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Sensors based on track-etched membranes and HKUST-1 for ammonia detection

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Abstract

Hybrid colorimetric sensor membranes based on PET track-etched membranes and the copper-based metal–organic framework HKUST-1 were developed for ammonia detection. PET track-etched membranes with an average pore diameter of approximately 300 nm were used as porous supports, whereas PVC/HKUST-1 nanofibrous layers were deposited onto their surface by electrospinning. The obtained PVC/HKUST-1@PET track-etched membranes exhibited a clear visual response to ammonia, changing color from light blue to dark blue after exposure. This colorimetric effect is attributed to the interaction of ammonia molecules with copper centers in HKUST-1 and the corresponding changes in the coordination environment of copper ions. The sensing response was confirmed by Fourier-transform infrared (FTIR) spectroscopy, which revealed characteristic bands associated with N–H vibrations and ammonium species after ammonia adsorption. UV–Vis spectroscopy showed an increase in absorption intensity in the 576–610 nm region with increasing ammonia concentration. The membranes demonstrated a linear response in the concentration range from 0.001 to 0.8 M, with a limit of detection of 0.0064 M. The repeatability of the sensor response was evaluated at an ammonia concentration of 0.01 M, giving a relative standard deviation of 24%. The results indicate that PVC/HKUST-1@PET track-etched membranes are promising materials for simple, visual, and rapid ammonia detection, with potential applications in environmental monitoring and smart food packaging.

1 Introduction

Ammonia detection is an important analytical task in environmental monitoring, industrial safety, healthcare, agriculture, and food quality control. Ammonia is widely used in chemical production, fertilizer manufacturing, refrigeration systems, and other technological processes. However, excessive ammonia concentrations can be harmful to human health and the environment. In addition, ammonia is one of the volatile nitrogen-containing compounds formed during the decomposition of protein-rich food products, including meat, seafood, cheese, and other perishable goods. Therefore, the development of simple, sensitive, selective, and visually readable materials for ammonia detection remains a relevant scientific and practical challenge [1–3].

A wide range of ammonia sensors has been reported, including metal oxide semiconductor sensors, conducting polymer-based sensors, electrochemical sensors, optical sensors, paper-based sensors, and metal–organic framework-based sensing materials [1, 4, 5]. Metal oxide semiconductors, such as ZnO, SnO_2, WO_3, TiO_2, and In_2O_3, have been widely investigated due to their high sensitivity and relatively simple fabrication [4]. Their sensing mechanism is usually based on changes in electrical resistance caused by the interaction of ammonia with adsorbed oxygen species on the oxide surface. However, many metal oxide sensors require elevated operating temperatures, which increases power consumption and limits their use in portable or disposable sensing platforms.

Conducting polymers, such as polyaniline, polypyrrole, and PEDOT:PSS, have also attracted considerable attention for ammonia detection because they can operate at room temperature and show a strong electrical response to electron-donating ammonia molecules [6]. In these systems, ammonia usually causes deprotonation or changes in charge transport within the polymer matrix, leading to measurable changes in conductivity. Despite these advantages, conducting polymer sensors may suffer from limited long-term stability, humidity interference, and signal drift. Electrochemical sensors, in turn, can provide high sensitivity and quantitative detection; however, their practical application often requires electrodes, electrolytes, calibration procedures, and additional electronic components.

Among different approaches to ammonia sensing, optical and colorimetric sensors are of particular interest because they enable rapid detection without complex analytical equipment. A visible color change provides a simple and convenient signal that can be detected by the naked eye, which is especially important for smart food packaging, portable test systems, and on-site monitoring [2, 3, 7]. Various colorimetric ammonia sensors have been developed using pH-sensitive dyes, natural indicators, polymer films, paper substrates, and porous hybrid materials. For example, indicator-based materials have been proposed for intelligent food packaging, where the color change is associated with the alkaline nature of ammonia released during food spoilage [3, 7, 8]. However, dye-based sensors may show limited chemical stability, leaching of the indicator, and insufficient selectivity in complex media.

Metal–organic frameworks (MOFs) are promising candidates for chemical sensing due to their high specific surface area, tunable pore structure, and the presence of active metal sites. MOF-based gas sensors can operate through different transduction mechanisms, including chemiresistive, capacitive, impedimetric, mass-sensitive, and optical responses [5, 9, 10]. In the case of ammonia detection, MOFs are particularly attractive because ammonia can interact with coordinatively unsaturated metal

centers, functional groups of organic linkers, and confined pore environments. Several MOF-based ammonia sensors have demonstrated high sensitivity and selectivity. In particular, MOF-based sensing materials can exhibit strong responses toward ammonia, including optical or visually detectable changes associated with interactions between ammonia molecules and metal centers [5, 9, 10]. These results confirm that MOFs are promising platforms for the development of both electronic and colorimetric ammonia sensors.

