

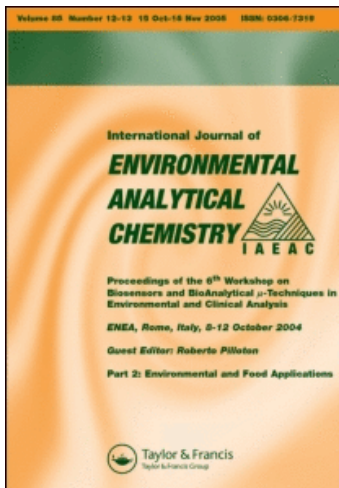
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DETERMINATION OF CHLORINATED HYDROCARBON SPECIES IN AQUEOUS SOLUTION USING TEFLON COATED ATR WAVEGUIDE/FTIR SPECTROSCOPY

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The potential of an optical sensor based on mid-infrared spectroscopy, utilising a zinc selenide (ZnSe) attenuated total reflectance (ATR) element coated with an amorphous Teflon polymer, to determine chlorinated hydrocarbon species (CHC) in an aqueous environment is examined. The polymer coating concentrates the analytes within the penetration depth of the Fourier transform infrared (FTIR) evanescent wave and excludes water from the region. Teflon AF (Amorphous Fluoropolymer) is a family of amorphous copolymers based on polytetrafluoroethylene (PTFE), and is commercially available in two polymeric grades. Teflon AF is highly amorphous in nature with a large 'void volume', exhibits excellent chemical resistance and low water absorption. Such properties identify it as an excellent candidate for enrichment coating on an ATR/FTIR sensor. The potential of both polymeric grades of Teflon AF as enrichment membranes for ATR/FTIR analysis of CHC species was examined and contrasted. A rapid, repeatable, reversible response was observed with both grades to a range of CHC species. Linear responses in the mg/L region, with detection limits in the low mg/L region were achieved with the system used.

Keywords: Chlorinated hydrocarbons; Mid-infrared spectroscopy; Attenuated total reflectance; Amorphous Teflon; Diffusion

INTRODUCTION

Chlorinated hydrocarbon species (CHCs), arising from both industrial and domestic waste, pose a serious threat to the environment. For example, trichloroethylene (TCE, C_2HCl_3) is a non-flammable, volatile liquid that has been used as an industrial solvent and degreaser, a dry-cleaning solvent, and a refrigerant. It is used in products such as typewriter correction fluids, paint removers/strippers, adhesives, and stain removers, and in the past TCE was used as a general anaesthetic and in the decaffeination of coffee. TCE is not thought to occur naturally in the environment, however, it has been commonly identified in ground water and many surface waters as a result of the manufacture, use, and disposal of the chemical. TCE is irritating to body tissues and, if inhaled, depresses the central nervous system and may even, in extreme

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cases, cause death. Effects on the gastrointestinal system, liver, and kidneys have also been reported [1]. The maximum allowable contaminant level for TCE, specified by the United States Environmental Protection Agency (U.S. EPA) in their National Primary Drinking Water Regulations is 0.005 mg/L [chlorobenzene = 0.1 mg/L, tetrachloroethylene = 0.005 mg/L, total trihalomethanes (including chloroform) = 0.08 mg/L] [1]. TCE is a suspected human carcinogen, and the U.S. EPA considers trichloroethylene as an intermediate between a probable and possible human carcinogen (Group B/C) [1]. Chlorinated hydrocarbons can be absorbed into the human body through the lungs, gastrointestinal tract, and skin, and most are stored in fatty tissue [2], with the potential for strong bioconcentration.

Standard procedures for the trace determination of CHCs in water include gas chromatography (GC) and GC-mass spectrometry (MS). Despite the fact that these methods are highly selective and sensitive, sampling steps by skilled operators are necessary. Coupled with the high consumables cost and long timeframe of such analyses, their potential as real-time, on-line techniques is limited.

The potential of an optical sensor based on mid-infrared spectroscopy ($4000\text{--}600\text{ cm}^{-1}$), utilising attenuated total reflectance (ATR) waveguides coated with various polymers, for the analysis of volatile organic compounds in solution has been reported [3–12]. The use of ATR/FTIR for analysing aqueous samples is limited due to the interference of water absorbance bands and the high concentration of analyte required for detection. However, the incorporation of a thin hydrophobic polymer film, which both excludes the interfering water bands and reversibly enriches the analyte within the penetration depth of the FTIR evanescent wave, on the surface of the waveguide greatly increases sensitivity.

