Synthesis of metal oxide decorated MXene nanosheets with their relevance in the detection of diabetes and other diseases

SUMMARY SUBMITTED FOR THE AWARD OF THE DEGREE OF

Doctor of Philosophy

in

Physics

Submitted by

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Enrolment no.- 1688/19

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LUCKNOW-226025 (U.P.) INDIA

2024

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SUMMARY

Air is essential for all living organisms, but industrialization and human activities have severely polluted it. Factories, refineries, waste, and fossil fuel burning release toxic gases, deteriorating air quality [1-3]. Without clean air, life on Earth would not be impossible. This pollution threatens the environment and human health, emphasizing the need for sustainable practices to protect the air and ensure the survival of all living organisms [2,3]. Volatile organic compounds (VOCs) are major contributors to air pollution, particularly in industrial settings where they are used as solvents [4, 5]. These hazardous pollutants are not only environmental threats but also hazards to human health, causing respiratory illnesses and vision disturbances. Prolonged exposure can be detrimental [8-10]. Monitoring VOC levels is important for human safety. Some VOCs found in exhaled breath have become biomarkers for early disease diagnosis, including lung infections [11,12]. Analysing VOCs in breath is a non-invasive and cost-effective method that provides insights into metabolic disorders and dysfunctions [13]. This dual role of VOCs as environmental pollutants and diagnostic tools highlights the need for rigorous monitoring and research to protect public health, environment, and improve disease management strategies [14–16]. Breath constituents like volatile organic compounds (VOCs) like isoprene, ammonia, and acetone, and a few inorganic gases like NO and CO, have been identified as potential biomarkers for specific diseases, providing valuable insights into health condition diagnosis and monitoring [17,18].

Acetone is a promising biomarker for metabolic monitoring, particularly in diabetes. It is produced through ketogenesis, a metabolic process where the body breaks down fats for energy [15,16]. Acetone metabolism is complex, involving formation, degradation, and elimination through urine, and its levels can be influenced by various physiological conditions [19,20]. Factors such as age, ketogenic diets, starvation, medications, exercise, lifestyle, addiction behaviours, and profession

can significantly influence breath acetone concentrations [21,22]. Detecting acetone in exhaled breath is a valuable tool for diagnosing diabetes and other disease states, as it can serve as a non-invasive marker to monitor the effectiveness of treatments [23,24]. Elevated breath acetone levels are common in uncontrolled diabetes, but can also increase during extensive physical activity or a ketogenic diet [25,26]. Accurately detecting changes in breath acetone levels allows healthcare providers to better diagnose, monitor, and manage various health conditions, making it a crucial tool in personalized medicine [27]. So, for real-world uses, a gas sensor with high sensitivity and selectivity for acetone detection is essential. This will increase the accuracy and reliability of monitoring acetone levels in environmental and medical diagnostics [28].

Gas sensor technologies include optical, electrochemical, capacitive, acoustic, and chemiresistive sensors shown in Figure 1(a). Semiconducting metal oxides (SMOx)-based chemiresistive sensors are highly researched due to their high sensitivity, low cost, ease of production, real-time detection, portability, and stability [29,30]. They offer affordable manufacturing, simple design, and easy integration into portable electronic devices, making them promising for non-invasive diabetes monitoring [31]. Metal oxide-based gas sensors, such as those using Fe_2O_3, MoO_3, WO_3, and Co_3O_4, offer significant benefits due to their high reliability and straightforward implementation. Among these semiconducting metal oxides, MoO_3 stands out as a promising candidate for gas sensing, catalytic, and electrochromic applications [27]. This potential is attributed to its wide bandgap, which ranges from 2.5 to 3.2 eV. To enhance the selectivity and decrease the operating temperature of MoO_3, it can be combined with other nanomaterials [32,33].

Figure 1(b) illustrates the widely used application of several nanostructured 2D materials in Chemiresistive-type gas sensing devices. These 2D nanomaterials include metal oxide, hBN, graphene, MXene and phosphorene, etc. MXenes, a novel class of two-dimensional (2D) materials, include transition metal carbides, nitrides, or carbonitrides [34]. First introduced by Gogotsi and colleagues in 2011, MXenes are created by selectively removing the ‘A’ layer from the layered MAX phase. In this structure, M represents an early transition metal, ‘A’ typically belongs to group IIIA or IVA elements, and X stands for carbon or nitrogen. This process yields MXenes, which exhibit unique properties and have potential applications in various fields [35]. As well as MXenes are promising sensor materials due to their high electrical conductivity and hydrophilic surface, which enhance their ability to adsorb gases effectively [36–38]. Their excellent conductivity and surface interaction make them suitable for various sensing applications, potentially improving performance in detecting analytes.

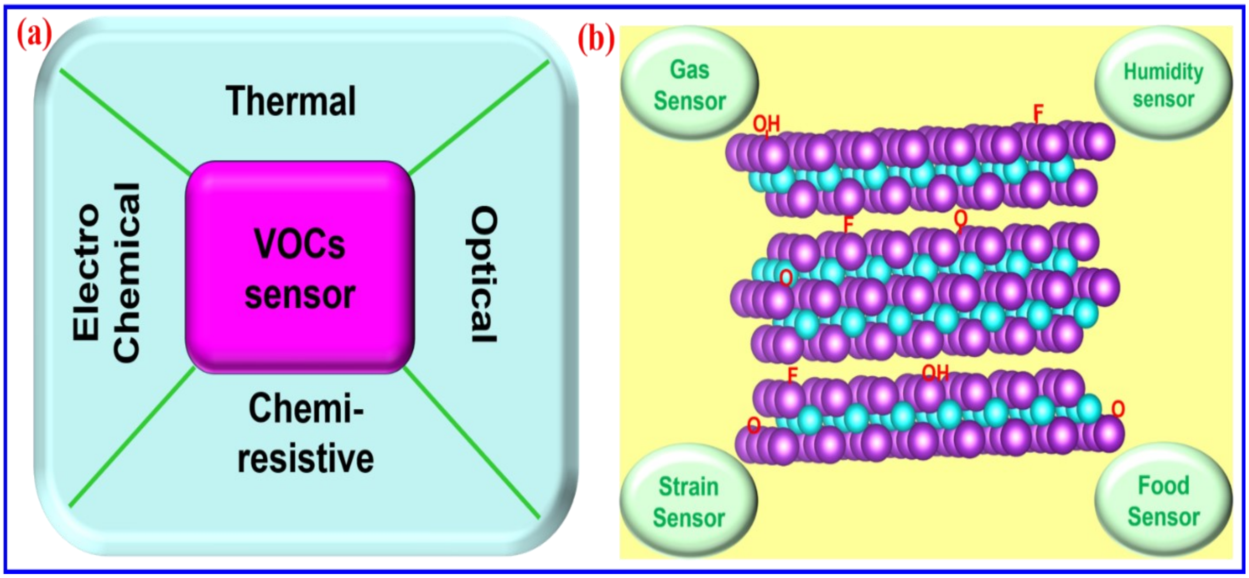

Figure 1 (a) Schematic diagram of various VOCs sensors, (b) different applications based on MXene.