HKUST-1, also known as $Cu_3(BTC)_2$, is one of the most widely studied copper-based MOFs. Its structure consists of copper paddle-wheel units connected by benzene-1,3,5-tricarboxylate linkers, forming a porous framework with accessible copper sites. These copper centers can interact with electron-donating molecules, including ammonia. Previous studies have shown that ammonia adsorption on HKUST-1 is associated with the coordination of NH_3 molecules to copper sites, the possible formation of copper–ammonia complexes, and structural changes of the framework under humid conditions [8, 11–13]. In particular, spectroscopic studies have demonstrated that ammonia and water can cooperatively interact with HKUST-1, influencing both the adsorption mechanism and the stability of the material [11]. These features make HKUST-1 a suitable active component for ammonia-sensitive materials.

At the same time, the practical use of powdered MOFs in sensor devices is often limited by difficulties in handling, poor mechanical stability, possible aggregation, and the need for immobilization on a suitable support. To overcome these limitations, MOFs are commonly incorporated into polymer matrices, deposited as thin films, or immobilized on porous substrates. Mixed-matrix systems containing HKUST-1 have been investigated for ammonia capture and sensing, showing that supporting matrices can improve the processability and practical applicability of MOF-based materials [14, 15]. Therefore, the development of stable membrane-supported MOF systems is an important direction for transforming powdered MOFs into functional sensing platforms.

PET track-etched membranes (PET TeMs) are attractive substrates for this purpose because of their well-defined pore structure, chemical stability, low thickness, mechanical flexibility, and possibility of surface modification [16, 17]. Their porous architecture can provide efficient mass transfer of analyte molecules, while the membrane format is convenient for integration into sensing platforms, filters, packaging materials, and portable devices. In addition, TeMs can serve as stable supports for the deposition of polymer nanofibers and functional porous materials [17].

In this work, PET track-etched membranes were used as porous supports for the fabrication of colorimetric sensing materials based on PVC nanofibers and HKUST-1. PVC/HKUST-1 layers were deposited onto the membrane surface by electrospinning, followed by additional decoration with HKUST-1 to increase the amount of active MOF on the membrane. This approach makes it possible to combine the structural advantages of TeMs, the flexibility of polymer nanofibers, and the ammonia-sensitive properties of HKUST-1. The obtained PVC/HKUST-1@PET track-etched membranes were evaluated as simple visual sensor materials for ammonia detection.

2 Experimental Part

2.1 Materials

Copper(II) nitrate trihydrate $Cu(NO_3)_2 \cdot 3H_2O$ (98%), benzene-1,3,5-tricarboxylic acid (BTC, 95%), poly(vinyl chloride) (PVC), N, N-dimethylformamide (DMF, 99.9%), ethanol (99.9%), and tetrahydrofuran (THF, 99.9%) were purchased from Sigma–Aldrich. Deionized water with a resistivity of 18.2 M$\Omega \cdot$ cm was used for all sample preparation procedures.

2.2 Preparation of PET Track-Etched Membranes

PET track-etched membranes (PET TeMs) were prepared by irradiation of Hostaphan® PET films with a thickness of 12 μm (Mitsubishi Polyester Film, Germany) using Kr ions with an energy of 1.75 MeV/nucleon at the DC-60 ion accelerator of the Astana Branch of the Institute of Nuclear Physics, Kazakhstan. The irradiated polymer films were chemically etched in a 2.2 M NaOH solution at 85 °C. After etching, the samples were rinsed with acetic acid and deionized water and then stored in air at room temperature. The resulting pore diameter of the PET TeMs was approximately 320 ± 10 nm.

2.3 Preparation of Colorimetric Membranes

The colorimetric membranes were produced by electrospinning according to the procedure described in [17], where the characterization methods for the obtained membranes are also presented. Briefly, HKUST-1 powder was dispersed in a DMF:THF solvent mixture with a volume ratio of 1:1 and ultrasonicated for 40 min at 80% power. The suspension was heated to 45 °C, after which PVC was added and stirred until complete dissolution.

