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RESEARCH ARTICLE

PERFORMANCE OF DMMP SENSING USING SILVER-MODIFIED ZEOLITE/POLYPYRROLE COMPOSITE MATERIALS

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ABSTRACT

We present the hydrothermal synthesis of Linde Type L zeolite and the preparation of its composite through chemical polymerization for gas-sensing applications. The main objective of this work is to improve the sensing performance of the zeolite-based nano-composite. The resulting materials were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), and Fourier-transform infrared spectroscopy (FTIR). XRD patterns confirmed the high crystallinity of the zeolite and Ag-modified zeolite, while the broad peaks observed for polypyrrole indicated its amorphous nature. SEM images revealed that incorporating polypyrrole into the zeolite matrix altered the morphology, shifting from a rectangular structure to more spherical features. This modification reduced particle size and increased the surface area, contributing to enhanced sensing capability. The gas-sensing performance of the composite toward DMMP was evaluated using copper electrodes coated with the material through a drop-casting approach. Key sensing parameters—including response and recovery times, sensitivity, and selectivity—were systematically examined. Overall, the nanocomposite exhibited excellent sensing performance across various concentrations of DMMP vapors.

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INTRODUCTION

The detection of hazardous chemicals and toxic gases is a major concern across defense, industrial safety, and environmental monitoring sectors. Among these substances, dimethyl methyl phosphonate (DMMP) is of particular interest because it is widely used as a simulant for nerve agents. DMMP is a colorless, volatile, and flammable organophosphate ($C_4H_{10}O_3P$) with a faint fruity odor. Although it is commonly employed in evaluating protective equipment such as respirators and gas masks, it is also utilized in the manufacture of plasticizers, flame retardants, and certain pesticides. Despite these applications, DMMP poses considerable health risks; exposure through inhalation, ingestion, or skin contact can lead to irritation, respiratory distress, headaches, and, at high levels, severe toxicity. As a result, the development of sensing systems with high sensitivity and selectivity toward DMMP is crucial for safeguarding both public health and the environment (1–2). In recent years, composite materials have attracted increasing attention for gas detection because they often offer enhanced sensitivity, stability, and selectivity compared to single-component systems. Among these, zeolites and polypyrrole (PPy) have emerged as promising sensing materials for monitoring hazardous vapors. Zeolites are crystalline aluminosilicates composed of interconnected SiO_4 and AlO_4 tetrahedra, forming open-framework structures with uniform micropores. Their large surface area, tunable pore architecture, and ion-exchange properties make them ideal candidates for adsorption-based sensing applications (3–4). Linde Type L (LTL) zeolite, in particular, possesses a hexagonal structure built from TO_4 (T = Si or Al) tetrahedra, resulting in a framework containing cancrinite-type cages and channels. The incorporation of aluminum introduces negative charges into the framework, which are balanced by cations residing within the pores (5). Owing to this unique architecture, LTL zeolite has been utilized in a wide range of applications, including ion exchange and molecular separation (6), catalytic processes (7), photonic and antenna materials (8), photosensitizers for optoelectronic devices (9), luminescent solar concentrators (10), color-modulating materials (11), microlasers (12), and biomedical technologies (13). Conductive polymers such as polypyrrole have similarly gained prominence due to their excellent thermal stability, environmental robustness, and adjustable electrical conductivity (14–16). Their conductivity stems from a conjugated π-electron system, which allows charge transport along the polymer chain and makes these materials appealing to diverse research fields (17–18). PPy has been extensively investigated for use in electronic devices, sensors, energy-storage systems, actuators, antistatic coatings, and biomedical applications (19–23). It can be synthesized through a variety of routes, most commonly via electrochemical methods (24–25) or chemical oxidative polymerization of pyrrole monomers (26–27), in both aqueous and organic media. The ease of synthesis, tunable conductivity, and strong interaction with various chemical species make polypyrrole an especially attractive material for chemical sensing. In this study, we present the synthesis and detailed characterization of a silver-modified zeolite/polypyrrole composite designed for high-sensitivity DMMP sensing. The composite was produced through in-situ polymerization of pyrrole in the presence of zeolite and silver ions, enabling uniform incorporation of the conductive polymer and metal species. The prepared material was subsequently examined using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and energy-dispersive X-ray analysis (EDAX) to evaluate its structural and morphological features.

