Art Photonics GmbH

Application Note

High-Sensitivity Transflection Fiber Probe

Submitted By :
Camillo Mizaikoff
Iskander Usenov

art photonics

Abstract

This article demonstrates art photonics' improved transflection fiber probe design and additionally uncovers the probe's increased functionality in both experimental and industrial applications. Possible applications of the probe range from biopharmaceutical analysis [1], real-time reaction monitoring [2], analytical characterization and the production and development of biofuel. These applications are possible thanks to the probe's specialization of transmission spectroscopy in liquids at long distance in the UV - VIS and VIS – NIR spectral ranges. Measurements were performed on Isopropanol and Ethanol water solutions of various concentrations in order to analyze the sensitivity of the fiber probe. These substances were selected thanks to a large amount of research done on their spectra as well as their universal, industry-wide application. A sensitivity to solute concentrations as low as 5 % could be reliably determined using the experimental data.

1 Description

"*Double-pass fiber probe*", is the common name given to transflection fiber probes, which transmit light through a sample, before reflecting it off an efficient optical mirror. The mirror transmits the light back through the sample, where it travels into the detection fiber. This repeated movement gives it the name "*double-pass probe*". The adaptable design of the probe permits it to measure both transmission and reflection, thus leading to the name *transflection*. The probe is immersion, i.e., it's directly submerged in the liquid sample to be studied, due to the source and return fibers being located on the same side of the sample [3]. This process is thus in contrast to methods which use flow cells or cuvettes. Available with a number of removable shaft heads, with slit width - 1, 2, or 5 mm (optical pass lengths 2, 4, 10 mm, respectively), these double-pass fiber probes have a bifurcated design, connecting the probe to both a light source and a spectrometer.



Figure 1: Transflection fiber probe (a) and shaft head (b).

2 Methods

An Ocean Optics *NIRQUEST* spectrometer and art photonics' improved transflection fiber probe were used to perform these measurements. The probe was first immersed in solutions of both Ethanol and Isopropanol of descending concentration (100%, 80%, 40%, 20%, 10%, 5%, 2.5% and 1%), at each stage of the process three spectra were noted and averaged for later evaluation. 6 ms was the integration time used for the measurements. The Ethanol solutions begin at an 80% concentration as an 80% Ethanol stock solution was used and further diluted in order to create the other Ethanol solutions. Three spectra of distilled water were also recorded for reference.

3 Measurements

Figures below show the averaged transmission spectra in the NIR spectral region for each measured concentration of Isopropanol (fig. 2) and Ethanol (fig. 4) respectively. Additionally, the absorbance spectra were calculated and the spectrum of distilled water (used to dilute the solutions) was deducted from each averaged measurement (fig. 3 & 5). Two exemplary peak decays based on absorbance are shown in figures 6 and 7.