During electrospinning, the hybrid solution was supplied using a syringe pump equipped with a 10 mL syringe at a flow rate of 0.5 mL/h. The applied voltage was maintained at 15.5 kV, and the distance between the needle tip and the PET TeM collector was 15 cm. After electrospinning, the obtained PVC/HKUST-1@PET TeMs were dried at 60 °C. Then, secondary decoration of the membranes with HKUST-1 was carried out to increase the MOF content on the surface of the colorimetric membranes.

2.4 Characterization

Fourier-transform infrared (FTIR) spectra of the samples were recorded using an InfraLUM FT-08 spectrometer equipped with a variable-angle specular reflectance accessory, VeeMAX™ III (PIKE Technologies, USA). The spectra were collected in the range of 400–4000 cm^{-1} with 25 scans, an incidence angle of 30°, and a spectral resolution of 2 cm^{-1} in order to evaluate chemical changes in the membranes.

The pore size of the PET TeMs was determined by the gas flow rate method at a pressure of 20 kPa.

2.5 Ammonia Sensing

Aqueous ammonia solutions with concentrations from 0.001 to 0.8 M were prepared. Colorimetric membrane samples with dimensions of 1×1 cm were immersed in the ammonia solutions for 10 min. After exposure, the membranes were removed from the solutions, and their optical response was analyzed using UV–Vis spectroscopy in the wavelength range of 190–900 nm.

3 Results and Discussion

The detection of ammonia is based on its interaction with the individual building blocks of HKUST-1, namely copper ions and benzene-1,3,5-tricarboxylate (BTC) linkers. In aqueous solution, ammonia acts as a weak base and generates hydroxide ions. In the presence of Cu^{2+} ions, this may initially lead to the formation of copper hydroxide, whereas at higher ammonia concentrations deep-blue ammine copper complexes, such as $[Cu(NH_3)_4]^{2+}$, can be formed. Carboxylic acid groups can also interact with ammonia to form ammonium carboxylate salts, which may be further converted into the corresponding amides under appropriate conditions [11]. A simplified scheme of ammonia adsorption and complex formation involving HKUST-1 can be represented as follows [14]:

$$NH_3 + H_2O \rightleftharpoons NH_4^+ + OH^-, \tag{1}$$

$$NH_3 + R{-}COOH \rightleftharpoons NH_4^+ + R{-}COO^-, \tag{2}$$

$$6NH_3 + Cu_3(BTC)_2 \rightarrow Cu_3(NH_3)_6(BTC)_2. \tag{3}$$

An alternative mechanism of the interaction between ammonia and HKUST-1 has also been proposed in the literature [12]. Under dry conditions, HKUST-1 can interact with ammonia with the formation of presumed diammine-copper(II) complexes. Under humid conditions, the formation of $Cu(OH)_2$ species and $(NH_4)_3BTC$ has been reported. X-ray diffraction (XRD) and NMR studies of fresh and ammonia-treated samples showed continuous changes in the structure and porosity of HKUST-1, with the most pronounced transformations occurring after ammonia treatment under humid conditions.

The structure and morphology of the obtained PVC/HKUST-1@PET membranes were described previously [17]. The resulting membranes exhibit a visually detectable color response, which can be easily observed by the naked eye. Such behavior is promising for practical applications, including smart food packaging [7]. Food spoilage and food poisoning remain serious problems affecting human health; therefore, spoiled products should be detected as early as possible [2]. Among the major volatile nitrogen-containing compounds formed during spoilage, ammonia is especially important because its concentration increases during the degradation of protein-rich products such as meat, seafood, cheese, and related food products [3]. Thus, the development of sensor materials that can be integrated into packaging and provide a visually detectable color change is highly relevant [9, 15].

The obtained PVC/HKUST-1@PET membranes had a blue coloration and can be used as sensing materials due to their ability to change color upon contact with

ammonia. After exposure to ammonia, the membrane color changed from blue to dark blue, as shown in Figure 1. This color change can be attributed to the interaction of ammonia molecules with the copper centers of HKUST-1 and the corresponding changes in the coordination environment of Cu^{2+} ions.