Chapter 1 includes a comprehensive analysis of VOCs sensors, classifications, challenges, and their sensing mechanisms. It examines various types of gas sensors, including electrochemical, optical, chemiresistive, and thermal sensors, detailing how it is functioning with their respective applications. This chapter also focuses on 2D materials, with a particular emphasis on MXenes. MXenes are noted for their distinctive properties, including tunable bandgaps, chemical, electrical characteristics, and unique physical attributes. These materials exhibit exceptional hydrophilicity, flexibility, and high conductivity, making them highly promising for advanced gas sensing applications. Figure 2 shows the synthesis process of MXenes, typically represented by the formula $M_{n+1}X_nT_x$, which involves three key steps: etching, intercalation, and exfoliation. In this formula, M stands for an early transition metal (e.g. Ti, V), X represents carbon or nitrogen, and T_x denotes surface terminations like -OH, -O, or -F, which are functional groups formed during the synthesis. The etching process typically uses hydrofluoric acid (HF) or a safer alternative like LiF/HCl to selectively remove the A-layer from MAX phases, resulting in multilayered MXenes. Intercalation follows, where larger molecules or ions (e.g., DMSO, TBAOH) are introduced to expand the interlayer spacing, making it easier to separate the layers. The final step, exfoliation, produces single or few-layered MXene sheets with enhanced surface area and reactivity. This method enhances gas sensing capabilities by increasing surface area, exposing more active sites, improving sensitivity, response time, and facilitating better gas molecule diffusion. MXenes-based gas sensors are highly effective and important for addressing environmental and health-related issues. They detect VOCs (acetone, ethanol, isopropanol, methane etc), essential for medical diagnostics and environmental monitoring. Also, this chapter explores their applications in real-life scenarios, highlighting their importance in enhancing the sensitivity and selectivity of gas sensors in contributing to public health, safety, and environmental protection.

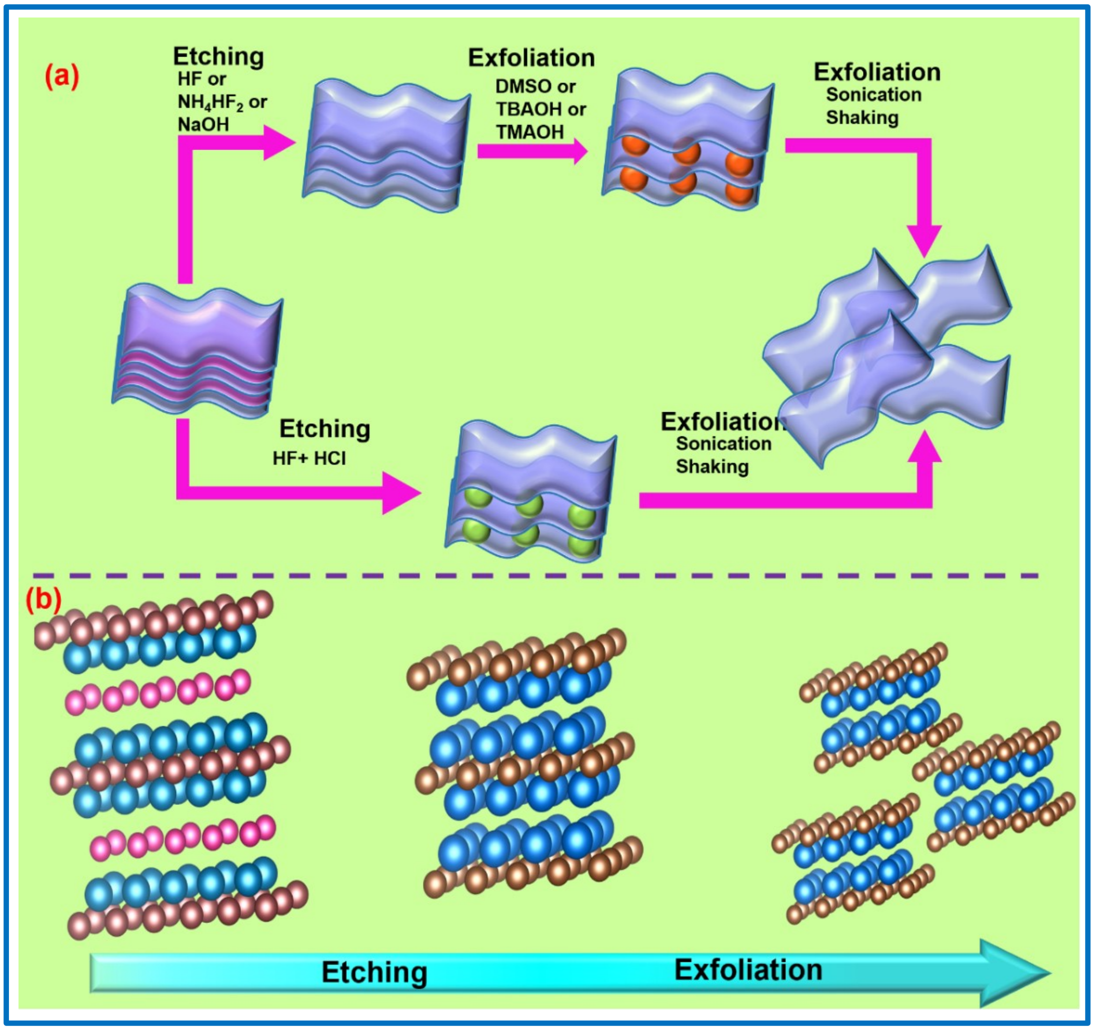

Figure 2. (a and b) MXene etching and exfoliation process depends on the synthesis process.

Chapter 2 focuses on acetone sensing, particularly its relevance in nail paint remover. Since acetone is commonly used as a cleaner in these products, inhaling it while applying or removing cosmetics can lead to severe health issues. Consequently, there is a pressing need for highly sensitive acetone sensors to detect and monitor acetone exposure effectively, ensuring safer use of these products and better protection of health. MoO_3-CdO nanocomposite were synthesized using a wet chemical method.