3.1 Isopropanol

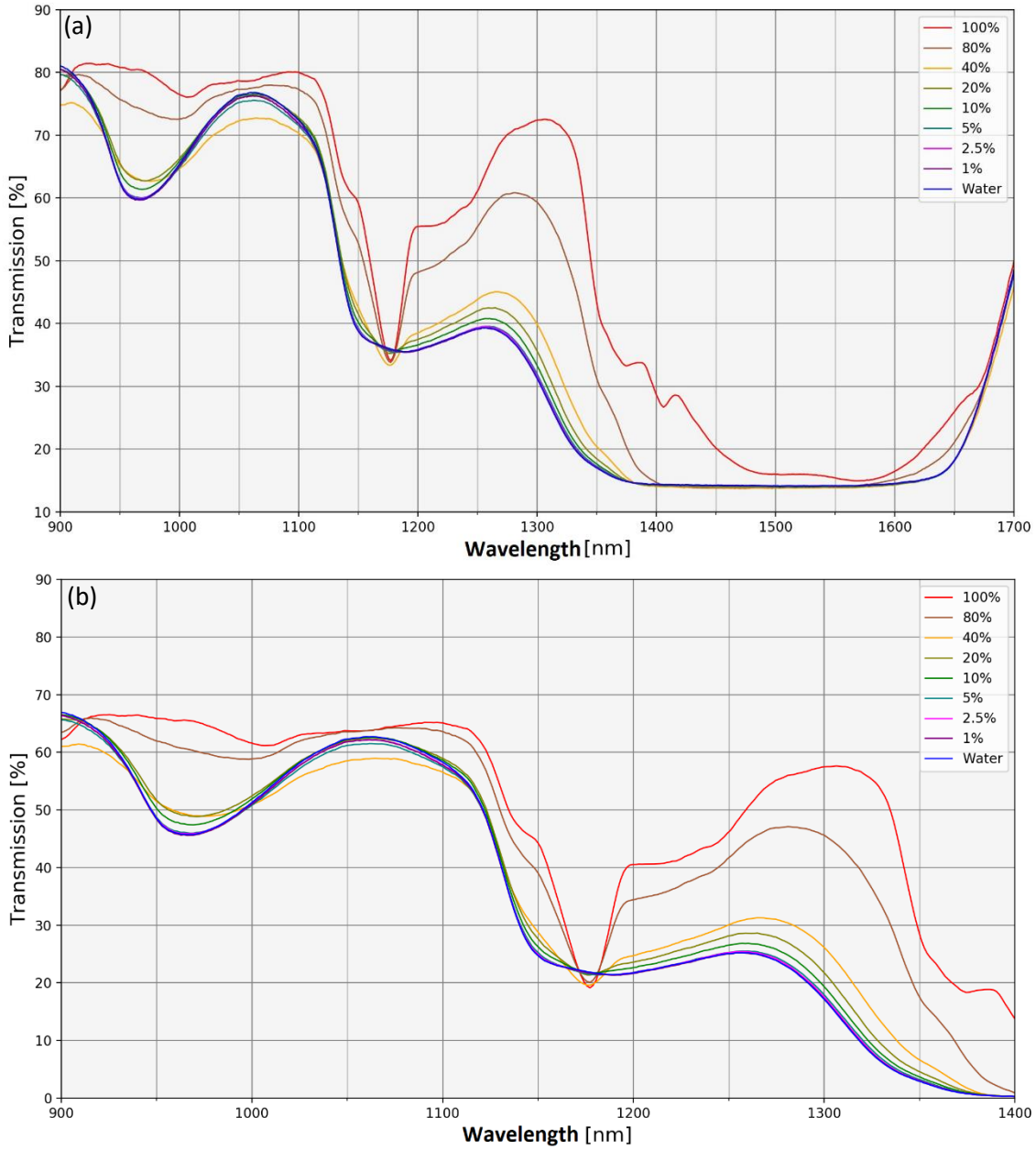


Figure 2: Raw (a) and preprocessed (b) averaged transmission spectra of 100% - 1% concentrated Isopropanol solutions.

Raw data were preprocessed before the following analysis: baseline subtraction, removing the range with 0% transmission (infinite absorption).