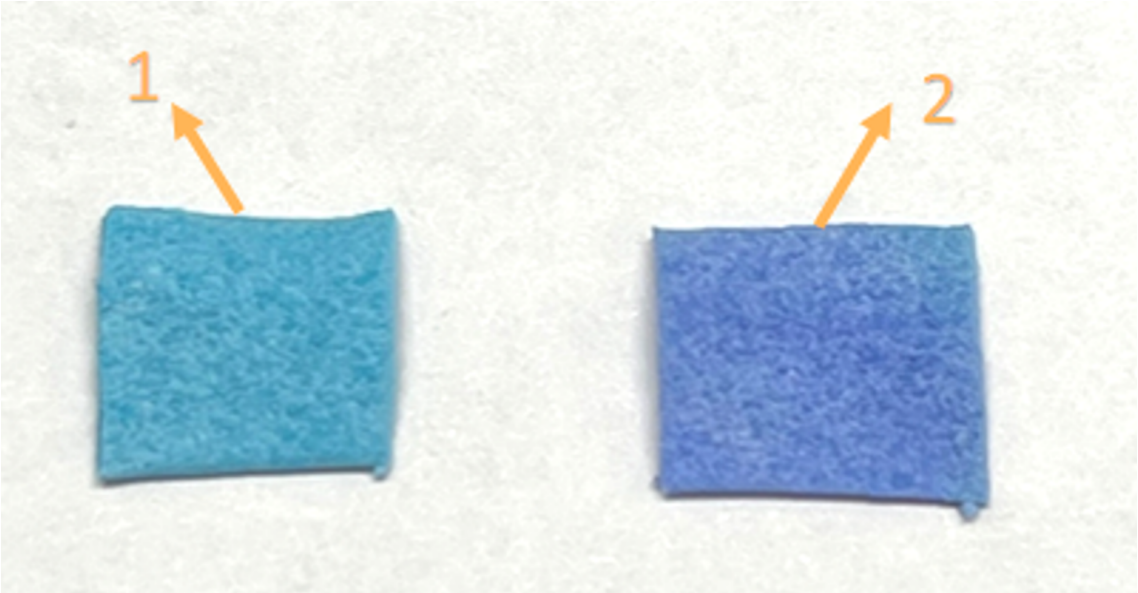

Figure 1: Colorimetric response of the PVC/HKUST-1@PET membrane: (1) before and (2) after interaction with ammonia.

To confirm the visual colorimetric response, the membranes were analyzed by ATR-FTIR spectroscopy. The ATR-FTIR spectra of the membranes before and after interaction with ammonia are shown in Figure 2. The initial PVC/HKUST-1@PET membrane exhibits characteristic absorption bands associated with the polymer matrix and HKUST-1 framework. After exposure to ammonia, noticeable changes appear in the spectrum, indicating the adsorption of ammonia and its interaction with the active sites of the membrane.

Several new bands appeared at approximately 3330 and 1550 cm^{-1} after ammonia treatment. These bands can be assigned to vibrations of NH_4^+ species formed as a result of acid–base interaction between ammonia and acidic groups of the BTC linker. The bands at 1610, 1430, and 1210 cm^{-1} are attributed to N–H vibrations. The appearance and increase in intensity of these bands confirm that ammonia molecules are retained in the membrane structure after exposure.

The ammonia-sensing ability of the membranes can be explained by the interaction between ammonia molecules and copper centers in HKUST-1 through the lone electron pair of nitrogen. This interaction changes the local coordination environment of Cu^{2+} ions and can affect both the vibrational spectrum and optical properties of the membrane. In addition, neutralization of acidic BTC groups by ammonia and possible hydrogen-bond formation between –COOH groups and NH_3 molecules may also contribute to ammonia adsorption and the observed spectral changes [12].

Thus, the ATR-FTIR results support the proposed colorimetric sensing mechanism. The spectral changes observed after ammonia exposure are consistent with the visual transition of the membrane from blue to dark blue, suggesting that the response is governed by a combination of ammonia coordination to copper sites and acid–base interactions involving BTC linker groups.