The general requirements for an analytical measurement technique include sensitivity, short response time, and reversibility. These requirements are met by choosing an appropriate polymer film for the ATR element. Various polymers, including polyisobutylene (PIB) [4–6,9], low density polyethylene (LDPE) [4–6], ethylene/propylene copolymer (E/Pco) [4–7], 1,2-polybutadiene (PBD) [4], plasticised poly(vinyl chloride) (PVC) [11], polypropylene (PP) [12], polystyrene (PS) [12], and poly(methyl methacrylate) (PMMA) [12] have been studied. However, in general, those polymers exhibiting the best enrichment characteristics for CHCs, e.g. PIB and E/Pco, tend to be rather soft due to their wholly amorphous nature. The use of amorphous *Teflon*[®] as an enrichment coating has been examined in a limited manner [3,10], with no previous comparison of the response characteristics of the two commercially available grades performed. Amorphous *Teflon*[®] possesses a clear spectral window within the mid-infrared region utilised for analysis of chlorinated hydrocarbons (approximately $1000\text{--}700\text{ cm}^{-1}$). It exhibits rapid analyte enrichment and reversibility, excellent robustness, chemical resistance and longevity characteristics, and is relatively easy to coat, and adheres well, onto a ZnSe ATR crystal waveguide.

***Teflon*[®] AF (Amorphous Fluoropolymer) [13]**

Teflon[®] AF is the DuPont registered trademark for its family of amorphous copolymers based on 2,2-bis(trifluoromethyl)-4,5-difluoro-1,3-dioxole (PDD) with other fluorine containing monomers, e.g. tetrafluoroethylene (TFE). At present, two general-purpose grades of *Teflon*[®] AF are available, both copolymers of PDD and TFE. The structure

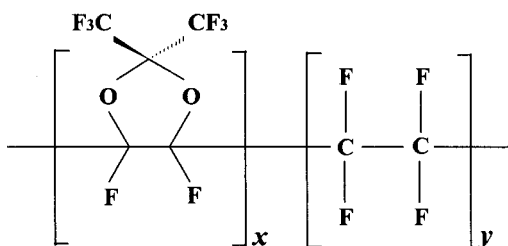


FIGURE 1 Proposed repeat unit present in *Teflon*[®] AF.

of the copolymer is shown in Fig. 1.

1. AF 1600, glass transition temperature (T_g) = 160°C, 65 mol% PDD.
2. AF 2400, glass transition temperature (T_g) = 240°C, 87 mol% PDD.

Copolymers of PDD with TFE having less than 20 mol% dioxole are no longer amorphous, due to the presence of TFE runs of sufficient length to crystallise. Crystalline polymers are marked by high compactness and small distances between chains, making it difficult for substances to diffuse through this phase. AF 2400, with its high proportion of bulky PDD groups, is more amorphous in nature, with a larger 'void/free volume', than AF 1600 and, it is therefore projected will facilitate more effective diffusion of CHCs. The specific free volume (SFV, free volume/g of polymer) and the fractional free volume (FFV, free volume/cm³ of polymer) are frequently employed as measures of the free volume potentially available for penetrant diffusion within polymers. Calculated values of 0.192 and 0.327 have been reported for the SFV and FFV respectively of AF 2400 [14]. These values are higher than those of other high free volume polymers, which typically possess FFVs within the range 0.18–0.22 [14].

Properties of Teflon[®] AF which Identify it as a Candidate for Enrichment Coating

1. Amorphous in nature, with very limited crystallinity.
2. Low water absorption.
3. Essentially non-polar, possesses no reactive chemical functional groups and exhibits excellent chemical resistance.
4. High temperature stability.
5. Soluble at room temperature in several perfluorinated solvents. Both grades are supplied in solution, making coating onto an internal reflection waveguide relatively easy, i.e., spin coating or dip coating technique.
6. Very low Refractive Index (AF 1600 = 1.31 and AF 2400 = 1.29, sodium D line (589 nm) at 20°C), promoting use as an ATR cladding material.
7. Possess high optical transmission across a broad wavelength region from the near infrared to the ultraviolet.
8. *Teflon*[®] AF polymers have extraordinarily high gas permeability compared with other polymers.