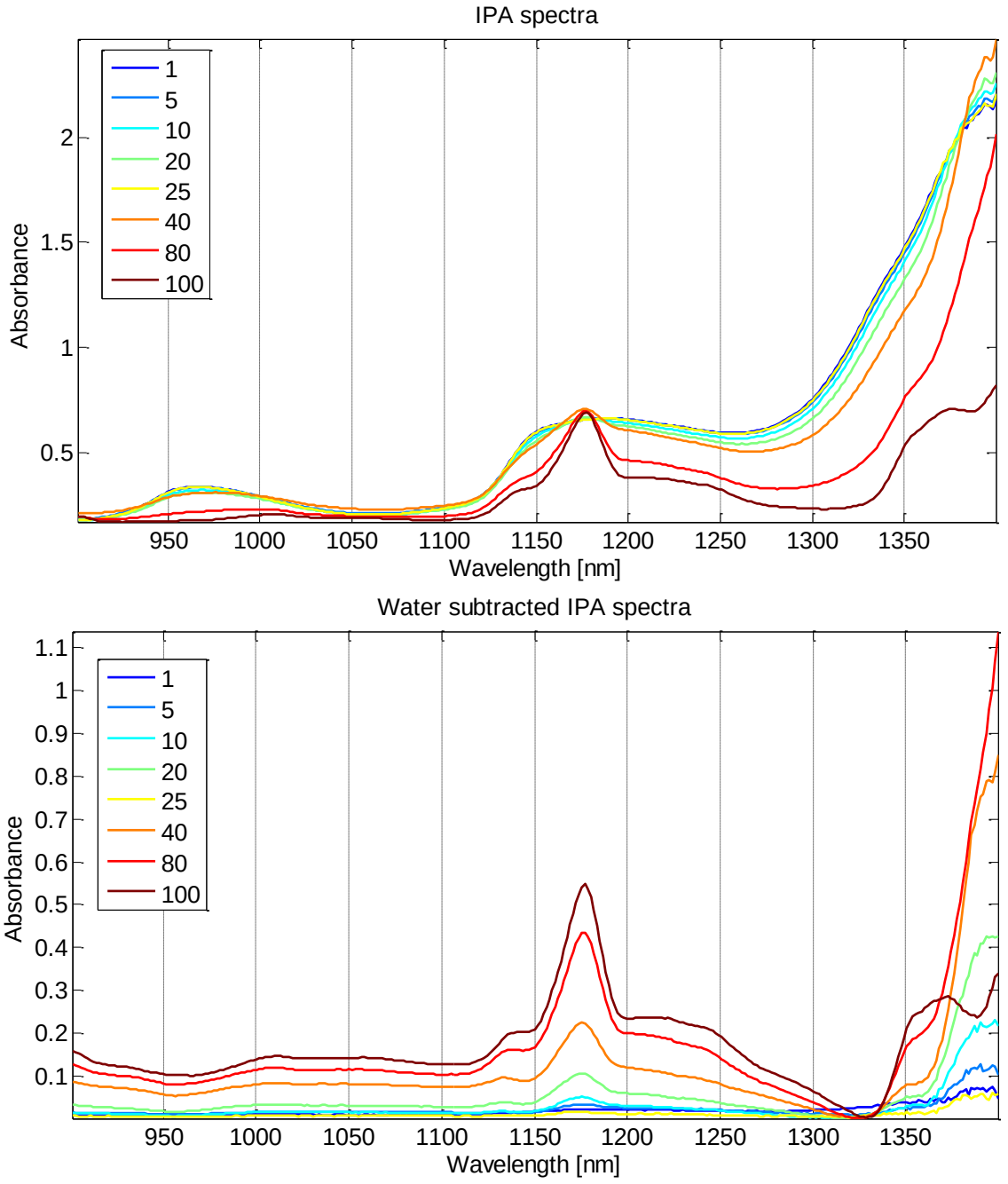


Figure 3: Water absorbance spectrum subtracted from averaged absorbance spectra of 100% - 1% concentrated Isopropanol water solutions.