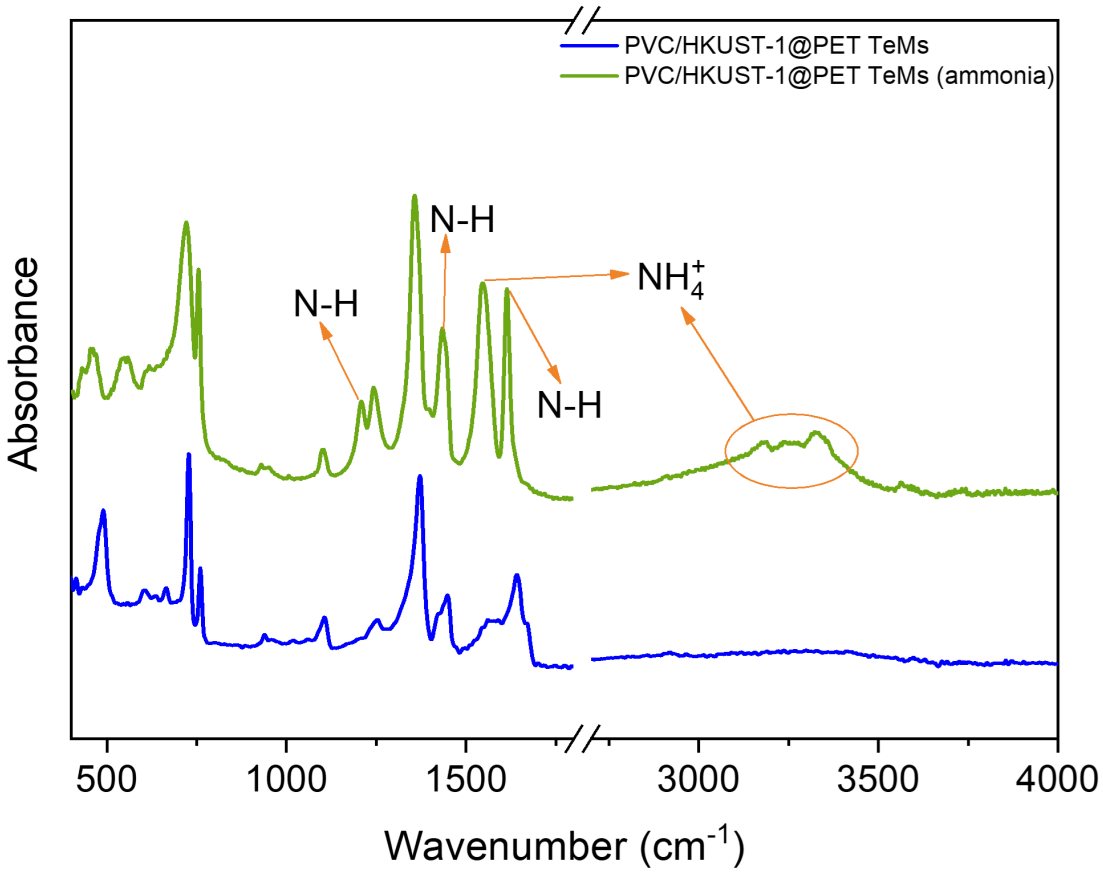

Figure 2: ATR-FTIR spectra of the PVC/HKUST-1@PET membrane before (1) and after (2) interaction with ammonia.

Figure 3 shows the UV–Vis spectra of aqueous ammonia solutions with concentrations ranging from 0.001 to 0.8 M after interaction with the PVC/HKUST-1@PET membrane. The measurements were performed 5 min after exposure of the membranes to ammonia. As shown in Figure 3, the absorption band in the 576–610 nm region gradually increases with increasing ammonia concentration. In addition, band splitting is observed at higher ammonia concentrations, particularly at 0.4 and 0.8 M. According to the literature, such spectral changes can be associated with changes in the coordination environment of copper ions in the membrane [10, 18]. The initial absorption band is related to ligand-field transitions of Cu^{2+} centers in HKUST-1. After exposure to ammonia, the observed color change and evolution of the UV–Vis spectra can be attributed to the coordination of ammonia molecules to copper centers, which changes the local ligand-field environment of Cu^{2+} ions. At higher ammonia concentrations, the splitting and broadening of the absorption band may also indicate partial structural transformation of HKUST-1 under alkaline conditions and the formation of copper–ammonia complexes. These processes are consistent with the visual color change of the membrane from blue to dark blue after ammonia exposure.

Figure 4 presents the calibration curve showing the dependence of absorbance intensity on ammonia concentration. A linear increase in absorbance intensity was observed in the studied concentration range from 0.001 to 0.8 M. The linear fitting gave a correlation coefficient of $R^2 = 0.90979$, indicating a concentration-dependent optical response of the PVC/HKUST-1@PET membrane toward ammonia. The

