corresponding Author S. K. Shukla: Department of Polymer Science, Bhaskaracharya College of Applied Sciences, University of Delhi, Delhi-110075, India, E-mail: sarojshukla2003@yahoo.co.in

range of materials like ceramic, polymers, and carbonaceous materials with certain limitations.

However, for advancing the humidity sensing properties, the different composites materials like metal oxide to metal oxides, metal to metal oxides, polymer to biopolymer, metal oxides to polymers, carbon nanostructure to the polymer are used for precised monitoring of humidity. For example, Shukla *et al.* have increased the humidity sensing range of pristine polyaniline after grafting it with cellulose by 33% [5]. Some other significant synergistic effect of material on humidity sensing is given in Table 1.

Table 1. Evolution of significant synergistic properties in nanocomposites.

S N.	Composition	Synergistic Properties	Remark	Ref.
1	WO ₃ and MWCNT	Synergistic evolution of the <i>p-p</i> junction	Enhanced sensitivity in wider range humidity	[6]
2	TiO ₂ (K,Na)NbO ₃	Evolved hetero structure	Improved sensitivity by two to four order	[7]
3	ZnO and Polyvinylidene fluoride	Improved surface oxygen vacancy defects	Improved response and recovery times of 30s and 51s recovery, respectively	[8]
4	SnO ₂ and TiO ₂	Heavy ion beam induced dense electronic excitation	Long term reproducibility and recovery	[9]
5	Molybdenum disulfide and polyvinyl pyrrolidone	Inkjet printable to fabricate humidity sensors.	High sensitivity, ultrafast response/recovery behavior, and good reproducibility	[10]

The finding reveals the importance of making nano-sized composites in humidity sensing in order to fulfill the requirement in different filed. Furthermore, the existing literature indicates the effective increment in humidity sensing technology due to advances in nanocomposites with certain limitations like wide range sensitivity from gas to solid. This synergized advancement in humidity sensing using nanocomposites is presented in this chapter, along with findings and challenges.

OVERVIEW ON NANOCOMPOSITE BASED HUMIDITY SENSORS

In general, the nanocomposites are multicomponent hybrid materials with at least one component in nanosize, *i.e.*, 1 to 100 nm. Further, depending on the materials, the nanocomposite is classified into the following three categories: a) metal matrix, b) ceramic matrix, and c) polymer matrix. In resultant materials, all matrices incorporate their unique features, such as metal stretchability, ceramic stability, and polymer processability. As a result, all features contribute to

humidity sensing; however, ceramic and polymer-based nanocomposites are most commonly used in humidity sensing applications. Another important aspect is to control dispersion and agglomeration of dispersed phase during composite formations along with size confinements [11, 12]. Therefore, several methods are used for making a composite, but broadly they are grouped as *ex-situ* or *in-situ* methods for composite formation. In the *ex-situ* method, both components are prepared separately, and after that, they are composed with a defined matrix using different energy like chemical energy in solution method, thermal in melt mixing, and electrical energy in electrical depositions. However, in the *in-situ* method, the composite is prepared through the process of the development of a matrix in the presence of components. Both methods are still in use for the preparation of different composites with their inherited properties. The basic comparison of both methods is given in Table 2, along with examples.

Table 2. Comparison between *ex-situ* and *in-situ* methods [13].

<i>Ex-situ</i>	<i>In-situ</i>
Time consuming, bulk interaction, suitable for large scale production with disadvantages of requisite nanoparticles, dispersibility and control of long term aggregations	The simple and effective route to control long-term aggregation and good spatial distribution along with the presence of unreacted educts.

The involved fundamental principle in humidity sensing is the monitoring of different induced electrical, optical and magnetic properties against different humidity levels.

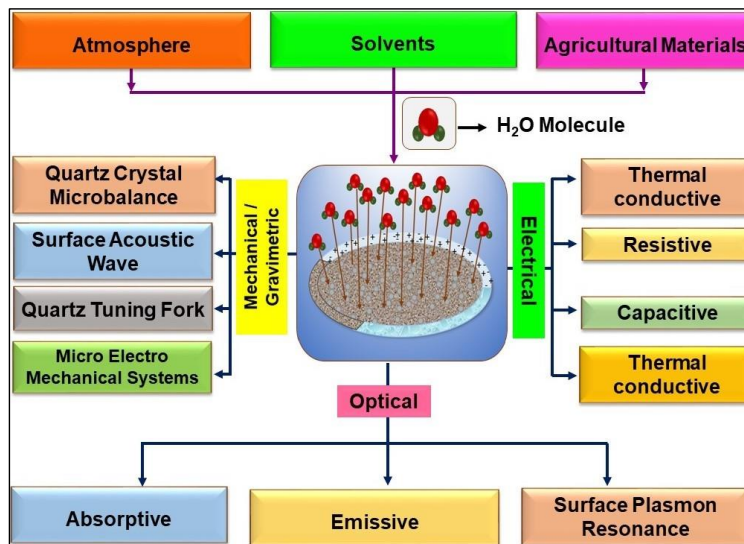


Fig. (1). Different class of humidity sensing principles [14].

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Manorama Singh

Dr Manorama, Department of Chemistry, GGV (A Central University), Bilaspur, C. G., India, has completed her Ph. D. in Chemistry from Banaras Hindu University, Varanasi, India. She has published several papers and book chapters in different international and national journals/books of high repute. Her research interests are electroanalytical chemistry, modified electrodes, electro-/organic catalysis and sensors.



Vijai K. Rai

Dr Vijai K. Rai, Department of Chemistry, GGV (A Central University), Bilaspur C. G., India has completed his D. Phil. in Chemistry from University of Allahabad, Allahabad, India. He has been a recipient of several awards viz., Golden Jubilee Award, NASI, India, D. S. Bhakuni Award, ICS etc. and has published several papers and book chapters in different international journals/books of high repute. His main research area is the development of green synthetic routes to new organic molecules using homogeneous and heterogeneous catalysts.



Ankita Rai

Dr Ankita Rai, Chemistry, School of Physical Sciences, JNU, New Delhi, India has completed her Ph. D. from the University of Allahabad, Allahabad, India. She has published several papers and book chapters in different international journals/books of high repute. Her research interests are synthetic organic chemistry especially environment-friendly organic transformations by developing new synthetic methodologies and new catalytic routes.