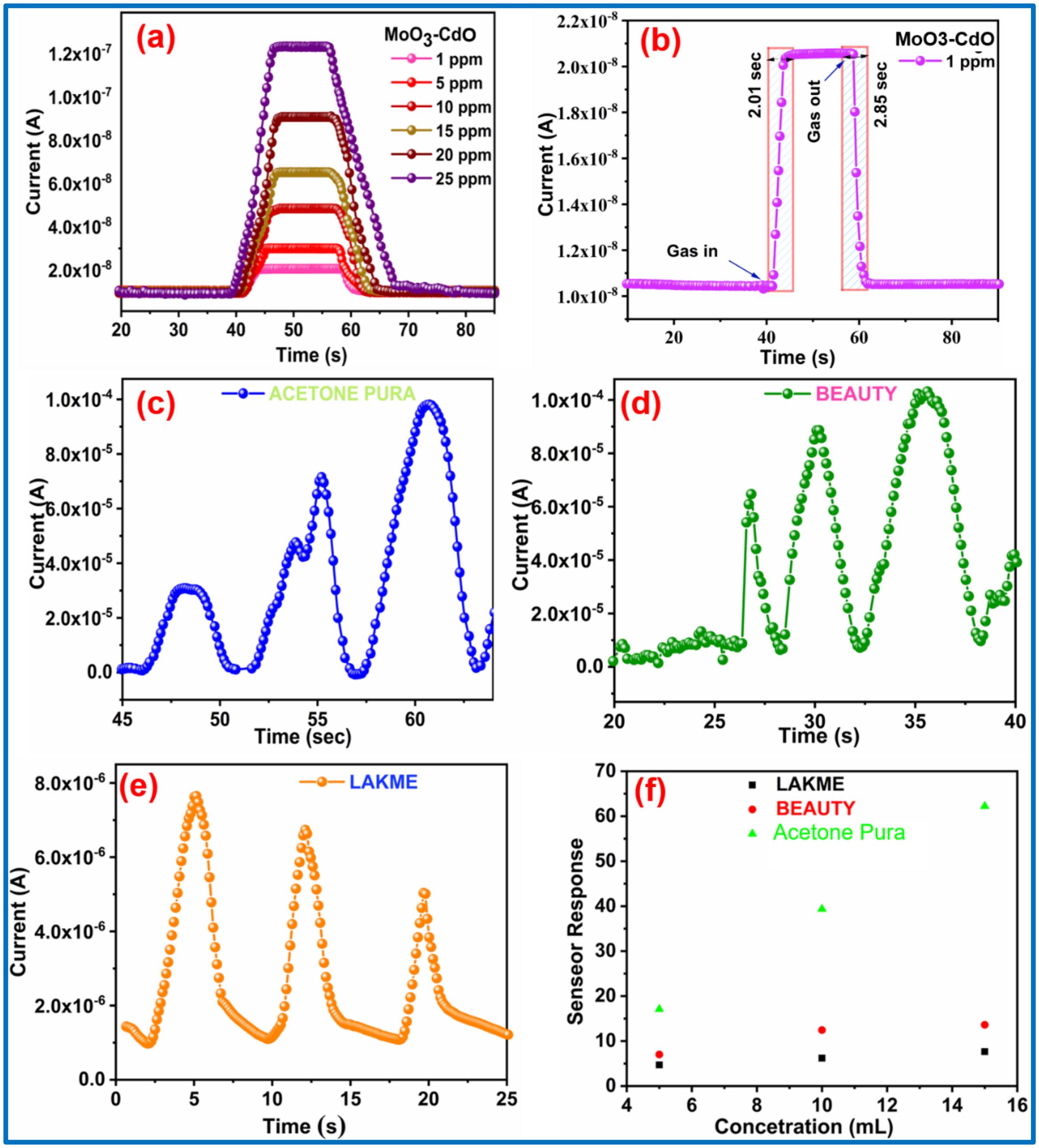

Figure 3. (a) The static response and recovery curves for 1–25 ppm acetone sensing at room temperature of MoO_3-CdO nanocomposite, (b) response and recovery time curves of MoO_3-CdO nanocomposite at 1 ppm acetone concentration, sensor characteristics at various concentrations (c)

Acetone Pura company (d) Beauty company (e) Lakme company (f) sensor responses for nail paint removers of various companies showing various concentrations (5–15 ml) of acetone gas.

The structural and morphological studies were carried out by XRD and SEM. The SEM micrographs confirm the successful formation of MoO_3 nanosheets and CdO nanoparticles, revealing the even dispersion of CdO nanoparticles across the surface of MoO_3 nanosheets within the MoO_3-CdO nanocomposite. The formation of 2D MoO_3 nanosheets decorated with CdO nanoparticles with a characteristic energy band gap, crystallite size, and particle size of 3.80 eV, 38.69 nm, and 2-50 nm, respectively.

Acetone gas sensing characteristics of MoO_3-CdO nanocomposite are exhibited in Figure 3(a) in the range of 1-25 ppm. At 1 ppm, the sensor demonstrated a response of 1.92, with response and recovery times of 2.01 s and 2.85 s, respectively as illustrated in Figure 3(b). The CdO nanoparticles significantly enhance the adsorption and desorption rates of acetone gas molecules, which contributes to the improved sensing performance of MoO_3-CdO nanocomposite. This advancement has promising commercial applications, raising awareness of health issues and fostering research in this field. Future commercial endeavours could lead to the development of more sensitive acetone gas sensors that operate efficiently at low temperatures. Additionally, acetone sensing studies were conducted on products from various commercial brands, including Acetone Pura, Beauty Company, and Lakme nail polish removers as shown in Figure 3 (c-f). The MoO_3-CdO nanocomposite sensing device shows potential for use in monitoring acetone levels in cosmetic applications, offering a valuable tool for protecting human skin.

Chapter 3 discusses the need for cost-effective and scalable methods to fabricate high-performance cotton-based VOC sensors, which are important for the advancement of wearable sensing devices. Cotton yarn-based sensors with their lightweight and adaptable nature, offer the perfect solution for diverse surface attachment and effective acetone tracking as illustrated in Figure 4(a-c). In this work, precise synthesized of $Ti_3C_2T_x$, MoO_3, and $MoO_3/Ti_3C_2T_x$ nanocomposite were confirmed by various characterization techniques such as Scanning electron microscopy (SEM) analysis, X-ray diffraction (XRD), Dynamic light scattering (DLS) analysis, Fourier transforms infrared spectroscopy (FTIR), UV-visible spectroscopy and X-ray photoelectron spectroscopy (XPS) respectively. SEM images confirmed the accordion-like structure of $Ti_3C_2T_x$ MXene with MoO_3 nanosheets evenly distributed across the surface of $Ti_3C_2T_x$ MXene as shown in Figure 4(d,e). This provides clear evidence of successful MoO_3 decoration on the $Ti_3C_2T_x$ MXene. The optical band gap of $Ti_3C_2T_x$ MXene and $MoO_3/Ti_3C_2T_x$ were confirmed to be 3.30 and 3.37 eV respectively using the Tauc's plot. Additionally, the diffraction pattern of $Ti_3C_2T_x$ explains a broad peak at $2\theta \sim 8.8°$ corresponding to the (002) Miller indices. The enlarged 'd' spacing was measured at 8.9 nm attributed to surface expansion which enhances the sensing properties of materials.

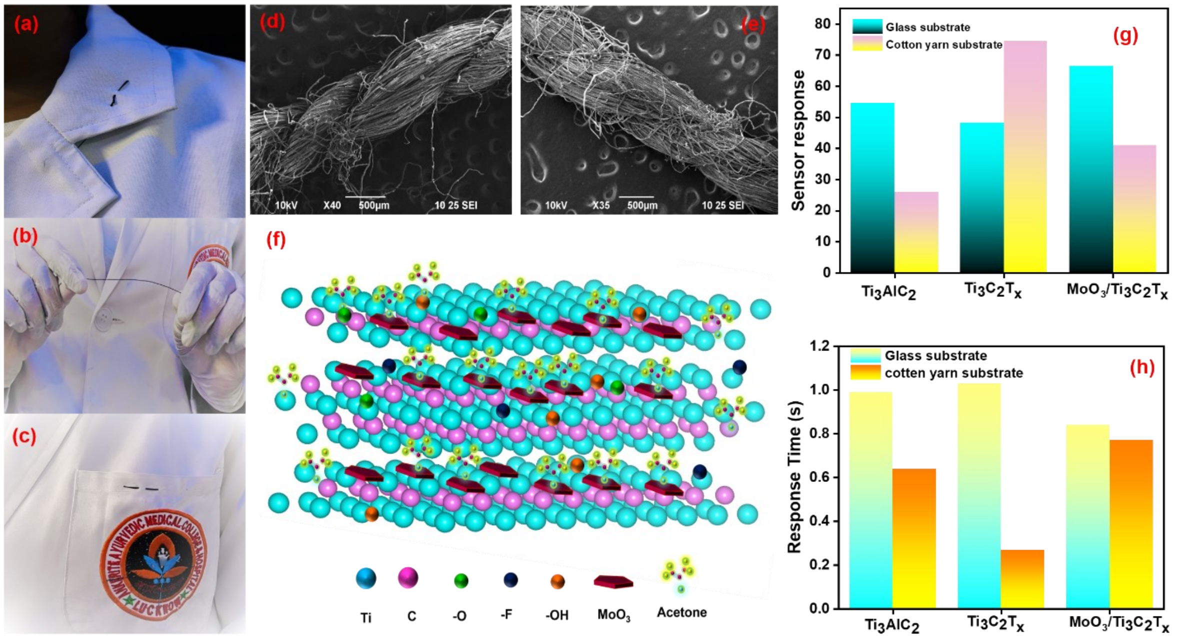

Figure 4. (a-c). Real-time detection of acetone in apron during lab testing, (d, e) SEM images of $MoO_3/Ti_3C_2T_x$ coated on glass and cotton yarn substrate, respectively (f) acetone sensing mechanism, (g) real-time sensor response for different samples and (h) comparative plot for response time demonstrated by fabricated sensors.