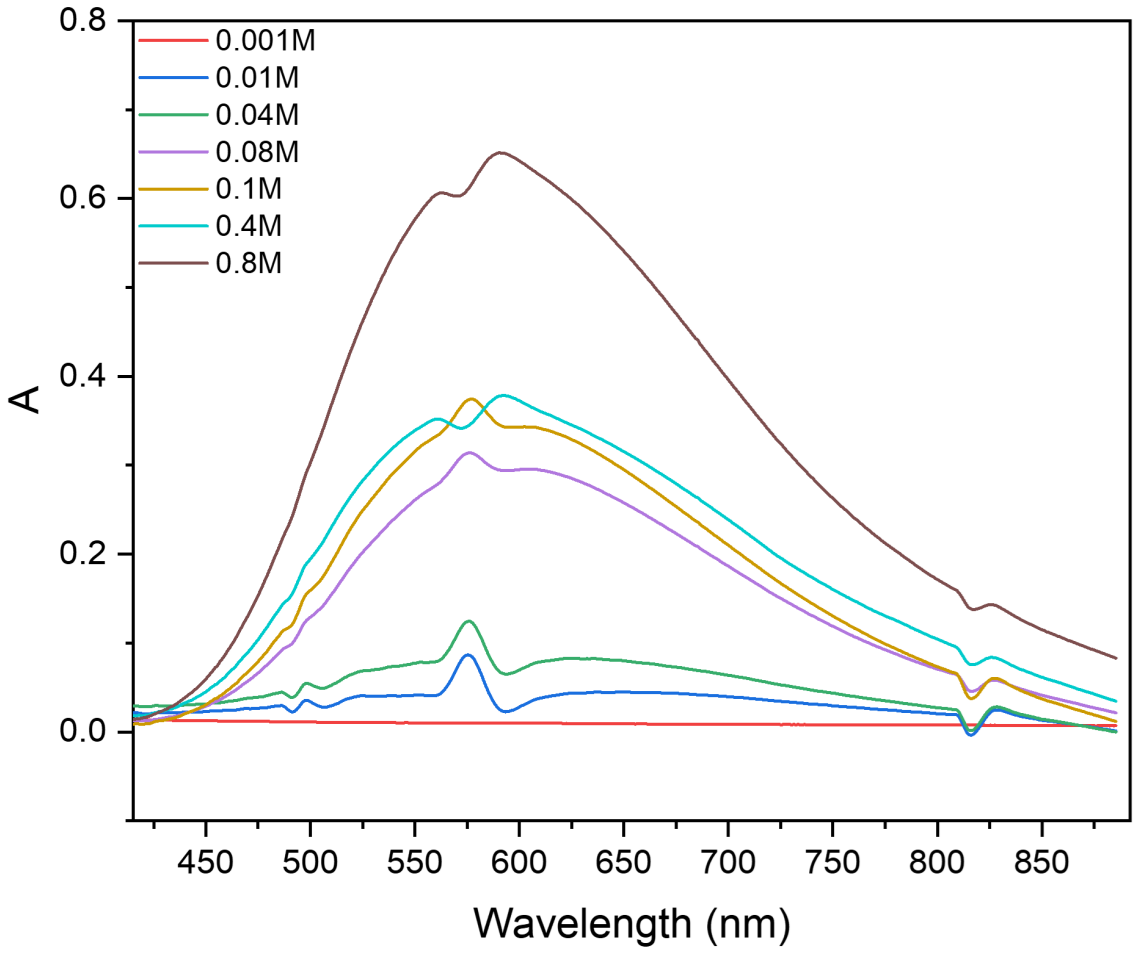

Figure 3: UV–Vis spectra of ammonia solutions in the concentration range from 0.001 to 0.8 M after interaction with the PVC/HKUST-1@PET membrane.

limit of detection was estimated to be 0.0064 M. The inset photograph shows the visible color change of the ammonia solutions after interaction with the membrane, which is consistent with the UV–Vis data.

For the calibration curve, ammonia concentrations in the range from 0.001 to 0.8 M were investigated. The correlation coefficient was $R^2 = 0.90979$, and the limit of detection (LOD) was 0.0064 M. The repeatability of the sensor response was evaluated by measuring the absorbance at an ammonia concentration of 0.01 M in five repeated analyses. The relative standard deviation was calculated from the five measurements and was found to be 23.87%. The ammonia adsorption time was studied in the range from 1 to 120 min. Figure 5 shows photographs of the membranes after different ammonia adsorption times.

The membranes were exposed to ammonia vapor above a 0.8 M ammonia solution. The measurements were carried out using UV–Vis and FTIR spectroscopy. The UV–Vis spectra did not show significant changes. The FTIR spectra of the colorimetric membranes after interaction with ammonia vapor are presented in Figure 6. In the FTIR spectra, the intensities of the bands at 3330 and 1210 cm^{-1} change with increasing ammonia adsorption time. The corresponding values are summarized in Table 1. For quantitative comparison, the peak intensities at 3330 and 1210 cm^{-1} were determined for each adsorption time.

