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The Development of Laboratory-made NTD Sensor Systems for the Analysis of Volatile Organic Compounds (VOCs) in Synthetic Breath Samples

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INTRODUCTION: In recent years, the analysis of volatile organic compounds (VOCs) has emerged as a highly attractive sample preparation method due to its ease of application to a wide range of samples. These techniques, which significantly increase the sensitivity of the analysis by reducing the interference effect, especially in biological samples containing many components, have the possibility of being applied in many ways.[1] These systems, which are widely used for the analysis of a wide range of compounds from pesticides to biomarkers, have many advantages over systems obtained by preparing the adsorbent polymer in fiber form. NTD sensor systems, which are prepared to be more protected by the presence of the polymer on the inner surface of the needle, have a longer service life.[2] For this purpose, NTD systems can be prepared using many different types of polymers.In addition, the surface area is significantly increased by coating it onto a cylindrical outer surface. This significantly increases the sensitivity of the method. Breath analysis has become quite popular over the years.[3] These analyses offer advantages such as ease of use and rapid diagnosis, particularly in crucial areas such as early-stage disease diagnosis, and can be performed without causing any pain to the patient due to their non-invasive nature.[4] Additionally, breath samples, which contain less interfering matrix than other biological samples, allow for highly sensitive analyses. Furthermore, due to the very low concentrations of VOCs in breath, more sensitive analytical techniques are needed for these analyses. In this context, the study investigated the applicability of laboratory-developed NTD sensor systems to synthetic breath samples. For this purpose, lab-made NTD sensors prepared with polyaniline polymer in laboratory environment were used for the determination of benzene, toluene, ethylbenzene and and o-, p-xylene (BTEX) compounds, which are important for environmental exposure, in synthetic breath medium.

MATERIAL AND METHODS: The NTD systems used in the study were made using four different conductive polymers. Surface and physical characterization of these polymers prepared by electropolymerization were investigated using scanning electron microscopy (SEM), fourier transform infrared spectroscopy (FTIR) and thermogravimetric analysis. Following surface characterization, the responses of NTDs prepared with each polymer to BTEX solutions were compared and the polymer with the best response was selected. Subsequently, the sampling technique (active and passive), adsorption temperature, adsorption time and desorption time were optimised. Finally, the analytical properties of the method developed for BTEX analysis in synthetic breath samples were determined using the optimum parameters obtained from the optimisation studies.

RESULTS: SEM images of the prepared NTDs showed that the polymers obtained were on porous surfaces, indicating

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that the lab-made NTDs had suitable properties for adsorption. FTIR spectra obtained with the attenuated total reflectance (ATR) probe confirmed the functional groups in the existing structures and supported the chemical properties of the structures by confirming that the targeted polymers were obtained. FTIR spectra obtained with the ATR probe confirmed the functional groups in the existing structures and supported the chemical properties of the structures by confirming that the targeted polymers were obtained. When the effects of four different polymers (polyaniline, polypyrrole, polythiophene and polyfuran) prepared in the study were examined on the responses of BTEX, it was seen that polyaniline gave the most effective response. When it comes to optimizing the experimental operating conditions, firstly the effects of active and passive sampling on BTEX responses were investigated (Figure 1). The results showed that passive sampling had better effect on analyte signals than active sampling. Optimizing the adsorption temperature revealed that an adsorption temperature of 45 °C provided the optimal response. Lower adsorption temperatures reduce analyte interaction with the polymer, while higher temperatures facilitate desorption from the surface, reducing adsorption efficiency. Therefore, 45 °C was determined to be the ideal value for adsorption. On the other hand, adsorption time is also a crucial parameter for establishing the necessary analyte-polymer balance. A 20-minute adsorption process is sufficient for this purpose. Longer adsorption processes do not result in a positive response due to surface saturation, but they do experience a loss of efficiency due to the loss of analytes from the surface due to environmental conditions. The final parameter optimized was the desorption time, which corresponds to the time spent by NTDs in the gas chromatography injection block. The optimum condition for this value was determined to be 4 min.

CONCLUSION: BTEX compounds are important in both environmental and biological samples. The PANI-based NTDs prepared in this study were successfully applied in these analyses in synthetic breath media. Data obtained through optimization of experimental conditions demonstrated that these analyses can be successfully applied in synthetic breath media at concentrations ranging from 89 to 395 μg/m^3.

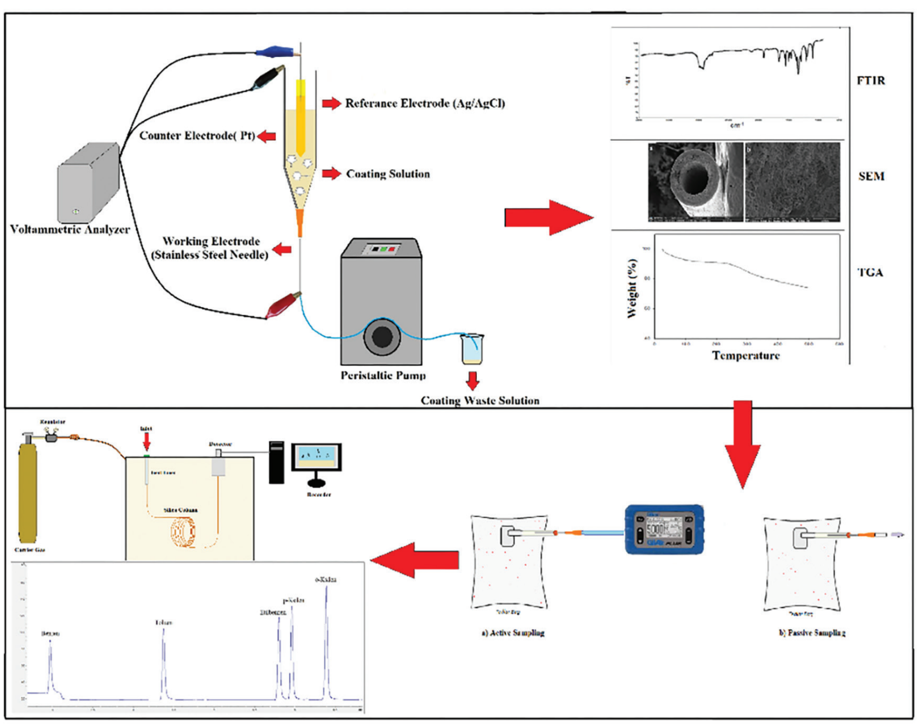

Figure 1. Experimental procedure of the study

KEYWORDS: Needle trap devices, breath analysis, gas chromatography, sample preparation

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