Table I clearly identifies a link between gas permeability and the amorphous nature of the polymer. PDD homopolymer, which is wholly amorphous in nature, exhibits

TABLE I Gas permeability data through thin films (centiBarrer) [13]

Gas	AF 1600	AF 2400	TFE homopolymer	PDD homopolymer
O ₂	34,000	99,000	420	154,000
N ₂	–	49,000	140	81,000
CO ₂	–	280,000	1,200	–

centiBarrer = $10^{-8} \times \text{cm}^3 \text{ cm/cmHg s cm}^2$.

high permeability, whereas, crystalline TFE homopolymer demonstrates very low permeability. Also, the more amorphous AF 2400 exhibits higher permeability than AF 1600.

The objectives of the study were to (a) examine and contrast the potential of the two commercially available amorphous *Teflon*[®] polymer materials as enrichment membranes for ATR/FTIR analysis of chlorinated hydrocarbon (CHC) species and (b) quantify the response characteristics of both grades to CHCs using validation parameters such as versatility of response, dynamic response, repeatability of response, detection limits, linearity/reversibility of response, and long term stability/robustness.

EXPERIMENTAL

A BIO-RAD Excalibur Series FTS 3000 FTIR spectrometer, equipped with a cryogenic mercury cadmium telluride (MCT) detector was used for measurements. The horizontal ATR element was a standard trapezoidal ZnSe crystal (Dimensions: 72 mm × 10 mm × 6 mm) with an angle of incidence of 45°, giving six reflections in contact with the sample. *Teflon*[®] AF was obtained in solution from DuPont (AF 1600 = 6% solids, AF 2400 = 1% solids in Fluorinert FC-75 perfluorinated solvent) and coated onto the crystals using a Chemat Technology KW-4A spin coater, followed by manufacturer recommended temperature curing [AF 1600: 25°C for 10 min, 112°C for 10 min, 165°C for 5 min, 330°C for 10 min; AF 2400: 25°C for 10 min, 112°C for 10 min, 245°C for 5 min, 330°C for 10 min]. The polymer-coated crystal was incorporated into a Specac Thermostabilised Top Plate stainless steel flow cell (110 μL volume). A Shimadzu LC-5A pump was used throughout, with all experimental runs performed in continuous flow mode at a rate of 5 mL/min. All sample spectra were ratioed against the polymer-coated crystal in equilibrium with DI water. During each kinetic run performed, spectra were recorded every 10 s, at a resolution of 4 cm⁻¹, for a 30 min timeperiod at room temperature. In order to limit losses due to diffusion processes, stainless steel tubing was used for transport of analyte solutions. When preparing solutions, all analytes were weighed directly into low headspace volumetric glassware, and dissolved with sonication, immediately prior to use. Fresh solutions were prepared for each run, and all solutions sealed during analyses.

RESULTS AND DISCUSSION

Polymer Coating

Amorphous *Teflon*[®] was chosen as a coating in order to (a) reduce water ingress, (b) achieve a rapid, significant response, and (c) prepare a cladding which is easy to handle, extremely physically robust, with a long operational lifespan.

The penetration depth of the evanescent wave in the mid-infrared region (4000–600 cm^{-1}) for the system used (i.e., ZnSe ATR crystal with the IR beam incident at an angle of 45°) was calculated to range from 0.7 to 2.5 μm for *Teflon*[®] AF 1600 and from 0.7 to 2.4 μm for AF 2400. Therefore, in order to reduce disruption from water absorptions in the analyte solution, a membrane thickness of two/three times the beam penetration depth was produced. Membrane thickness was measured using a simple gravimetric technique as highlighted in Eq. (1).

$$d = \frac{\text{Mass of Membrane}}{\text{Density of Membrane} \times \text{Surface Area coated}} \quad (1)$$

where d = Teflon layer thickness, and the specific gravity of AF 1600 is taken to be 1.78 and AF 2400 to be 1.67. Coating of the ZnSe waveguide with *Teflon*[®] AF was performed through spin coating of commercially available AF solutions, followed by manufacturer recommended temperature curing.

Versatility of Response

The response of the system, with the waveguide coated alternatively with a 6.8 μm AF 1600 membrane and a 5.3 μm AF 2400 membrane, to a range of CHCs was examined, and is shown in Table II. All spectra were recorded at a solution concentration of 100 mg/L. The time constant (T_{90}) value is defined as the time taken for the analyte absorbance value to reach 90% of its saturation value [4].