3.2 Ethanol

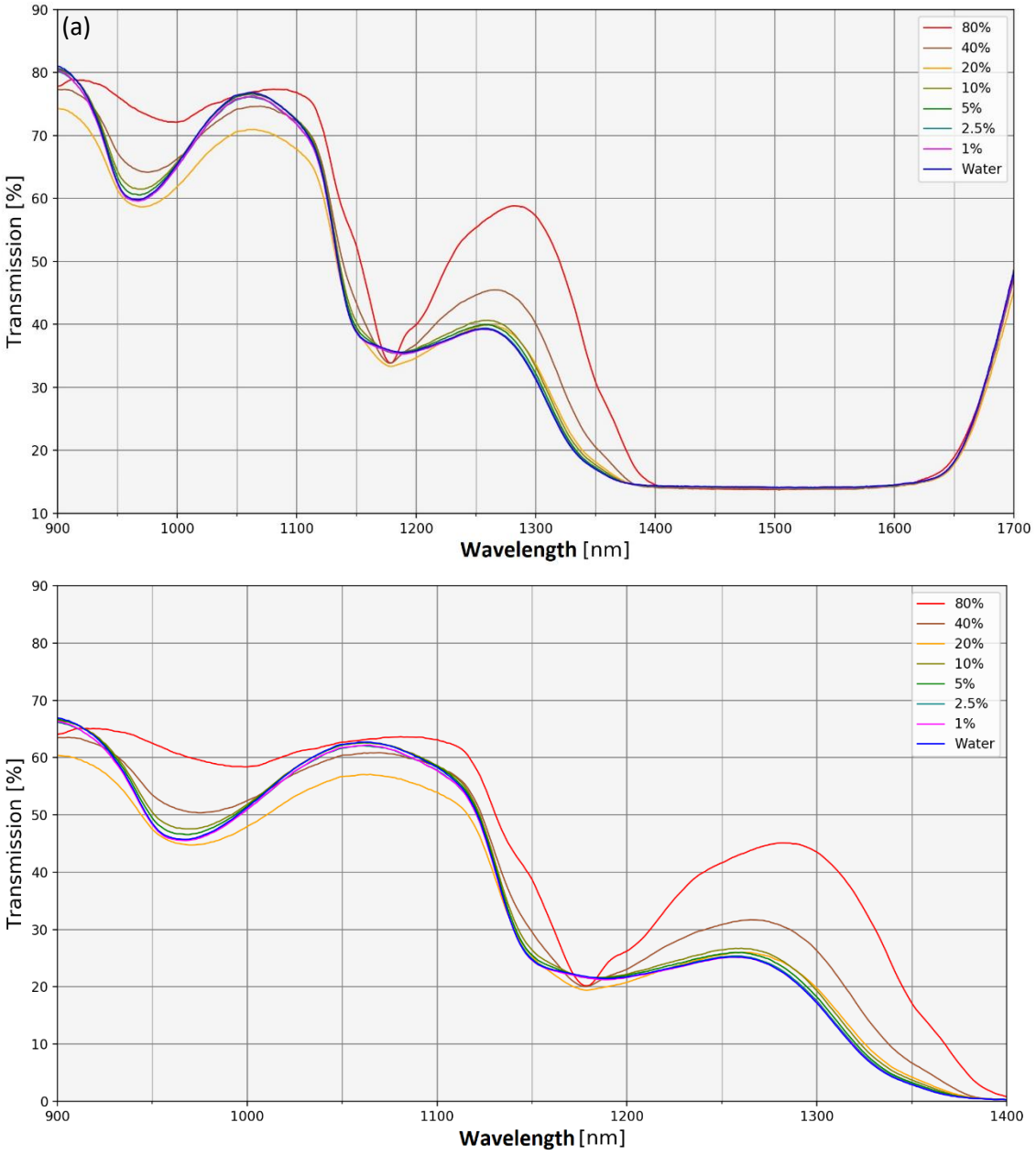


Figure 4: Raw (a) and preprocessed (b) averaged transmission spectra of of 80% - 1% concentrated Ethanol solutions.