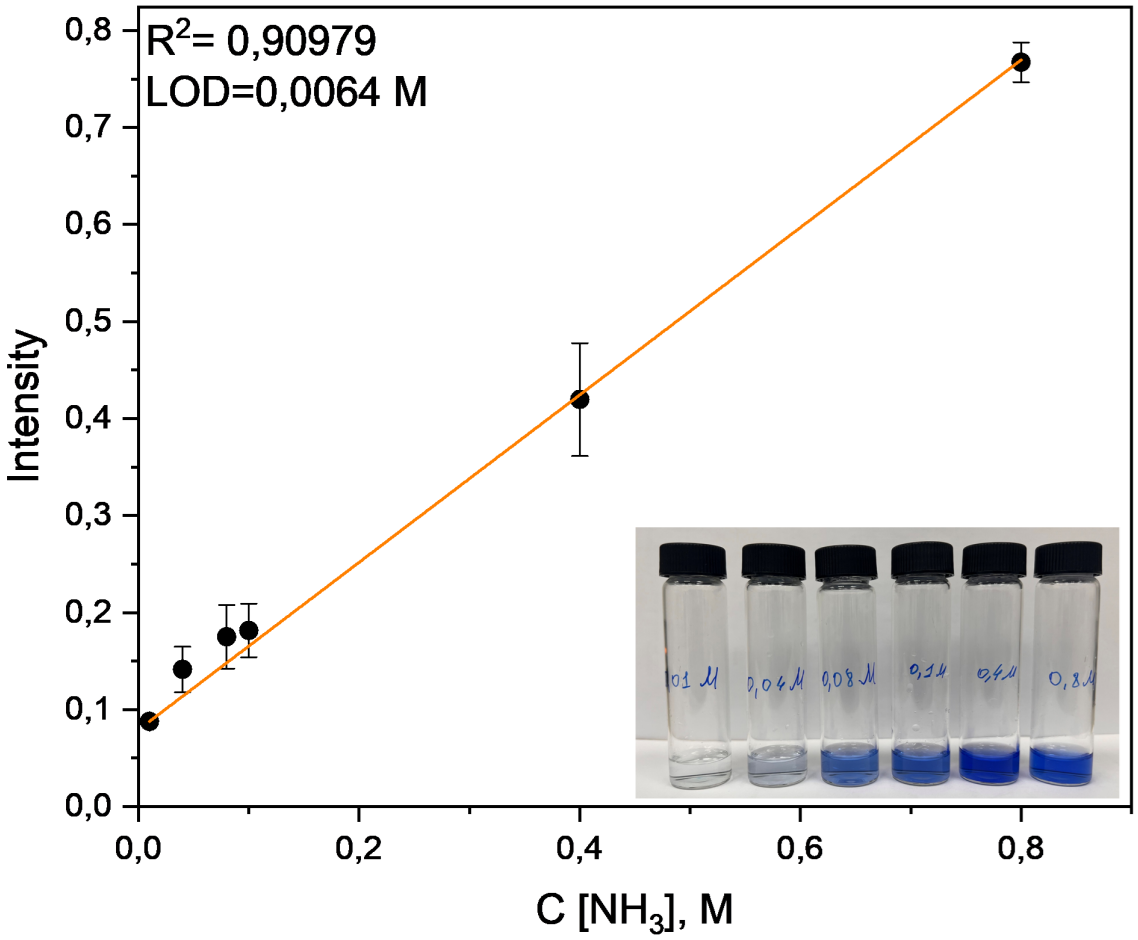

Figure 4: Calibration curve showing the dependence of absorbance intensity on ammonia concentration. The inset shows the color change of ammonia solutions after interaction with the PVC/HKUST-1@PET membrane.

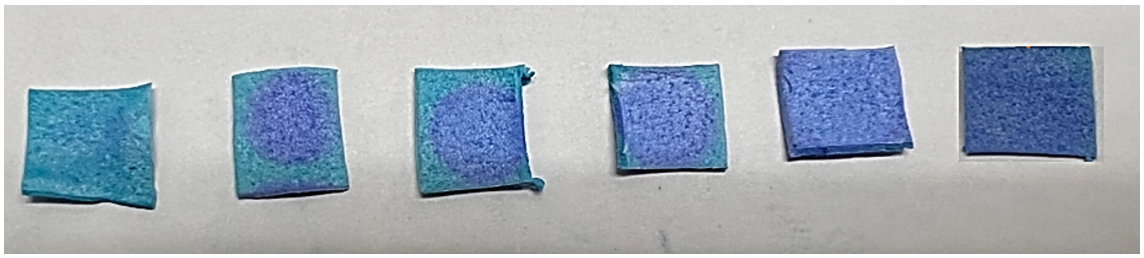

Figure 5: Photographs of the membranes after exposure to ammonia at different adsorption times from left to right: 1, 5, 15, 30, 60, and 120 min.