The results presented in Table II indicate the potential of the system for the analysis of a range of pollutant species, with rapid diffusion and low equilibration times observed for both *Teflon*[®] AF grades. The observed infrared absorption bands for the four analytes examined are spectrally well resolved (i.e. trichloroethylene (TCE) = 935 cm^{-1} /844 cm^{-1} /782 cm^{-1} , tetrachloroethylene (TeCE) = 913 cm^{-1} , chloroform (CF) = 767 cm^{-1} , chlorobenzene (MCB) = 741 cm^{-1} /1481 cm^{-1}), indicating the potential of the system for multicomponent analysis.

The molar volume for each of the four analytes examined was calculated from available data [15] (TCE = 90.07 cm^3 @ 20°C, TeCE = 103.64 cm^3 @ 20°C, CF = 80.12 cm^3 @ 20°C, MCB = 101.41 cm^3 @ 20°C). Therefore, an apparent link between molar volume and diffusion rate is noted, with the smallest analyte (CF) diffusing into both AF grades most rapidly and the largest analyte (TeCE) entering at

TABLE II Response of *Teflon*[®] AF membranes to various CHCs

Analyte	Concentration (mg/L)	Band Wavenumber (cm^{-1})	Band Intensity		T_{90} Value (min)	
			AF 1600	AF 2400	AF 1600	AF 2400
Trichloroethylene (TCE)	100	935	0.008	0.012	6.6	5.6
Tetrachloroethylene (TeCE)	100	914	0.118	0.171	16.6	13.6
Chloroform (CF)	100	767	0.013	0.021	6.1	3.8
Monochlorobenzene (MCB)	100	741	0.036	0.066	9.6	5.0
		1481	0.008	0.012	9.8	4.8

the slowest rate, suggesting that physical size exhibits a major impact on diffusion rates into the essentially non-polar, unreactive AF membranes.

Dynamic Response and Repeatability

The consistency of response of the measurement procedure to a series of 5 repeated measurements of 100 mg/L TCE standard solutions, with a fresh water reference between each run, was examined. Due to a higher extinction coefficient, the absorption band at 935 cm^{-1} was chosen for the analyses, rather than the bands at 844 cm^{-1} or 782 cm^{-1} .

A. *Teflon*[®] AF 1600 membrane – thickness = $6.8\text{ }\mu\text{m}$

Number of standards (100 mg/L TCE)	= 5
Band wavenumber	= 935 cm^{-1}
Mean band intensity	= 0.008
Relative standard deviation	= 1.4%
Mean T_{90} value	= 6.6 min

B. *Teflon*[®] AF 2400 membrane – thickness = $5.3\text{ }\mu\text{m}$

Number of standards (100 mg/L TCE)	= 5
Band wavenumber	= 935 cm^{-1}
Mean band intensity	= 0.012
Relative standard deviation	= 6.9%
Mean T_{90} value	= 5.6 min

A typical diffusion curve for 100 mg/L TCE into the AF 2400 membrane is shown in Figure 2.

When developing a sensor for *in situ* analysis, it is necessary that the analyte enriches rapidly, in a repeatable manner. The low T_{90} values for both AF 1600 and AF 2400 highlight efficient diffusion and a rapid response to TCE, with AF 2400 exhibiting a more timely response than AF 1600. The low %RSD values highlight repeatability

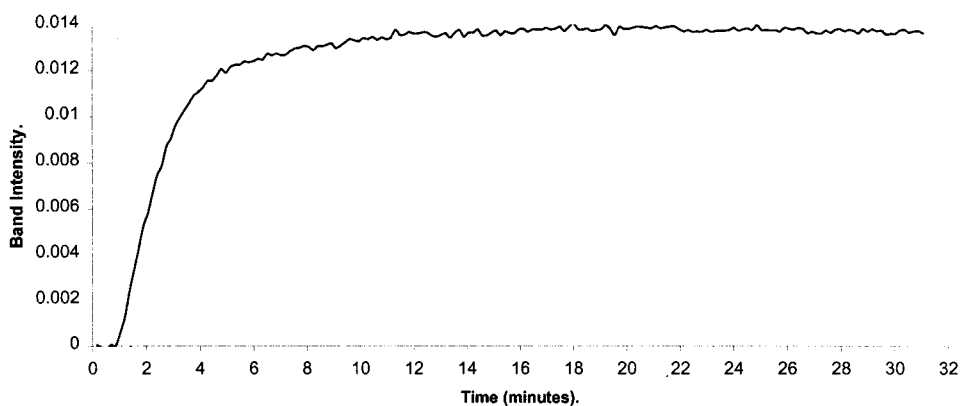


FIGURE 2 Diffusion curve of 100 mg/L TCE into AF 2400. Membrane thickness = $5.3\text{ }\mu\text{m}$. Infrared band at 935 cm^{-1} . Room temperature.

