

Simultaneous Capacitance and DRIFTS Analysis of Zeolites for Dosimeter-Based Epichlorohydrin Sensing at Room Temperature

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Summary:

This work investigates a dosimeter-based sensing concept for the detection of carcinogenic trace gases using a Cu-exchanged MFI zeolite. Simultaneous capacitance measurements and DRIFTS analysis were employed to monitor both the electrical response and molecular adsorption of epichlorohydrin under exposure at room temperature. The capacitance signal shows a concentration-dependent cumulative behavior, indicating adsorption of the analyte. DRIFT spectra confirm this interpretation by revealing characteristic bands of adsorbed epichlorohydrin, enabling dosimeter-type trace gas sensing.

Keywords:

DRIFTS Analysis, Dosimeter-based Gas Sensing, Carcinogenic Trace Gases, Zeolite Adsorption, Capacitance Measurement

Motivation

The detection of carcinogenic trace gases is highly relevant for protecting human health, as long-term exposure even at very low concentrations can exceed regulatory workplace exposure limits and present serious health risks [1]. In many applications, the critical factor for risk evaluation is the cumulative dose rather than short-term concentration peaks.

Dosimeter-type sensors are therefore particularly suitable, as they integrate the gas exposure over time and directly reflect the total amount of hazardous substances [2]. Zeolites are promising materials in this context due to their strong and selective adsorption of different gas species [3].

Combining capacitance measurements with DRIFTS analysis enables a deeper understanding of adsorption mechanisms and supports the development of reliable dosimeter-based sensing concepts for carcinogenic trace gases.

Measurement Setup

For the characterization of the zeolite material, two measurement techniques were used simultaneously. The investigated sensing film consisted of a copper-exchanged MFI zeolite with a $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio of 200, which was processed into a screen-printable paste and deposited onto an interdigital electrode (IDE) structure.

This configuration allows the determination of dielectric properties via a capacitance measurement at an excitation frequency of 32 kHz. In parallel, Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) was used to monitor adsorbed species on the zeolite. DRIFTS enables the in-situ observation of adsorption processes without extensive sample preparation [4, 5].

The schematic layout of the setup is shown in Fig. 1.

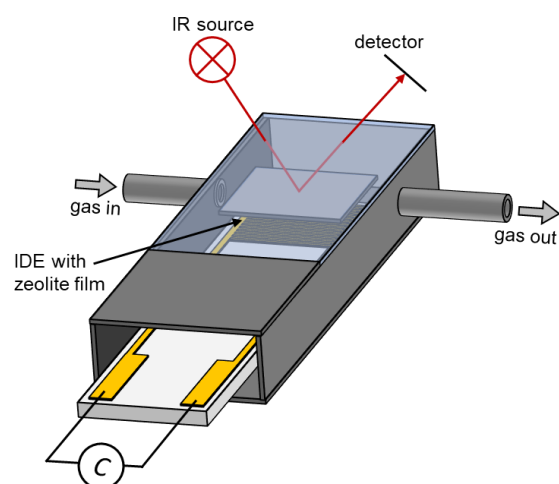


Fig. 1. Schematic diagram of the measurement setup for simultaneous capacitance measurement and DRIFTS characterization of a Cu-MFI zeolite film on an interdigital electrode structure.

The measurements were performed at room temperature using gaseous epichlorohydrin (ECH) in the low-ppm concentration range. This test gas is known to be carcinogenic. The adsorption of the analyte onto the zeolite is expected to result in both a detectable change in the adsorbed species visible in the IR spectrum and a change in the dielectric properties, which affects the capacitance measurement [6].

Results and Discussion

Fig. 1 shows a room-temperature experiment in which the epichlorohydrin (ECH) concentration was varied between approximately 2 and 7 ppm while capacitance signals and DRIFT spectra were recorded simultaneously.

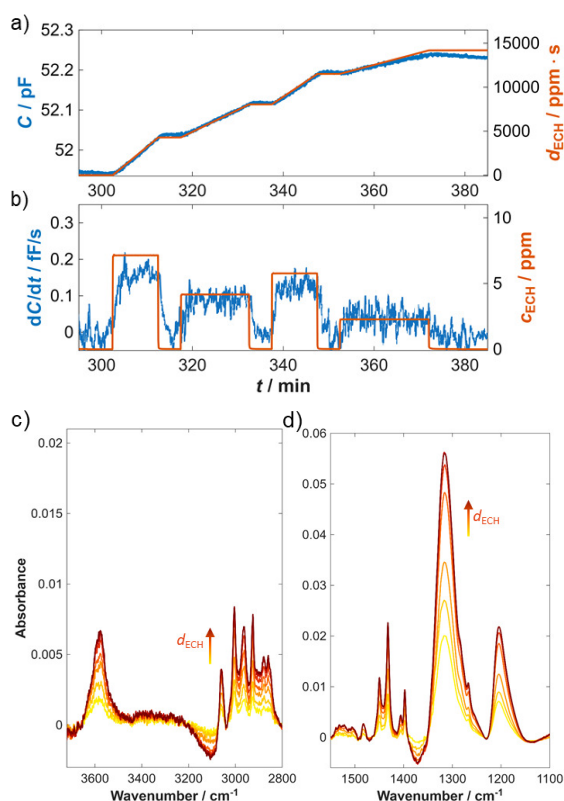


Fig. 2. Measurements during the dosing of different ECH concentrations. a) Time course of the capacitance signal versus the ECH dose (calculated from the concentration values of the dosed gases). b) Time derivative of the sensor capacitance versus the ECH concentration. c) and d) DRIFT spectra with increasing ECH loading.

The capacitance signal exhibits a clear dependency on the ECH dose. Following a dosimeter-type behavior, the signal increases linearly during gas exposure and remains at an elevated, nearly constant level once the ECH supply is stopped (see Fig. 1a)). The slope of the signal shows a linear dependency on the applied ECH concentration (see Fig. 1b)). This behavior supports the assumption of an adsorption process of ECH on the Cu-exchanged MFI zeolite.

The DRIFTS results confirm this interpretation. During ECH exposure, distinct absorption bands emerge mainly in the spectral regions between 1150 and 1500 cm^{-1} and between 2800 and 3600 cm^{-1} (see Figs. 1c) and d)). The intensity of these bands increased with higher ECH dose.

According to literature on the adsorption properties of zeolites towards hydrocarbons, these bands can be assigned to CH_2 deformation modes, C–O–C stretching vibrations of the epoxide ring, and C–H stretching vibrations of adsorbed organic species [4]. The persistence of these spectral features after ending of the ECH dosing further indicates stable adsorption onto the zeolite. The combined electrical and spectroscopic results therefore demonstrate that the observed capacitance response directly reflects the accumulation of carcinogenic ECH molecules in the zeolite. This confirms the suitability of this sensor setup for dosimeter-based trace gas sensing at room temperature.

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