Since one of the potential applications of the developed colorimetric membranes is smart food packaging, the effect of humidity should also be considered. In humid environments, water molecules may compete with ammonia for coordination sites in HKUST-1 and may facilitate partial structural transformation of the framework [12]. Therefore, elevated humidity may influence both the stability of the HKUST-1 phase and the kinetics of the colorimetric response. Further studies under controlled relative humidity conditions are required to evaluate the long-term performance of PVC/HKUST-1@PET membranes in real packaging environments.

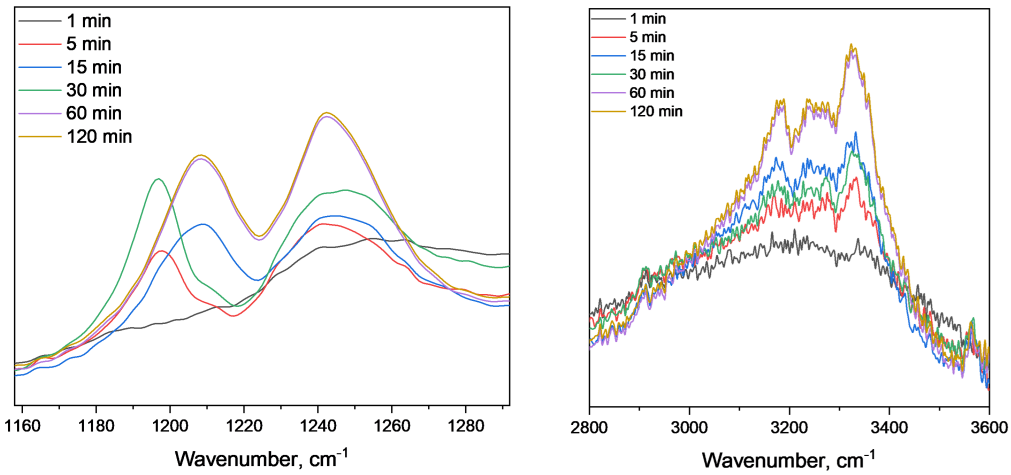

Figure 6: FTIR spectra of the membranes after ammonia adsorption over a time range from 1 to 120 min.

Table 1: Peak intensity values at different ammonia adsorption times.

Adsorption time	Intensity of NH group I_{1210}/I_{1100}	Intensity of NH_4^+ group I_{3330}/I_{1100}
1 min	1.0167	0.7631
5 min	1.4137	0.9830
15 min	1.6373	1.1362
30 min	1.7784	1.0861
60 min	1.8593	13.008

4 Conclusions

In this study, hybrid colorimetric sensor membranes based on PET track-etched membranes, PVC nanofibers, and HKUST-1 were successfully prepared and investigated for ammonia detection. PET track-etched membranes served as porous and mechanically stable supports, whereas the PVC/HKUST-1 nanofibrous layer provided active sites for interaction with ammonia molecules. The obtained PVC/HKUST-1@PET track-etched membranes demonstrated a clear visual color change from light blue to dark blue after exposure to ammonia.

The observed response is associated with the interaction of ammonia molecules with copper centers in HKUST-1, possible changes in the coordination environment of copper ions, and the formation of ammonia-related species. FTIR spectroscopy confirmed the appearance and changes of characteristic bands associated with N–H vibrations and ammonium species after ammonia adsorption. UV–Vis spectroscopy showed an increase in absorption intensity in the 576–610 nm region with increasing ammonia concentration.

The developed membranes exhibited a linear response to ammonia in the concentration range from 0.001 to 0.8 M, with a calculated limit of detection of 0.0064 M. The repeatability study showed a relative standard deviation of 23.87%, indicating that further optimization of the membrane preparation and sensing procedure is required to improve reproducibility. In future studies, this parameter may be improved by more precise control of electrospinning conditions, including solution viscosity, flow

rate, applied voltage, and collector distance, as well as by optimizing the thickness and uniformity of the PVC/HKUST-1 sensing layer. Additional standardization of membrane cutting, active layer loading, and ammonia exposure conditions may also contribute to better reproducibility of the colorimetric response.

At the same time, the visually distinguishable color change and spectroscopically confirmed response demonstrate the potential of these materials for simple and rapid ammonia detection. Overall, PVC/HKUST-1@PET track-etched membranes can be considered promising colorimetric sensor materials for ammonia monitoring. Due to their membrane format, visible optical response, and relatively simple preparation approach, these materials may be useful for applications in environmental monitoring, safety control, and smart food packaging.

Acknowledgments

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