and the potential for both membranes. The level of response (i.e., absorbance value) of the AF 2400 coating is substantially larger than that of a similar thickness AF 1600 cladding to a 100 mg/L TCE solution. This is as a result of the higher amorphous nature of AF 2400 (i.e. larger void volume) and shows a similar trend to that observed for the gas permeability properties observed in Table I. This higher void volume available to analyte molecules accounts for the higher concentration observed in the evanescent wave region at equilibrium.

Detection Limits

The Limit of Detection (LOD=three times the baseline noise level) and Limit of Quantitation (LOQ=six times the baseline noise level) for TCE in both *Teflon*[®] AF grades was calculated. LOD and LOQ values of 9 and 18 mg/L respectively were observed for the 5.3 μm AF 2400 membrane, and values of 18 and 36 mg/L were measured for the 6.8 μm AF 1600 film.

Although the observed values are noted to be relatively high, it is noted that the ATR element used was a standard trapezoidal ZnSe crystal with only six reflections in contact with the sample. Therefore, the LOD/LOQ values obtained are promising, with much increased sensitivity to be achieved using Fibre Optic Evanescent Wave Sensor (FEWS) systems [7], laser sources [8], and/or evanescent wave enhancement techniques [11].

Linearity of Response

Standard calibration curves for TCE in both grades of *Teflon*[®] AF were performed. The plot obtained for AF 2400 is illustrated in Fig. 3. System equilibration was achieved in approximately 10 min for AF 1600 runs, and approximately 9 min for AF 2400 runs.

A regression coefficient of 0.992 was obtained for AF 1600 for standards in the range 25–100 mg/L, and a value of 0.994 was obtained for AF 2400 for standards in the range 10–100 mg/L. The linearity of response observed with both membranes illustrates the analytical potential of this optical sensor approach.

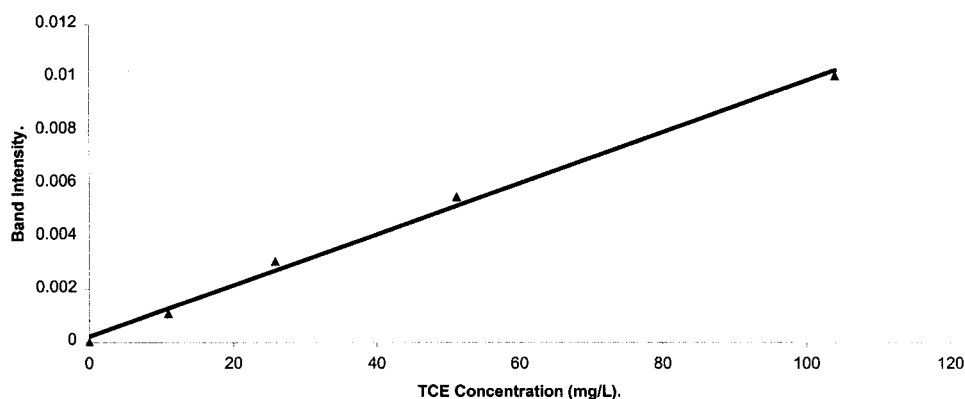


FIGURE 3 TCE standard calibration curve – *Teflon*[®] AF 2400 (5.3 μm) – C–Cl band @ 935 cm^{-1} .