The $MoO_3/Ti_3C_2T_x$ thin film coated on glass substrate exhibited a sensor response of 66.50, with response and recovery time of 0.84 and 1.29 s, respectively as compared to $MoO_3/Ti_3C_2T_x$ coated cotton yarn exhibiting a senor response of 40.9 with response and recovery time of 0.77 and 0.70 s at 25 ppm acetone concentration shown in Figure 4(g and h). Fast response and recovery time are devoted to the rich concentration of available adsorption sites on cotton yarn.

This research provides a plausible solution for developing a cotton yarn-supported $MoO_3/Ti_3C_2T_x$ acetone sensor as the wearable monitoring system. Figure 5(a and b) illustrates the transient sensor response of the $MoO_3/Ti_3C_2T_x$ nanocomposite to human exhaled breath, specifically with blood glucose levels. The $MoO_3/Ti_3C_2T_x$-based device, being biodegradable and low-cost, utilizes cotton yarn-based acetone gas sensor technology. This approach may be employed as a textile-based wearable platform for real-time sensing and monitoring.

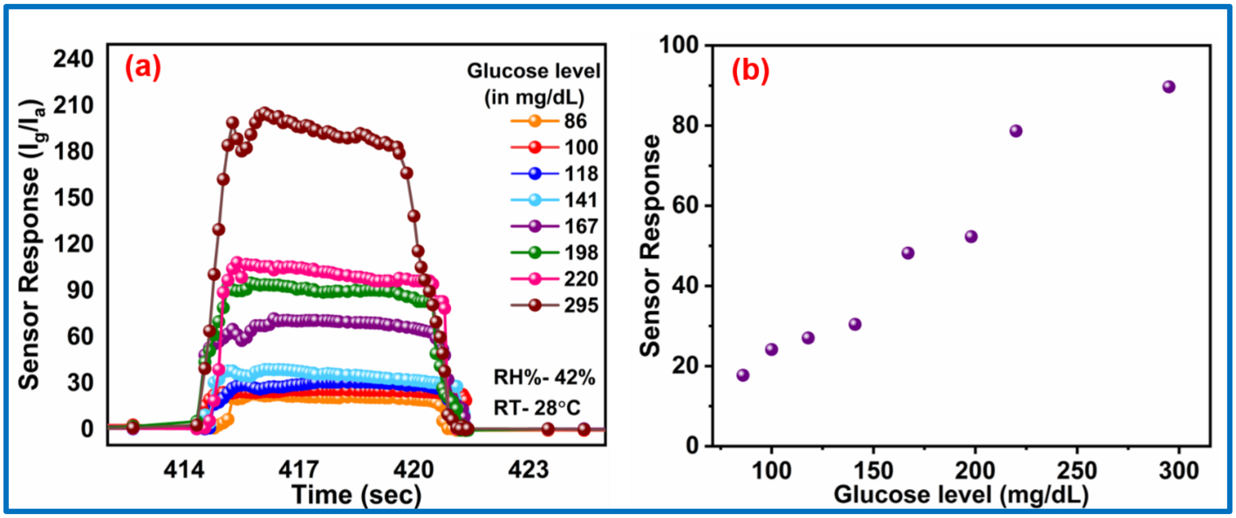

Figure 5. (a) Human exhalation breath's transient sensor response to blood glucose level and (b) linearity of the MoO_3/Ti_3CT_x in terms of glucose level.

Chapter 4 embodies the paper-based wearable acetone sensing applications that are favoured for their bio-degradable, environmentally friendly, robust, and cost-effective properties. In this work, we investigated the effectiveness of MoO_3 nanosheets decorated by V_2CT_x nanocomposite (MoO_3/V_2CT_x) as promising materials for acetone sensing along with hand sanitizer monitoring capabilities for the protection of skin. Here, 2D V_2CT_x was etched from the MAX phase (V_2AlC) and conjugated with 2D MoO_3 by the wet chemical method shown in Figure 6(a). The sensing setup for acetone sensing for device MoO_3/V_2CT_x is shown in Figure 6(b).

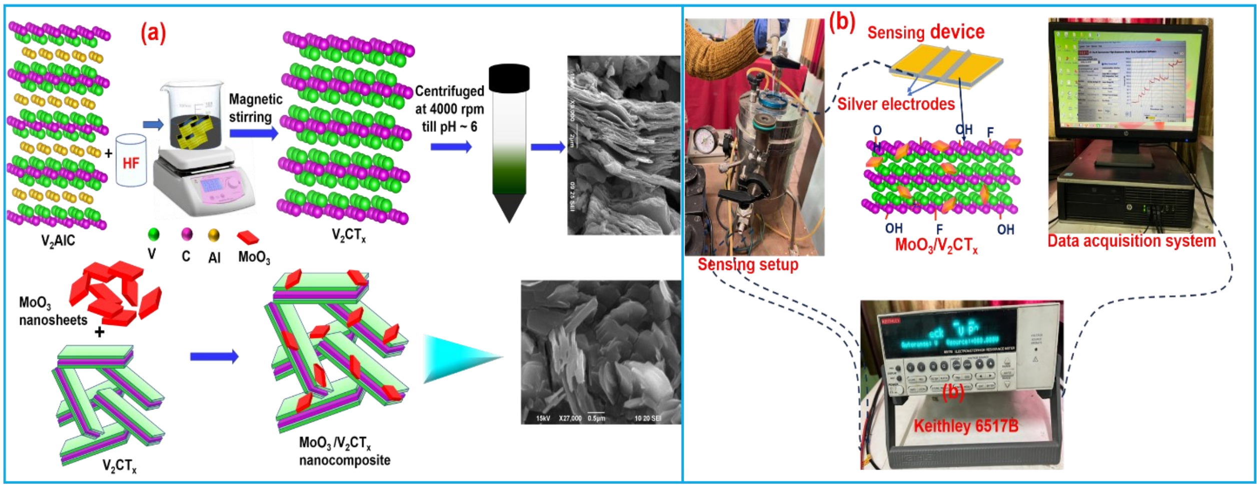

Figure 6. Schematic diagram of (a) synthesis process of V_2CT_x MXene, and MoO_3/V_2CT_x nanocomposite and (b) acetone sensing measurement setup.

The HR-TEM micrograph of the as-prepared MXene at low magnification confirms that the MXene sheets as stacked-layered characteristics. The hexagonal symmetry of the structure is further demonstrated by the selected area electron diffraction (SAED) pattern of the as-prepared MXene sample.