MATERIALS AND METHODS

Synthesis of Silver modified Zeolite-Linde Type L: To synthesize zeolite LTL, 10.13 g of potassium hydroxide and 5.27 g of aluminum hydroxide were dissolved in 16.66 ml of double-distilled water, and the resulting solution was heated until it became clear (Solution A). After cooling to room temperature and correcting for water loss due to heating, 50.08 ml of silica sol, 4.83 g of magnesium nitrate solution, and 33.0 ml of water were mixed in a separate beaker and stirred for 3 minutes to form a homogeneous solution (Solution B). Solutions A and B were then combined and stirred until a thick gel was formed, to which 8.33 ml of water was added. The resulting gel was transferred to a Teflon-lined autoclave and heated at 175°C for 48 hours. After cooling, the material was centrifugedat 10,000 rpm, washed until its pH value reached 9, and dried in an oven at 150°C for 16 hours. The dried sample was ground into a fine powder and calcined at 540°C for 7 hours. Finally, the zeolite LTL was modified through an ion exchange process, where 1 g of zeolite and 0.25 g of $Ag(NO)_3$ were mixed in 50 ml of double-distilled water and stirred at 50°C for 4 hours in the dark. The resulting solution was filtered, washed with double-distilled water, dried at 110°C for 16 hours, and calcined at 550°C for 4 hours.

Preparation of composites: The process involved in synthesizing the composite material is as follows: A solution of Pyrrole 0.1 M is prepared in deionized water (DI), and 1 g of zeolite Ag-LTL is added while stirring continuously for 15 minutes. The resulting mixture is sonicated for 10 minutes before slowly adding a dropwise 0.1 M $FeCl_3$ solution and stirring for 2 hours. To stop the polymerization reaction, 15 ml of acetone is added to the mixture, and the resulting composite is filtered, washed with double-distilled water, and then dried at 50^{o}C for 24 hours in the oven.

Characterization techniques: The scanning electron microscope (SEM) with energy dispersive X-ray spectroscopy (EDS) attachment (JEOL JSM 5800) was used to investigate the surface morphology of the synthesized composites. The elemental analysis of the composites was performed using the Oxford INCA energy dispersive X-ray spectroscopy (EDS) attached to the SEM. The Fourier-transform infrared (FTIR) study was conducted using SHIMADZU FT-IR equipment. The powder X-ray diffraction (XRD) pattern of the composites was recorded using Shimadzu XRD 6000 equipment.

RESULT AND DISCUSSION

XRD Analysis: X-ray diffraction (XRD) analysis was performed using a Shimadzu XRD-6000 instrument after loading the samples onto an aluminum sample holder. Measurements were carried out using Cu Kα radiation operated at 40 kV and 30 mA, with scans recorded over a 2θ range of 20–70°. The diffraction patterns were evaluated primarily for basal reflections along the c-axis.

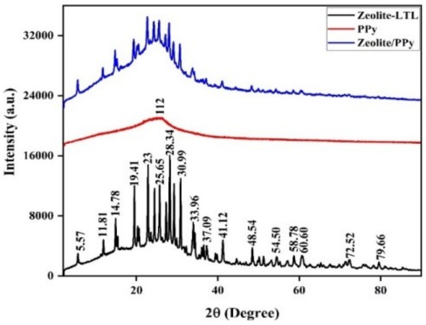

Figure 1. XRD spectra of a) Ag form of zeolite LTL b) PPy and c) PPy-LTL composite

Figure 1 presents the powder XRD profiles of calcined LTL zeolite, Ag-exchanged LTL (Ag-LTL), and the PPy-LTL composite. The LTL and Ag-LTL samples exhibit well-defined crystalline patterns, confirming the high structural order of the framework. In contrast, the composite shows reduced peak intensity due to the inherent amorphous character of polypyrrole. Notably, the characteristic reflection of LTL near 2θ ≈ 28.34° remains visible, indicating that the framework remains stable after calcination at 550 °C for 5 hours in dry air. The similarity between the diffraction patterns of pristine LTL and Ag-exchanged samples suggests that ion incorporation did not significantly disturb the zeolite lattice, and the slight variations in peak intensity reflect the accommodation of metal ions within the supercages without imposing structural strain. The preserved peak positions further indicate that the ion-exchange process did not compromise the crystalline framework. XRD patterns of zeolite, PPy, and their composites additionally demonstrate that the introduction of polypyrrole influences the overall intensity due to the amorphous nature of the polymer chains. While pure PPy exhibits broad amorphous peaks, the presence of zeolite improves polymer chain organization, leading to enhanced diffraction features. The retention of characteristic zeolite reflections in the composite confirms that the ordered host structure remains intact during synthesis. The observed decrease in intensity relative to pure zeolite is attributed to reduced scattering contrast caused by PPy occupying the microporous channels.

FTIR Spectroscopy Analysis: FTIR spectroscopy was employed to examine the structural features and functional groups present in the zeolite samples, as well as to identify potential adsorption sites. The measurements were performed using a SHIMADZU FT-IR spectrophotometer, with each sample placed in a standard sample holder. Spectra were collected in the 4000–500 cm^{-1} range at a resolution of 4 cm^{-1}.

