

MXene-Functionalised U-Shaped SMF-NC-SMF Optical Fibre Sensor for Label-Free Detection of PFOA and PFOS in Water

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Summary: A U-shaped single-mode–no-core–single-mode (SMF-NC-SMF) optical fibre sensor functionalised with a sequential PEI/PDADMAC/MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) multilayer interface is presented for the label-free, real-time detection of perfluorooctanoic acid (PFOA) and perfluoro octane sulfonate (PFOS) in aqueous media. Exploiting evanescent wave interactions at the fibre–solution interface, the sensor transduces analyte binding events as measurable wavelength shifts. PFAS contaminants produced clear, concentration-dependent responses spanning 0.1–100 pg/mL, establishing the platform's suitability for sensitive water quality monitoring applications.

Keywords: PFAS, PFOA, PFOS, optical fibre sensor, MXene, evanescent wave, water quality monitoring

Background, Motivation and Objective

Per- and polyfluoroalkyl substances (PFAS) comprise a family of over 4,700 synthetic chemicals characterised by exceptionally strong carbon–fluorine bonds that confer extreme environmental persistence and biological accumulation [1]. Among these, PFOA and PFOS are the most globally regulated, having been detected in surface water, groundwater, and treated drinking water at concentrations associated with hepatotoxicity, immunosuppression, endocrine disruption, and elevated cancer risk [2]. Under the European Union's revised Drinking Water Directive (EU 2020/2184), a combined permissible limit of 0.1 µg/L has been set for 20 listed PFAS compounds [3], placing stringent requirements on detection sensitivity. While liquid chromatography–tandem mass spectrometry (LC-MS/MS) remains the benchmark analytical technique, its dependence on centralised laboratory facilities, labour-intensive sample preparation, and specialist expertise significantly constrains its utility for real-time or on-site monitoring. Label-free optical fibre sensors based on evanescent wave interactions offer a compelling alternative, providing real-time, in-situ measurements in a miniaturised, low-cost format without the need for fluorescent labels [4]. This work presents a U-shaped SMF-NC-SMF fibre sensor with a layered PEI/PDADMAC/MXene functionalisation for sensitive detection of PFOA and PFOS.

Description of the New Method or System

The sensing element was fabricated by fusion splicing a segment of no-core fibre (NC, ~125 µm outer diameter, no guiding core) between two sections of standard single-mode fibre

(SMF-28), forming an SMF-NC-SMF multimode interference structure. This structure was tapered to ($\sim 9\text{ }\mu\text{m}$) then assembly was bent into a U-shape ($\sim 4\text{ mm}$ bend radius), significantly enhancing evanescent field penetration depth at the fibre–solution interface and increasing sensitivity to local refractive index changes induced by surface-bound analytes.

Surface functionalisation employed a sequential layer-by-layer (LbL) electrostatic deposition strategy. First, polyethylenimine (PEI), a branched polycation, was adsorbed onto the cleaned fibre surface to act as a primer layer and introduce abundant cationic binding sites. Second, poly(diallyldimethylammonium chloride) (PDADMAC), a strongly cationic polyelectrolyte, was deposited to build up the positive surface charge and improve adhesion of the subsequent layer. Finally, $\text{Ti}_3\text{C}_2\text{T}_x$ MXene nanosheets—two-dimensional transition metal carbide materials bearing abundant terminal functional groups ($-\text{OH}$, $-\text{F}$, $=\text{O}$) were electrostatically adsorbed as the terminal sensing layer [5]. The inherent fluorophilicity of MXene surfaces, combined with hydrophobic interactions, facilitates preferential capture of perfluorinated analytes. Sensing measurements were performed using a broadband light source and optical spectrum analyser; PFAS binding events were transduced as red shifts ($\Delta\lambda$) in the fibre transmission spectrum.

Results

Figure 1 presents the real-time $\Delta\lambda$ response of the PFOA sensor following introduction of analyte solutions at 0.1, 1, 10, and 100 pg/mL, alongside a deionised water (DI) blank. After a stable baseline period, PFOA introduction produced an immediate and sustained red shift,

with response magnitude increasing monotonically with concentration. The highest tested concentration (100 pg/mL) produced a maximum $\Delta\lambda$ of ~ 0.23 nm, while even at 0.1 pg/mL a distinguishable shift was recorded, indicating that sub-pg/mL detection limits are achievable with further optimisation. The DI blank yielded no measurable shift, confirming negligible non-specific response from the sensing interface.