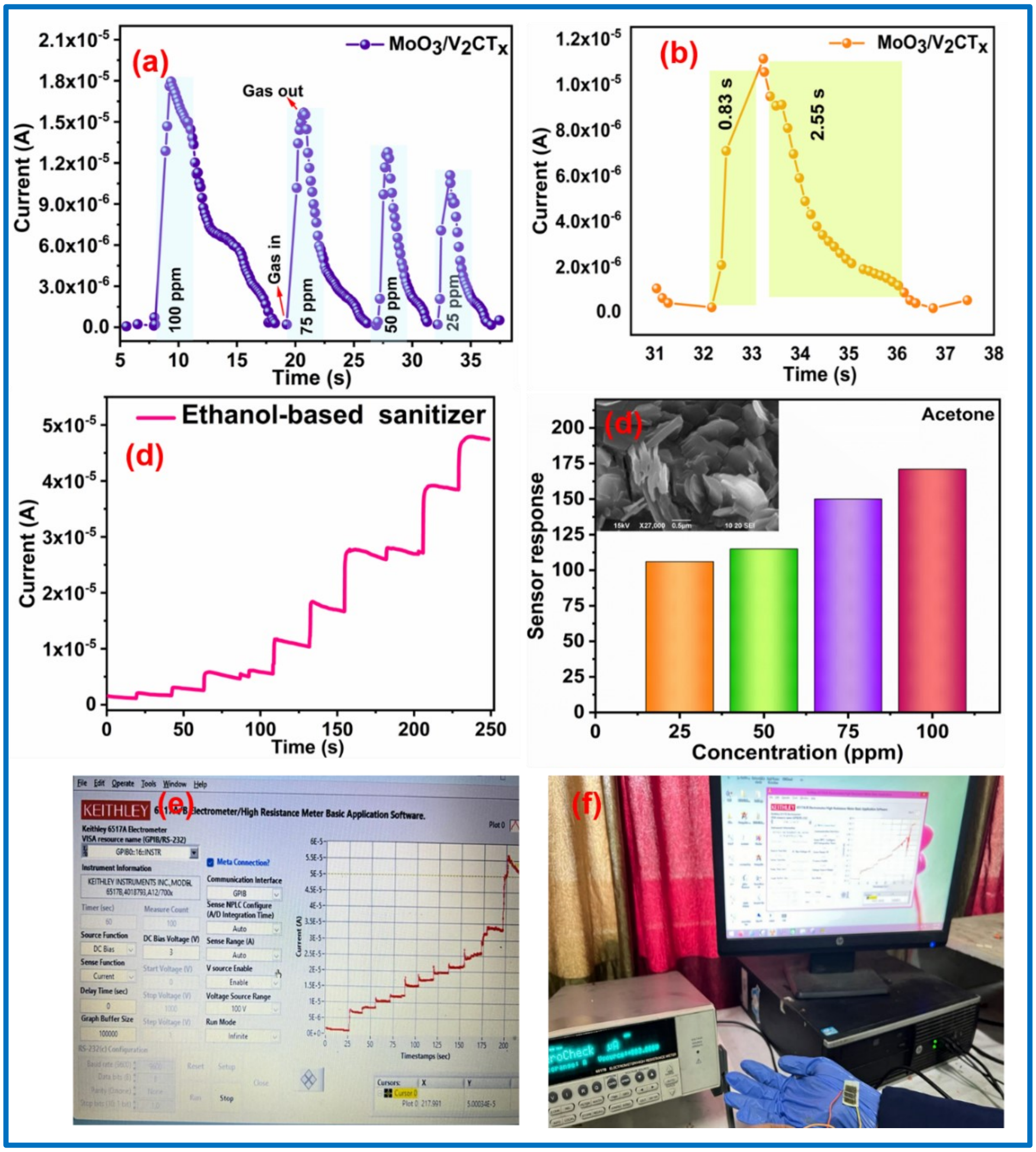

Figure 7. (a) Acetone sensing demonstration in the various concertation (25-100 ppm) for MoO_3/V_2CT_x nanocomposite, (b) for 25 ppm, (c) sensor response graph for MoO_3/V_2CT_x nanocomposite, (d) real-time ethanol-based sanitizer sensing, (e) real-time isopropanol-based sanitizer sensing, (f) wearable device connected with data acquisition system.

The MoO_3/V_2CT_x sensor demonstrated outstanding performance in the detection of acetone, showing fast response/recovery times (0.83/2.55 s) at 25 ppm of acetone concentration shown in Figure 7(a and b). It was observed that the MoO_3/V_2CT_X nanocomposite-based sensor exhibits good selectivity with long-term stability. At room temperature, the sensor response of MoO_3/V_2CT_x was found to be 106.21 for the acetone concentration of 25 ppm, which is better than the sensor response of V_2CT_x of 28.00 at the same acetone concentration. Additionally, the MoO_3/V_2CT_x device offers a practical application for monitoring the quality of hand sanitizers produced by different companies as shown in Figure 7(c-f). The MoO_3/V_2CT_x sensor demonstrates exceptional rapid sensitivity compared to previous reported sensors, providing significant potential for real-time monitoring. The MoO_3/V_2CT_x-based device could serve as a prototype tool for ensuring the consistency and safety of hand sanitizer formulations and may be widely adopted across the industry for monitoring products from various manufacturers.

Chapter 5 deals with monitoring of exhaled human breath via cost-effective MoO_3/Nb_2CT_X chemiresistor. Breath analyzer is a noninvasive way to diagnose diseases and provides important biochemical and physiological information about the health of an individual. Acetone originating from exhaled breath is increasingly recognized as a valuable biomarker with substantial potential for diagnosing and monitoring diabetics. This work demonstrated the sensitive and highly selective acetone characteristics of chemiresistive sensing devices based on MoO_3 nonrectangular bars (NRBs), Nb_2CTx, and MoO_3/Nb_2CT_X materials. The SEM morphology confirmed the accordion-like structure, indicating the successful removal of the Al atomic layer and successfully synthesized the Nb_2CTx MXene. Additionally, it reveals several gaps within the layers, suggesting that the Al layers have been eliminated through complete exfoliation of the 2D MXene multilayers from its MAX phase. As well as the SEM images of the MoO_3/Nb_2CT_X nanocomposite structure explained the formation of a porous structure MoO_3 NRBs on the surface of the Nb_2CT_x, which helps to enhance the sensitivity of the gas sensor. According to XPS data, levels of Mo $3d_{5/2}$ (232.60 eV) and Mo $3d_{3/2}$ (235.97 eV) can be attributed to the Mo^{6+} oxidation state. As well as Nb 3d spectrum which were discovered with binding energies of Nb-C ($3d_{5/2}$) (199.8 eV) and ($3d_{3/2}$) (208.5 eV), respectively, indicating an oxidation state of Nb^{4+}. This work demonstrated the sensitive and highly selective acetone characteristics of chemiresistive sensing devices based on MoO_3, Nb_2CTx, and MoO_3/Nb_2CT_X materials. The flexible, biodegradable cellulose paper was used to create the MoO_3/Nb_2CT_X-based sensor for acetone detection at low ppm. The MoO_3/Nb_2CT_X sensor exhibits remarkable sensitivity, showing a high sensor response of 10.24 at 1 ppm which further rises to 180.30 at 100 ppm acetone concentration as shown in Figure 8 (a and b).