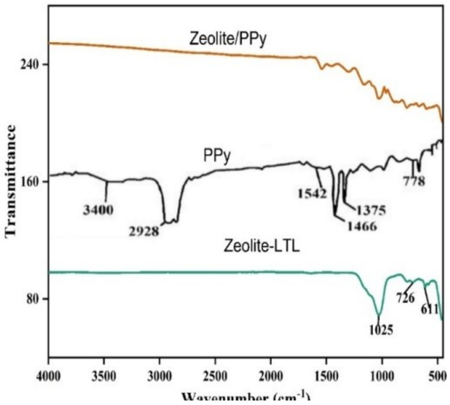

Figure 2. FTIR spectra of a) Ag-LTL Zeolite, b) PPy and Ag-LTL Zeolite/PPy

Figure 2 shows the FTIR spectra of LTL zeolite, polypyrrole (PPy), and the PPy–LTL composite. In the zeolite spectrum, the band near 550 cm^{-1} corresponds to Si–O and Al–O bending vibrations of the framework. Additional features at approximately 1000 cm^{-1} and 750 cm^{-1} are associated with the asymmetric and symmetric stretching modes of the tetrahedral units. The band around 1400 cm^{-1}, attributed to bending vibrations of adsorbed water molecules, confirms the presence of structural water within the zeolite. A broad absorption near 3500 cm^{-1} represents O–H stretching vibrations. The incorporation of polypyrrole into the LTL framework is supported by the appearance of characteristic PPy absorption bands in the composite spectrum. The Ag-LTL sample exhibits prominent features at 611, 726, and 1025 cm^{-1}. Pure PPy shows distinct bands at 788, 1375, 1466, 1542, 2928, and 3400 cm^{-1}, corresponding to C–H bending, quinoid ring vibrations, C–N stretching, benzenoid ring stretching, N–quinoid ring stretching, and N–H stretching modes, respectively. These characteristic peaks, along with their modified shapes in the composite, indicate strong interactions between PPy and the zeolite framework.

SEM and EDAX Analysis: The surface morphology of calcined zeolite LTL, PPy, and PPy-LTL composite was analyzed using a JEOL JSM 5800 SEM EDAX instrument. Scanning electron micrographs were taken to study their surface and crystal morphology, which are displayed in Figure 3.

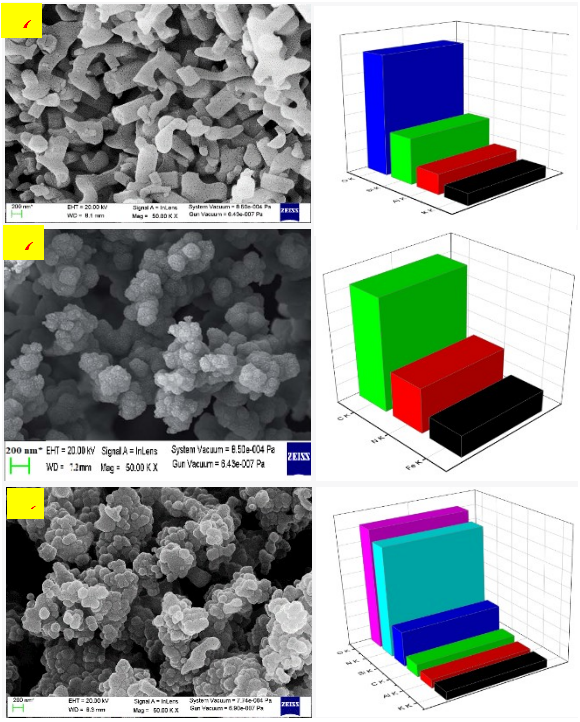

Figure 3: SEM and EDAX of the a) Ag-LTL Zeolite, b) Polypyrrole and c) Ag-LTL/PPy composite

The surface morphology of calcined zeolite LTL, PPy, and the PPy–LTL composite was examined using a JEOL JSM 5800 SEM equipped with EDAX. Scanning electron micrographs were captured to evaluate the surface features and crystal structures of the samples, as shown in Figure 3. The SEM images indicate that zeolite LTL exhibits a non-uniform surface with slightly irregular pore structures and variations in particle size, with individual particles displaying a hexagonal-like shape. The micrographs of the PPy-based composites show similar structural features, suggesting that the polymer forms a consistent framework surrounding the incorporated zeolite particles. To obtain more detailed images of the zeolite phase, a composite containing a higher proportion of zeolite was used for the SEM study. The images reveal that the natural alignment of zeolite crystals is reduced within the composite due to the amorphous nature of the polymer matrix, providing useful insight for interpreting zeolite-related surface features. The elemental composition of zeolite LTL, PPy, and the PPy–LTL composite was further analyzed using energy-dispersive X-ray spectroscopy (EDX) with the Oxford INCA system. Before analysis, the samples were coated with a thin layer of gold to minimize charging effects. The corresponding spectra, shown in Figure 3, confirm the incorporation of metal ions into the zeolite structure through ion exchange, as well as the characteristic elemental composition of polypyrrole. Additionally, the unit cell formula of zeolite LTL is identified as $K_9(Al_9Si_{27}O_{72})\cdot H_2O$.

