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Research Paper

# Development of Solid Phase Micro-Extraction Method Based on Nano Zno and Multi-Walled Carbon Nanotube Composite for Extraction and Measurement of Some Polycyclic Aromatic Hydrocarbons in Tobacco

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## Extended Abstract

**Introduction** Polycyclic aromatic hydrocarbons (PAHs) are a class of organic pollutants consisting of multiple benzene rings. They are generated both naturally and anthropogenically, particularly through the combustion of fossil fuels, industrial activities, and tobacco processing. PAHs pose serious health risks, including carcinogenic, mutagenic, and toxic effects on humans. Their lipophilic nature allows them to accumulate in biological tissues, leading to long-term exposure risks. Due to their toxicity, many regulatory agencies have established permissible limits for PAHs in food and environmental samples. Sensitive and accurate analytical methods are required to detect trace levels of PAHs in various matrices. Conventional extraction methods, such as liquid-liquid extraction (LLE) and solid-phase extraction (SPE), have limitations, including high solvent consumption, lengthy preparation times, and low recovery rates. Solid-phase micro-extraction (SPME) is an innovative, solvent-free sample preparation technique that enhances analytical sensitivity and efficiency. In this study, a novel ZnO/multi-walled carbon nanotube (MWCNT) nanocomposite was developed as an SPME fiber coating for the extraction and quantification of PAHs in tobacco samples.

**Methods** A ZnO/MWCNT nanocomposite was synthesized and applied as a fiber coating for SPME to enhance the extraction efficiency of PAHs. The nanocomposite was characterized using Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) to determine its structural and morphological properties. Optimization of SPME conditions was performed by evaluating factors such as extraction temperature, extraction time, desorption temperature, desorption time, and salt concentration using the one-variable-at-a-time (OVAT) approach. The method's performance was assessed in terms of linearity, sensitivity, precision, and limits of quantification (LOQ). The developed method was applied to the extraction and quantification of four PAHs naphthalene, fluorene, anthracene, and phenanthrene in tobacco-derived hookah water samples. Gas chromatography with flame ionization detection (GC-FID) was used for the final quantification of PAHs.

**Results and Discussion** The SEM analysis revealed that the ZnO/MWCNT nanocomposite coating exhibited a porous structure with a high surface area, facilitating efficient PAH adsorption. The FTIR spectra confirmed the successful incorporation of ZnO and MWCNT components in the nanocomposite. Optimization studies indicated that the best extraction efficiency was achieved under extraction temperature: 25°C, extraction time: 60 minutes and salt concentration: 3 g NaCl per 10 mL sample. The method exhibited excellent linearity over a concentration range of 1–20 µg/L, with correlation coefficients exceeding 0.99 for all four PAHs. The relative standard deviation (RSD) was below 9%, indicating high precision. The LOQ was determined to be approximately 0.3 µg/L. Application of the developed method to real tobacco samples demonstrated high recovery rates, confirming its suitability for trace-level PAH analysis. The method effectively detected PAHs in hookah water samples from various tobacco sources, providing valuable data on contamination levels. The results highlight the effectiveness of the ZnO/MWCNT nanocomposite coating as an adsorbent material for sensitive and precise PAH determination. The high surface area and porous structure, as evidenced by SEM analysis, significantly enhance adsorption capacity, while the stability of the composite, confirmed by FTIR, ensures consistent analytical performance. The low LOQ and strong linearity across a relevant concentration range indicate the method's potential for routine monitoring of PAHs in complex matrices. Notably, the high recovery rates from real tobacco and hookah water samples suggest that the method is both reliable and adaptable to real-world applications, where matrix interferences often pose analytical challenges. These findings not only provide a valuable tool for environmental and public health monitoring but also underscore the importance of developing advanced nanocomposite-based extraction techniques for trace contaminant detection. Future studies could focus on expanding the range of target analytes, evaluating long-term stability of the coating, and applying the method to broader environmental and food safety contexts.

**Conclusion** The study successfully developed and validated a novel ZnO/MWCNT-coated SPME fiber for the extraction and quantification of PAHs in tobacco samples. The nanocomposite exhibited superior extraction efficiency due to its high surface area and strong adsorption capacity. The optimized method provided excellent sensitivity, reproducibility, and robustness, making it a promising tool for monitoring PAH contamination in tobacco products and other environmental samples. Future research should focus on expanding the application of this nanocomposite for detecting other environmental contaminants, improving its long-term stability, and exploring potential modifications to enhance its selectivity for different classes of pollutants.

**Keywords:** Zinc oxide nanoparticles, Multi-walled carbon nanotube, Nanocomposite, Head space solid phase microextraction, Gas chromatography.

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