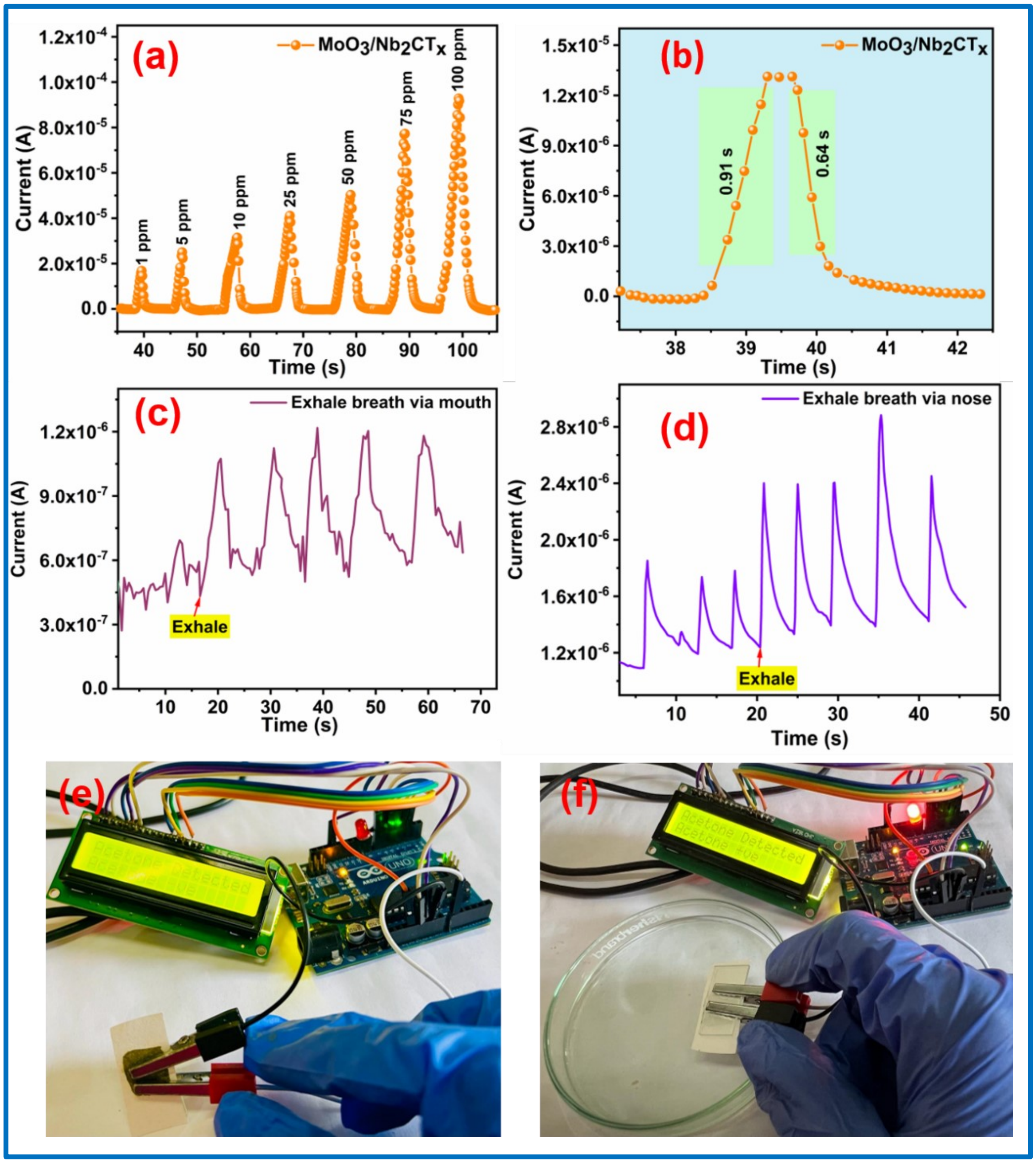

Figure 8. (a) The dynamic response-recovery curve of MoO_3/Nb_2CT_x nanocomposite sensors to 1-100 ppm acetone concentration, (b) MoO_3/Nb_2CT_x nanocomposite at 1 ppm, (c) continuous detection of exhaled breath via the mouth, (d) continuous detection of exhaled breath via the nose. (e) Photograph shows the acetone detector consisting of LCD, (f) shows a live paper-based acetone sensor setup after exposing the acetone vapour coming from the petri dish.

Notably, it possesses a lower limit of detection (LOD) of 0.55 ppm in the acetone concentration range of 1-100 ppm. The MoO_3/Nb_2CT_x sensor used for real-time monitoring is shown in Figure 8 (c and d) for continuous breath monitoring via mouth and nose. The sensor response for breath through the mouth is 2.00, whereas for breath through the nose it is 1.61. The higher sensor response for breath via the nose compared to the mouth is due to the presence of water vapour. Figure 8 (e and f) displays the various components and electronic circuits of the acetone detector prototype. Here, Arduino was used for basic health monitoring applications. This work showed remarkably high sensor response and selectivity of MoO_3/Nb_2CT_x sensor for detecting acetone. This may lead to important progress in noninvasive chemiresistive sensing for monitoring diabetes and other diseases expanding the healthcare horizon.

Table 6.1 Chapter wise outline of the Thesis

Chap .No.	**Materials**	**Acetone (ppm)**	**Res. time (s)**	**Reco. time (s)**	**Sensor response**	**Porosity %**	**Diseases**
1.	Introduction and aim of present research work						
2.	MoO_3-CdO	1	2.01	2.85	1.92	22.54	Dermatitis
3.	$MoO_3/Ti_3C_2T_x$	1	0.48	0.44	5.60	49.20	Diabetes
4.	MoO_3/V_2CT_x	1	0.68	1.91	9.30	54.00	Dermatitis
5.	MoO_3/Nb_2CT_x	1	1.06	0.64	10.24	57.80	Diabetes
6.	Concluding remarks and scope of future research						

Scope of further research

- MXene and its metal oxide composites-based materials with large specific surface areas an abundance of porosity, and good conductivity are expected to be promising candidates for room-temperature gas detection, particularly for hazardous gases and volatile organic compounds (VOCs).
- Also, MXene may be more effective because of its capacity to create proper chemical bonds with a various substance due to different functional groups such as -C and -O with synthesis of the single layer MXene.
- MXene has different termination groups with interlayer spacing which make surface-modified MXene-based materials and may be used in various applications.
- Use of MXene composites for environmental monitoring and biological sensing is a promising area. Therefore, researches may be carried out in future for how well they can detect pollutants, biomarkers, and other important targets related to health and the environment.
- Incorporating MXene composites into advanced sensing platforms with real-time and remote monitoring capabilities could enhance their application scope. This includes developing wireless sensing systems and integrating them with IoT technologies for remote data collection and analysis.
- Developing efficient synthesis and processing techniques for improving the aspect ratio may be crucial for commercializing the gas sensors and other energy storage devices.

References

[1] G.I. Edo, L.O. Itoje-akpokiniovo, P. Obasohan, V.O. Ikpekoro, P.O. Samuel, A.N. Jikah, L.C. Nosu, H.A. Ekokotu, U. Ugbune, E.E.A. Oghroro, others, Impact of environmental pollution from human activities on water, air quality and climate change, Ecol. Front. (2024).

[2] I. Manisalidis, E. Stavropoulou, A. Stavropoulos, E. Bezirtzoglou, Environmental and health impacts of air pollution: a review, Front. Public Heal. 8 (2020) 14.