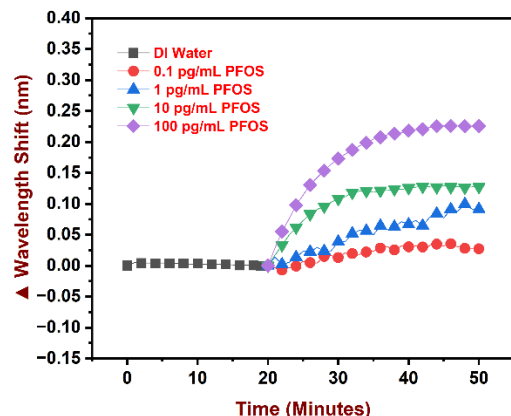


Fig. 1. Real-time wavelength shift ($\Delta\lambda$) of the MXene-functionalised SMF-NC-SMF sensor upon exposure to PFOA at concentrations of 1, 10, 100, and 1000 pg/mL, and DI blank.

PFOS produced analogous concentration-dependent wavelength shifts (Fig. 2), with $\Delta\lambda$ magnitudes broadly comparable to those recorded for PFOA across the same concentration range. This correspondence is consistent with the structural similarity of the two compounds and their equivalent fluorophilic affinity towards the MXene sensing layer. For both analytes, responses reached steady-state within approximately 15–20 minutes of introduction, a behaviour characteristic of diffusion-limited binding kinetics at the MXene–solution interface.

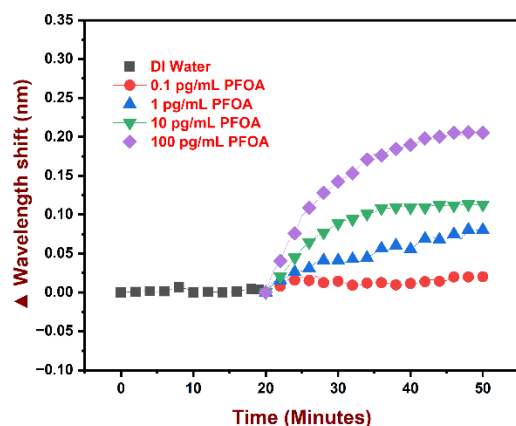


Fig. 2. Real-time wavelength shift ($\Delta\lambda$) of the MXene-functionalised SMF-NC-SMF sensor upon exposure to PFOS at concentrations of 1, 10, 100, and 1000 ng/mL, and PBS blank (0 ng/mL).

SEM analysis confirmed successful MXene deposition on the functionalised fibre surface. At magnifications discrete near-spherical MXene nanostructures are visible across the silica surface, a morphology consistent with $\text{Ti}_3\text{C}_2\text{T}_x$ self-assembly under LbL electrostatic deposition conditions. Nanostructure clustering along the fibre surface suggests effective immobilisation via the PEI/PDADMAC scaffold, and the homogeneous surface coverage observed across both fields of view reflects a reproducible sensing interface capable of sustaining consistent evanescent wave interaction with PFAS analytes.

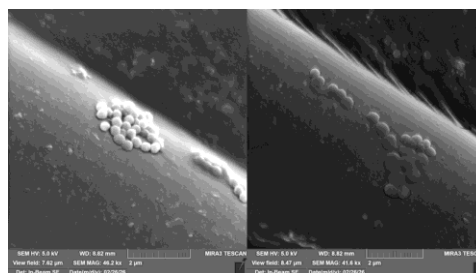


Fig. 3. SEM micrograph confirming MXene nanostructure deposition on the fibre surface.

The reproducible, dose-dependent shifts observed for PFOA and PFOS collectively confirm the effectiveness of the PEI/PDADMAC/MXene architecture for evanescent wave-based PFAS sensing, supporting the platform's applicability to water quality monitoring.

References

- [1] Zexi Zhang, Kaixuan Chen, Bruno Ameduri, and Mao Chen, *Chemical Reviews* **2023** 123 (22), 12431–12470; DOI: 10.1021/acs.chemrev.3c00350
- [2] Grandjean P, Clapp R. Perfluorinated Alkyl Substances: Emerging Insights Into Health Risks. *New Solut.* 2015 Aug;25(2):147-63. doi: 10.1177/1048291115590506.
- [3] Marco Dettori, Antonella Arghittu, Giovanna Deiana, Paolo Castiglia, Antonio Azara, the revised European Directive 2020/2184 on the quality of water intended for human consumption. *Environmental Research*, Volume 209, 2022, 112773, ISSN 0013-9351; doi.org/10.1016/j.envres.2022.112773.
- [4] Fan X, White IM. Optofluidic Microsystems for Chemical and Biological Analysis. *Nat Photonics*. 2011 Oct 1;5(10):591-597; doi:0.1038/nphoton.2011.206.
- [5] B. Anasori, M.R. Lukatskaya, Y. Gogotsi, *Nat. Rev. Mater.* 2, 16098 (2017); doi: 10.1038/natrevmats.2016.98