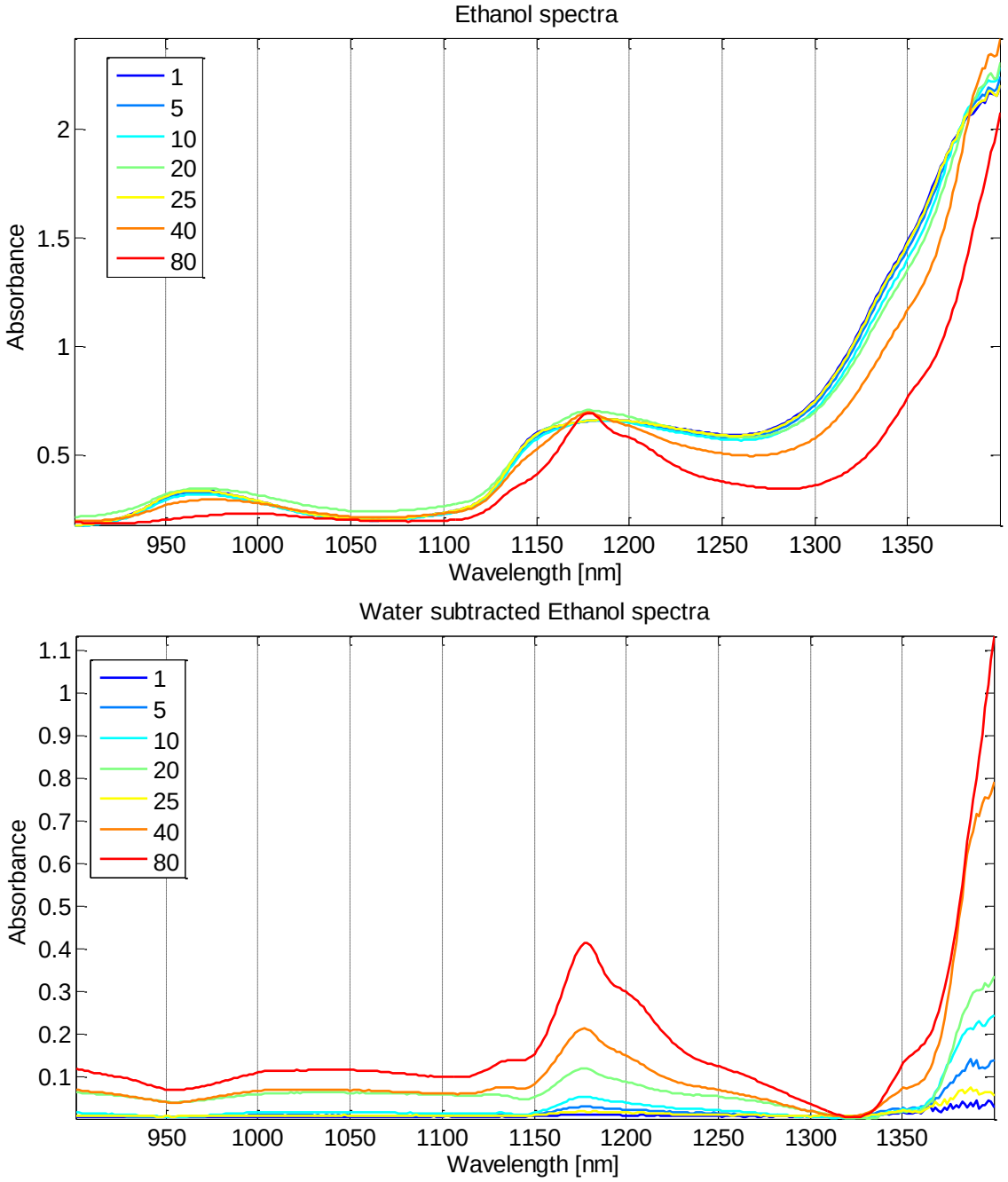


Figure 5: Water absorbance spectrum subtracted from averaged absorbance spectra of 80% - 1% concentrated Ethanol water solutions.