[3] G. Richard, S.C. Izah, M. Ibrahim, Air pollution in the Niger Delta region of Nigeria: sources, health effects, and strategies for mitigation, J. Environ. Stud. 29 (2023) 1–15.

[4] M. Devaraj, S. Rajendran, T.K.A. Hoang, M. Soto-Moscoso, A review on MXene and its nanocomposites for the detection of toxic inorganic gases, Chemosphere. 302 (2022) 134933.

[5] A.M.O. Mohamed, E.K. Paleologos, Sustainable pollution assessment practices, in: Pollut. Assess. Sustain. Pract. Appl. Sci. Eng., Elsevier, 2021: pp. 3–42.

[6] J. Ma, L. Li, VOC emitted by biopharmaceutical industries: Source profiles, health risks, and secondary pollution, J. Environ. Sci. 135 (2024) 570–584.

[7] F. Nawaz, M. Ali, S. Ahmad, Y. Yong, S. Rahman, M. Naseem, S. Hussain, A. Razzaq, A. Khan, F. Ali, others, Carbon based nanocomposites, surface functionalization as a promising material for VOCs (volatile organic compounds) treatment, Chemosphere. (2024) 143014.

[8] T.T. Mashangva, A. Goel, U. Bagri, S. Prasher, A. Sharma, M. Kumar, P.K. Singh, Frontiers in MXene research: Pioneering synthesis, unveiled properties, and emerging applications in VOC detection, Appl. Mater. Today. 38 (2024) 102163.

[9] D.E. Schraufnagel, J.R. Balmes, C.T. Cowl, S. De Matteis, S.-H. Jung, K. Mortimer, R. Perez-Padilla, M.B. Rice, H. Riojas-Rodriguez, A. Sood, others, Air pollution and noncommunicable diseases: A review by the Forum of International Respiratory Societies' Environmental Committee, Part 2: Air pollution and organ systems, Chest. 155 (2019) 417–426.

[10] S.S. Shetty, D. Deepthi, S. Harshitha, S. Sonkusare, P.B. Naik, H. Madhyastha, others, Environmental pollutants and their effects on human health, Heliyon. 9 (2023).

[11] J.J. Haworth, C.K. Pitcher, G. Ferrandino, A.R. Hobson, K.L. Pappan, J.L.D. Lawson, Breathing new life into clinical testing and diagnostics: perspectives on volatile biomarkers from breath, Crit. Rev. Clin. Lab. Sci. 59 (2022) 353–372.

[12] S. Das, M. Pal, Non-invasive monitoring of human health by exhaled breath analysis: A comprehensive review, J. Electrochem. Soc. 167 (2020) 37562.

[13] F. Yin, W. Yue, Y. Li, S. Gao, C. Zhang, H. Kan, H. Niu, W. Wang, Y. Guo, Carbon-based nanomaterials for the detection of volatile organic compounds: A review, Carbon N. Y. 180 (2021) 274–297.

[14] Y.Y. Broza, R. Vishinkin, O. Barash, M.K. Nakhleh, H. Haick, Synergy between nanomaterials and volatile organic compounds for non-invasive medical evaluation, Chem. Soc. Rev. 47 (2018) 4781–4859.

[15] N. Lagopati, T. F. Valamvanos, V. Proutsou, K. Karachalios, N. Pippa, M. A. Gatou, I. A. Vagena, S. Cela, E. A. Pavlatou, M. Gazouli, others, The role of nano-sensors in breath analysis for early and non-invasive disease diagnosis, Chemosensors. 11 (2023) 317.

[16] N. Nath, A. Kumar, S. Chakroborty, S. Soren, A. Barik, K. Pal, F.G. de Souza Jr, Carbon nanostructure embedded novel sensor implementation for detection of aromatic volatile organic compounds: an organized review, ACS Omega. 8 (2023) 4436–4452.

[17] M. Sun, Z. Wang, Y. Yuan, Z. Chen, X. Zhao, Y. Li, C. Wang, Continuous monitoring of breath acetone, blood glucose and blood ketone in 20 type 1 diabetic outpatients over 30 days, J. Anal. Bioanal. Tech. 8 (2017) 2155–9872.

[18] A.T. Güntner, J.F. Kompalla, H. Landis, S.J. Theodore, B. Geidl, N.A. Sievi, M. Kohler, S.E. Pratsinis, P.A. Gerber, Guiding ketogenic diet with breath acetone sensors, Sensors. 18 (2018) 3655.

[19] G. Teresiński, G. Buszewicz, R. M kadro, The influence of ethanol on the level of ketone bodies in hypothermia, Forensic Sci. Int. 127 (2002) 88–96.

[20] V. Ruzsányi, M.P. Kalapos, Breath acetone as a potential marker in clinical practice, J. Breath Res. 11 (2017) 24002.

[21] P. Puchalska, P.A. Crawford, Multi-dimensional roles of ketone bodies in fuel metabolism, signaling, and therapeutics, Cell Metab. 25 (2017) 262–284.

[22] A.T. Guntner, N.A. Sievi, S.J. Theodore, T. Gulich, M. Kohler, S.E. Pratsinis, Noninvasive body fat burn monitoring from exhaled acetone with Si-doped WO_3-sensing nanoparticles,

Anal. Chem. 89 (2017) 10578–10584.

[23] M. Mahnoor, A.A. Shah, A. Inam, Acetone detection using various techniques for diagnosis of diabetes mellitus from human exhaled breath: A review, in: AIP Conf. Proc., 2024.

[24] X. Qu, Y. Hu, C. Xu, Y. Li, L. Zhang, Q. Huang, S.S. Moshirian-Farahi, J. Zhang, X. Xu, M. Liao, others, Optical sensors of volatile organic compounds for non-invasive diagnosis of diseases, Chem. Eng. J. (2024) 149804.

[25] T.D.C. Minh, D.R. Blake, P.R. Galassetti, The clinical potential of exhaled breath analysis for diabetes mellitus, Diabetes Res. Clin. Pract. 97 (2012) 195–205.

[26] M. Shokrekhodaei, S. Quinones, Review of non-invasive glucose sensing techniques: optical, electrical and breath acetone, Sensors. 20 (2020) 1251.

[27] N. Alizadeh, H. Jamalabadi, F. Tavoli, Breath acetone sensors as non-invasive health monitoring systems: A review, IEEE Sens. J. 20 (2019) 5–31.

[28] G. Wang, Z. Fu, T. Wang, W. Lei, P. Sun, Y. Sui, B. Zou, A rational design of hollow nanocages Ag@ CuO-TiO_2 for enhanced acetone sensing performance, Sensors Actuators B Chem. 295 (2019) 70–78.

[29] V. Saasa, B. Mwakikunga, Facile synthesis, characterization and acetone sensing properties of n-type WO_3, SnO_2 and VO_2 semiconducting materials and their cobalt doped performance: Outstanding SnO_2-Co acetone selectivity and sensitivity, Mater. Res. Bull. 164 (2023) 112288.

[30] A. Staerz, F. Roeck, U. Weimar, N. Barsan, Electronic nose: Current status and future trends, Surf. Interface Sci. Vol. 9 Appl. Surf. Sci. I. 9 (2020) 335–379.

[31] S.K. Vashist, Non-invasive glucose monitoring technology in diabetes management: A review, Anal. Chim. Acta. 750 (2012) 16–27.