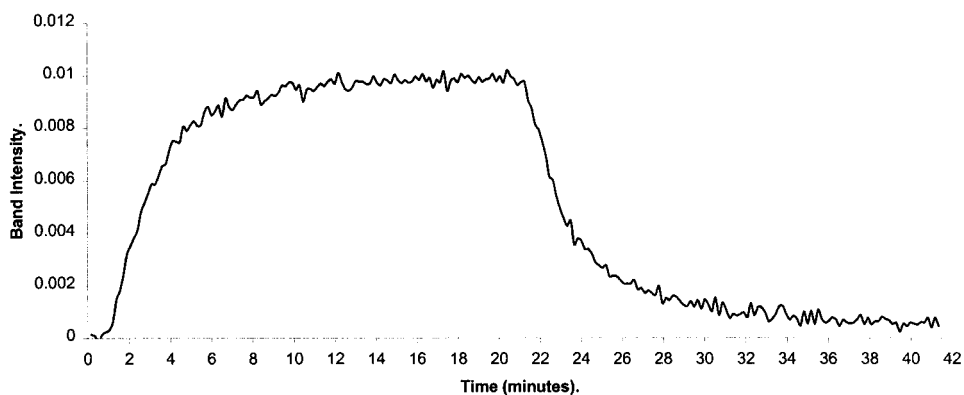


FIGURE 4 Reversibility of TCE enrichment into *Teflon*[®] AF 1600 (6.8 μm). 119 mg/L TCE in water. 20-min enrichment time followed by flushing with water (flow rate = 5 mL/min). C-Cl band at 935 cm^{-1} . Room temperature.

Reversibility of Response

A further requirement for in situ analysis is that analytes can be removed rapidly from the polymer for subsequent analyses. The removal of TCE (approximately 100 mg/L) from both membranes, through flushing with water at room temperature, was examined.

Timely removal of TCE, from both membranes, was observed using a water flush following enrichment. A regeneration time of approximately 10 min was observed for AF 1600, and a time of approximately 9 min was noted for AF 2400. Figure 4 illustrates the reversibility of enrichment into the AF 1600 film. The analyte may be removed more rapidly through the incorporation of an air flush step into the measurement sequence [3]. However, as a HPLC pumping system was utilised, flushing with air was not possible on the set-up used.

Long Term Stability

Teflon[®] AF exhibits excellent mechanical and physical properties at temperatures up to the glass transition temperature (AF 1600 = 160°C , AF 2400 = 240°C). It also exhibits good dimensional stability, a smooth surface, rigidity at high use temperatures, and, as a fluoropolymer, has extremely high resistance to chemical attack. Therefore, *Teflon*[®] AF can be fabricated into films that can function in severe exposure conditions of high temperatures, harsh chemicals, and destructive environmental agents [16].

Both grades have been used over many months in the laboratory without degradation of response. For example, when initially examined the AF 2400 film (5.3 μm) yielded band intensity (at 935 cm^{-1}) and T_{90} values of 0.012 and 5.6 min respectively for a 100 mg/L TCE solution. Following an eight-month period of intermittent usage the same film exhibited band intensity and T_{90} values of 0.013 and 6.2 min respectively for the same concentration of analyte. Within the intervening period, the pumping system was changed and the inlet line slightly increased in volume, contributing to the small increase observed in the T_{90} value.

Teflon[®] AF is robust, easy to handle and strongly adhered to the crystal surface. Removal of the membranes from the crystals was achieved through dissolution in the Dupont supplied solutions.

Previously Published Data

A paper evaluating *Teflon*[®] AF 1600 as an enrichment membrane for the fibre-optic determination of CHCs was identified [3]. No earlier work that examined *Teflon*[®] AF 2400 membranes was located. The sensitivity, selectivity, linearity, reversibility, rapidity, and stability of the AF 1600 coated ATR element based system described here (six reflections in contact with the sample) is qualitatively comparable with the results obtained for the multiple reflection fibre-optic coated system reported previously.

CONCLUSION

The potential and performance of a *Teflon*[®] AF coated ATR waveguide/FTIR spectroscopy system for the analysis of CHCs in aqueous solution has been assessed. The potential for multi-component applications has been highlighted, with rapid, reversible, sensitive, and selective responses to trichloroethylene (TCE), tetrachloroethylene (TeCE), chloroform (CF), and chlorobenzene (MCB) identified (multi-component affects are currently under investigation within the group). Convenient coating of the ZnSe waveguide with AF polymer was performed through spin coating of commercially available AF solutions followed by temperature curing. A rapid response to the analytes was observed for both commercially available grades of *Teflon*[®] AF. AF 2400, with its more amorphous nature and larger free volume, exhibited a larger and faster response to the CHCs examined and an enhanced limit of detection. Repeatability of response to 100 mg/L TCE aqueous solutions, and linearity in the mg/L region, was observed with both polymer grades. Removal of TCE, and hence rapid regeneration of the sensor, was achieved through flushing with water. *Teflon*[®] AF is extremely physically robust in nature and long-term stability of the membranes was observed.

The sensitivity levels achieved with the simple laboratory set-up used (LOD for AF 2400 = 9 mg/L, AF 1600 = 18 mg/L) are promising. Increased sensitivity is achievable through the use of multiple reflection fibre optic waveguides [7], laser sources [8] and/or evanescent wave enhancement techniques [11].

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