3.3 Peak Decay

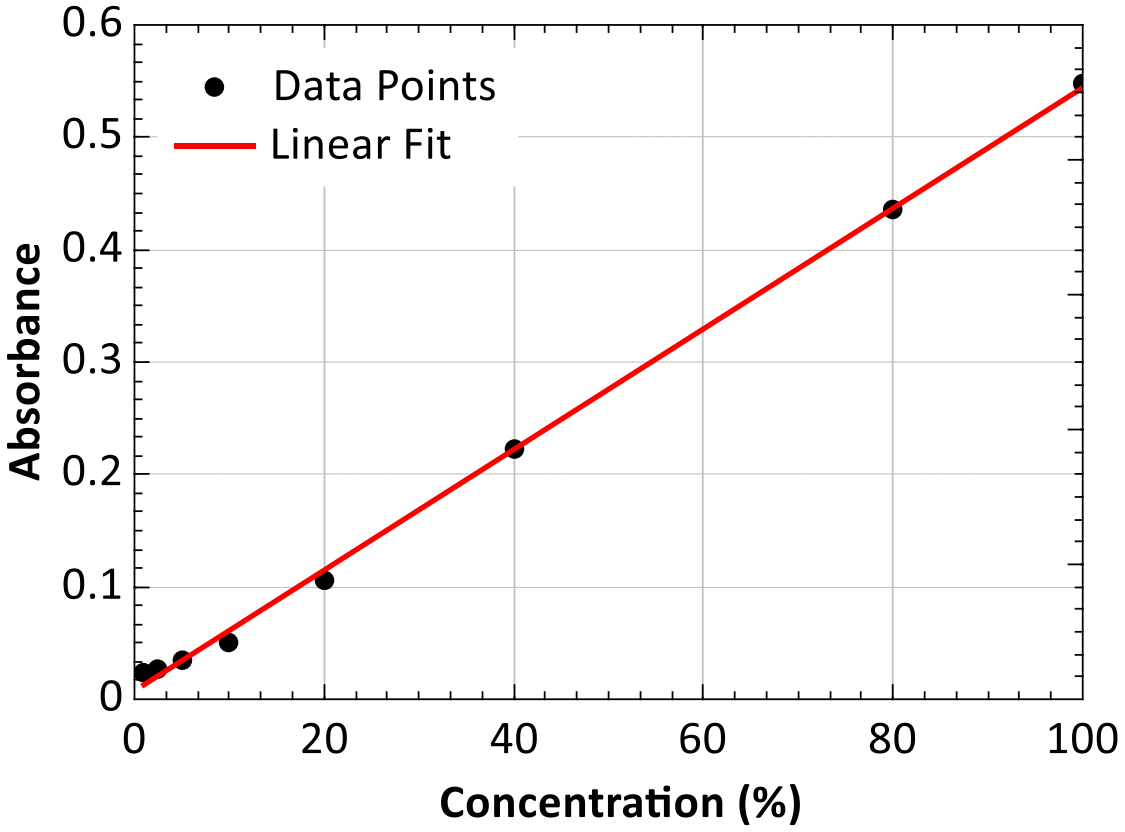


Figure 6: Isopropanol peak decay at ~ 1177 nm with linear fit.

The data was fitted with a linear function, yielding the following results:

Linear: $y = 0.00536 \cdot x + 0.0075$; $R^2 = 0.9988$.