[32] J. Wu, Z. Chen, X. Xu, P. Wei, G. Xie, X. Zhang, The Growth Process and Photocatalytic Properties of h-MoO_3 and α-MoO_3 under Different Conditions, Crystals. 13 (2023) 603.

[33] E. de Barros Santos, F.A. Sigoli, I.O. Mazali, Structural evolution in crystalline MoO_3 nanoparticles with tunable size, J. Solid State Chem. 190 (2012) 80–84.

[34] A. Yadav, H. Kumar, R. Sharma, R. Kumari, Synthesis, processing, and applications of 2D (nano) materials: A sustainable approach, Surfaces and Interfaces. 39 (2023) 102925.

[35] S. Venkateshalu, A.N. Grace, MXenes—A new class of 2D layered materials: Synthesis, properties, applications as supercapacitor electrode and beyond, Appl. Mater. Today. 18

(2020) 100509.

[36] Y. Chen, X. Li, C. Zhu, G. Fan, S. Khademolqorani, S.N. Banitaba, Recent insights on MXene-based architectures for monitoring and sensing of gaseous pollutants: A review, Talanta. 280 (2024) 126700.

[37] Q. Xia, Y. Fan, S. Li, A. Zhou, N. Shinde, R.S. Mane, MXene-based chemical gas sensors: Recent developments and challenges, Diam. Relat. Mater. 131 (2023) 109557.

[38] K. Deshmukh, T. Kovávrik, S.K.K. Pasha, State of the art recent progress in two dimensional MXenes based gas sensors and biosensors: A comprehensive review, Coord. Chem. Rev. 424 (2020) 213514.

LIST OF PUBLICATIONS

Part of the thesis published and communicated in the refereed journals:

1. ***Monu Gupta***, Arpit Verma, Priyanka Chaudhary, Bal Chandra Yadav, MXene and their integrated composite-based acetone sensors for monitoring of diabetes, *Materials Advances,* 2023, 4, 3989-4010.
2. ***Monu Gupta***, Priyanka Chaudhary, Ajeet Singh, Arpit Verma, Deepankar Yadav, Bal Chandra Yadav, Development of MoO_3-CdO nanoparticles-based sensing device for the detection of harmful acetone levels in our skin and body via nail paint remover, *Sensors and Actuators B: Chemical*, 2022, 368 132102.
3. ***Monu Gupta***, Priyanka Chaudhary, Dheeraj Maurya, Bal Chandra Yadav, Investigations on wearable cotton yarn-based exhaled breath analyser for diabetes diagnosis and medical industry, Surface and Interfaces (Under review: Manuscript number- SURFIN-24-04825)
4. ***Monu Gupta***, Arpit Verma, Priyanka Chaudhary, Bal Chandra Yadav, MoO_3/V_2CT_X Nanocomposite-Based Wearable Sensor with High Sensitivity for Acetone and Hand Sanitizer Detection at Room Temperature with DFT Analysis, *ACS Applied Electronic Materials* 2024, 6, 8, 5626-5639.
5. ***Monu Gupta***, Arpit Verma, Priyanka Chaudhary, Gyan Prabhakar, Bal Chandra Yadav, Portable MoO_3 Decorated Nb_2CT_x Sensor for Detection of Diabetes Level through Exhaled Breath at Room Temperature with DFT Analysis, *Applied Materials Today* (communicated).

Work not included in Thesis

1. Arpit Verma, Deepankar Yadav, Ajeet Singh, ***Monu Gupta***, KB Thapa, Bal Chandra Yadav, Detection of acetone via exhaling human breath for regular monitoring of diabetes by low-cost sensing device based on perovskite $BaSnO_3$ nanorods, *Sensors and Actuators B: Chemical*, 2022, 361, 131708.
2. Sonam Sharma, Arpit Verma, Priyanka Chaudhary, Ajeet Singh, ***Monu Gupta*** Ravi Kant Tripathi, Bal Chandra Yadav, Structural and photodetection studies of hydrothermally grown anatase TiO_2 nanomaterial, *Materials Today: Proceedings*, 2023, 255-262.

3. Arpit Verma, Deepankar Yadav, Subramanian Natesan, ***Monu Gupta***, Bal Chandra Yadav, Yogendra Kumar Mishra, Advancements in nanohybrid material-based acetone gas sensors relevant to diabetes diagnosis: A comprehensive review, *Microchemical Journal*, 2024, 201, 110713.
4. Manmohan Mishra, Vishwas Pratap Banga, Mahendra Kumar, ***Monu Gupta***, Effect of aging on transmittance, and effect of annealing temperature on CO_2 sensing of ZnO thin film deposited by spin coating, e-Prime-Advances in *Electrical Engineering, Electronics and Energy*, 2024, 7, 100405.
5. Arpit Verma, Deepankar Yadav, ***Monu Gupta***, Bal Chandra Yadav, Heterostructure nanohybrid MoO_3/Bi_2MoO_6 based device relevant to non-invasive monitoring of diabetes and other diseases through improved VOCs sensing, communicated in Advanced Composites and Hybrid Materials.
6. Priyanka Chaudhary, ***Monu Gupta***, and Bal Chandra Yadav, Polymer/metal oxide composites and their humidity sensing characteristics, In Complex and Composite Metal Oxides for Gas VOC and Humidity *Sensors Volume 1, pp. 393-407, Elsevier, 2024*.
7. Deepankar Yadav, Priyanka Chaudhary, Priya Sing, ***Monu Gupta***, Subhini Saraf, Resveratrol-Quantum dots loaded albumin nanoparticles for antioxidant potential and fluorescent probes, RSC Pharmaceutics, 2024.
8. Kewal Bharti, ***Monu Gupta***, Rajkumari, Prabhat Tiwari, Rahul Singh, Bala Bharadwaj, Kumar Singh, Bal Chandra Yadav, Shipra Tripathi, Santosh Kumar, LPG sensing study of Calcium doped Praseodymium orthoferrite nanomaterial, Analytical Chemistry (Communicated)

Papers presented in Conferences/Seminar/Workshop.

1. **Poster presented** at "International Conference of Material for Sustainable Development (ICMSD)-2022" organized by the Department of Nanoscience and Materials, Central University of Jammu, J&K India, held on 19-20 October 2022.
2. **Oral presentation** at the "National Seminar on Recent Advance in Multifunctional Materials" organized by the Department of Physics, M.L.K.(P.G.) College, Balarampur, held on 17 & 18 December 2022.
3. **Oral presentation** at "International Conference of Advancement in Functional Materials (ICAFM)" on 8-10 February 2024, organized by the Department of Institute of Physical Science for Study and Research, Veer Bahadur Singh Paranuchal University, Jaunpur, U.P. India.
4. **Oral presentation** at "National Conference on Recent Development in Physical Science (2024)" 15-16 February 2024, organized by the Department of Physics, School of Physical and Decision Science, Babasaheb Bhimrao Ambedkar University, Lucknow, U.P. India.
5. **Participated** at "International webinar on nanoscience and technology (IWNN)" 2020, organized by the Department of Physics, School of Physical and Decision Science, Babasaheb Bhimrao Ambedkar University, Lucknow, held on 27-29 November 2022.
6. **Participated at** "Synergistic Training program Utilizing the Scientific and Technological Infrastructure (STUTI) organized by the Department of Physics and University Sophisticated Instrumentation Center (USIC) at BBAU Lucknow, held on 22-28 August 2022.
7. **Participated in Webinar on "**Latest research on Additive Manufacturing & Organic Polymers" 26 July 2024 organized by EduCare Taiwan.