Gas Sensing Studies: In this study, a PPy/zeolite LTL–based sensor was developed and evaluated for its response to varying concentrations of ammonia gas (0.5, 1, and 2 ppm). When exposed to ammonia, the resistance of the Ag-LTL/PPy composite increased. This behavior is attributed to the formation of ammonium ions (NH_4^+) at the N^+–H adsorption sites, which reduces the density of positive charge carriers and consequently raises the material's resistance. To further assess sensing performance, 1 ppm of DMMP was introduced into a 1 L gas chamber, and the influence of temperature—ranging from 100°C to 250°C—on the sensor's response was examined. The results showed that the sensor's response strengthened with increasing temperature, reaching its highest value at 250°C. The composite also demonstrated an effective operating temperature window of 150–250°C and exhibited rapid dynamics, with response and recovery times of 21 s and 24 s, respectively, at 0.5 ppm. These observations indicate that the fabricated sensor is capable of detecting DMMP vapor at elevated temperatures with high sensitivity and quick response characteristics. The corresponding results are presented in Figure 4 (a and b).

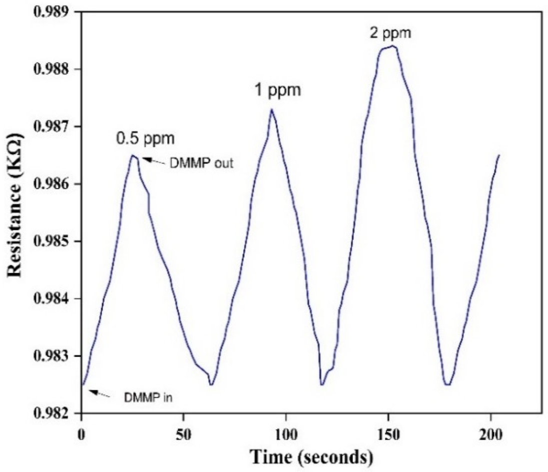

Figure 4(a): Response of sensor toward DMMP for Zeolite Ag-LTL/PPy composite for Different Concentration

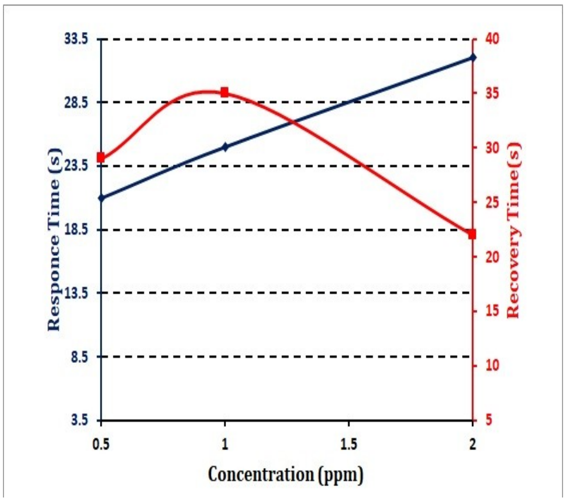

Figure 4(b). Graph between Response times, and recovery time with the Concentration

CONCLUSION

The primary aim of this study is to evaluate the sensing performance of a silver-modified zeolite/polypyrrole (PPy) composite for detecting DMMP. To improve PPy's sensitivity toward DMMP, zeolite LTL was directly incorporated during synthesis. The PPy/zeolite composite was prepared through the polymerization of polypyrrole in the presence of ion-exchanged, silver-modified zeolite. Sensor response was found to increase with rising DMMP concentration. Additionally, the influence of key parameters—such as response time and recovery time—on overall sensor performance was examined. Developing highly sensitive DMMP sensors is crucial for protecting both human health and the environment. The silver-modified zeolite/PPy composite demonstrated strong potential as a DMMP-responsive material, suggesting that further optimization could enhance its capabilities and expand its applicability to other hazardous analytes. In this study, the composite exhibited response and recovery times of 21 s and 24 s, respectively, at a DMMP concentration of 0.5 ppm.

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