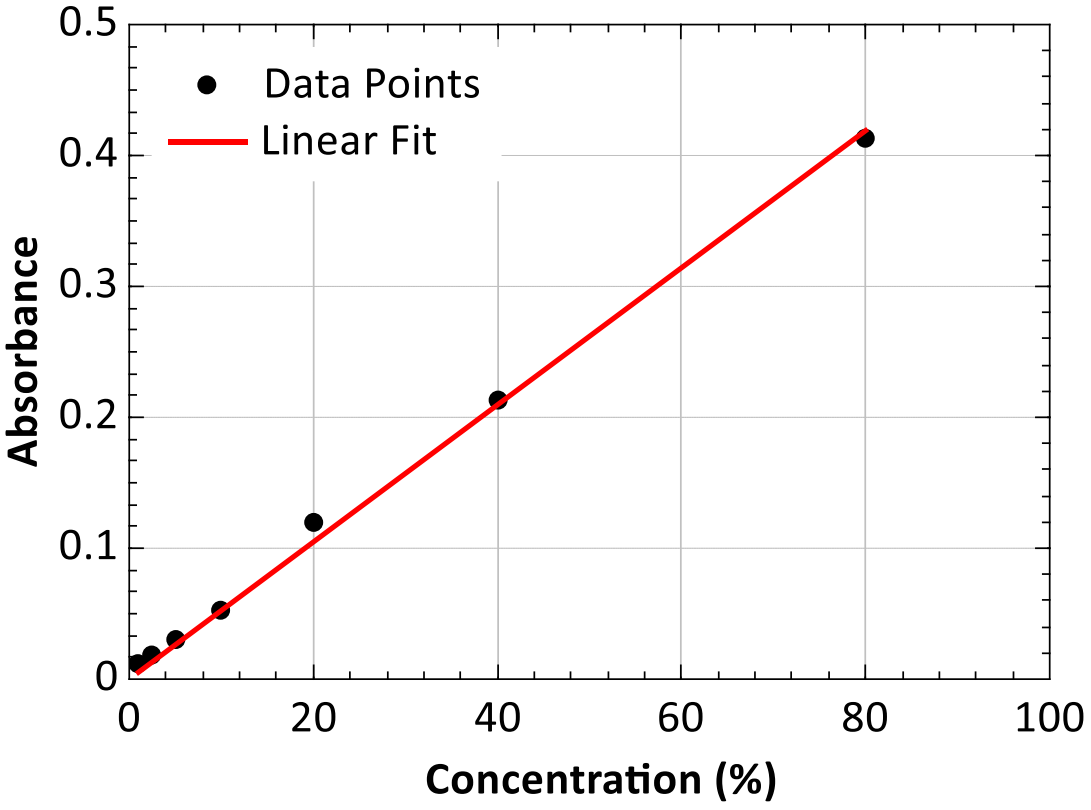


Figure 7: Ethanol peak decay at ~ 1177 nm with linear fit.

The data was once again fitted with a linear function, yielding the following results:

Linear: $y = 0.00523 \cdot x$; $R^2 = 0.9973$.

4 Results

The results of the recorded spectra display a signal with minimal noise and well-defined peaks. A linear correlation is suggested by the analysis of the Isopropanol/Ethanol peak decays at the previously depicted wavelengths. The probe can be used to accurately detect unique peaks at solute concentrations less than 1% by using the visually represented data in the water subtracted spectra.

5 Conclusion

In conclusion, the fiber probe permitted the accurate and dependable detection of a solute down to a concentration of less than 1%. The enhanced design of the transflection fiber probe offers a clear signal with well-defined spectra using the given spectrometer as well as an integration time of 6 ms. The need for curve smoothing algorithms is thus eliminated, which, by extrapolating from the original data, cause a loss of information. Analysis of the peak decays shows high linearity in the sensitivity of the system (probe + spectrometer). Finally, the design of the fiber probe, in particular the shaft head - although it does not prevent the problem from occurring entirely - renders it significantly easier to see and remove air bubbles from the window/reflector in the optical pass in comparison to other immersion fiber probe designs. This prevents the recording of incorrect measurements.

References

[1] John M. Chalmers; Peter R. Griffiths (2007), "*Sampling Techniques and Fiber- Optic Probes*". Handbook of Vibrational Spectroscopy, Online.
DOI: 10.1002/9780470027325.s8902

[2] S. Küppers (2014), "*Application of Optical Spectroscopy to Process Environ- ments*". Handbook of Spectroscopy

[3] Terry R. Todd (2006), "*Fiber-optic Probes for Near-infrared Spectrometry*". Handbook of Vibrational Spectroscopy, Online.
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