Two-Regime Hollow PDMS Abbe Prism for µ-TAS Applications

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ABSTRACT

A new robust and compact system, which comprises a PDMS-based hollow Abbe prism and microlenses, together with self-alignment channels for optical fibre positioning, is presented. This optical detection method has two different regimes for signal transduction, namely absorption and refractive index shift. µM concentrations of fluorescein are detected with this system using standard equipment (SLED and photodetector). The results clearly show the regions in which each physical effect is significant.

Keywords: micro total analysis systems, laser induced fluorescence, optical detection.

1 INTRODUCTION

The application of micromachining to microfluidic systems has been successfully proved in the development of several components, as could be mixers, valves and pumps. In the early 90's, the microfluidic concept was extended with the introduction of a new concept, called miniaturized chemical analysis systems, also known as micro total analysis systems. A scaling down of the analytical systems resulted not only in the reduction in size and price, but also in a significant improvement of the analytical performance **[1]**.
Chip-based capillary electrophoresis (CE) systems have shown to be powerful tools for many promising applications **[2]**,**[3]**. Very often, laser-induced fluorescence detection, a well-established detector in conventional CE-systems, is the method of choice due to its high sensitivity. For efficient measurements the excitation wavelength is focused into the channel. Pure emission fluorescence wavelength measurements are done by placing the collection system and the photodetector at 90° with respect to the direction of the excitation wavelength. Emission and excitation wavelengths are collected together if the photodetector is placed at 180°. In both cases, these set-ups require a considerable amount of optical components, such as lenses, collimators, mirrors, pinholes and filters that have to be aligned very carefully. In relation to the microfluidic device this assembly becomes fairly bulky. The tackling of this drawback goes together with the use of poly(dimethylsiloxane) (PDMS) finding its way into microfabrication. Recently, the integration of optical fibres and lenses for fluorescence detection by taking advantage of the elastomer's extraordinary properties has been reported, on the basis of forming complex three-dimensional structures from a given mold **[4]**,**[5]**. Although the use of PDMS helps on solving some optical problems on LIF devices, the multiplexation of the excitation and emission wavelengths is a problem that still remains. So far, they are incorporated by either using conventional optical components (filters) or complex and expensive technological steps **[6]**, **[7]**.
Exploiting the merits of PDMS in combination with a prism-based optical set-up, we were able to design an optical transducer that is suitable for on-chip detection with a high degree of monolithic integration and without requiring the use of filtering. It comprises a hollow prism, that can be filled with the fluid under investigation via two fluidic ports, and a pair of 2D biconvex lenses that allows obtaining parallel beams both at the input and the output. All these components are fabricated in a single process step by replica molding with PDMS. To seal the microfluidic device, the PDMS is bonded to conventional soda-lime glass. Light is coupled into the system through a multimode optical fibre inserted into a channel. This channel has the same width as the optical fibre. Hence, the optical fibres are directly aligned with the biconvex lenses simply by placing them in the input/output channels. To demonstrate the utility of this detector we examined solutions with different concentrations of fluorescein, diluted in buffer solution, in a range between 5 and 1000 µM. Nevertheless, the use of such a hollow Abbe prism cannot be only restricted to fluorescence-based measurements, since it could also be used for absorption detection of chemical species that does not have appreciable fluorescence, or transparent fluids with different refractive index.

2 WORKING PRINCIPLE

One of the best known applications of the prisms is the measurement of the refractive index (RI). For a given prism, by measuring the incident light angle and the so-called minimum deviation **[8]**, the RI as a function of the wavelength can be obtained. This technique is nowadays one of the most precise ways of measuring the refractive indices of substances (including transparent gases and liquids) in a wide range of wavelengths.

Among the different prisms already been designed, we have focused on the Abbe prism due to its simplicity. Its basic structure can be observed in Fig. 1. The Abbe prism is based upon the combination of three prisms (ADE, AEB and BEC), where δ stands for the total deviation of a ray propagating through the Abbe prism.

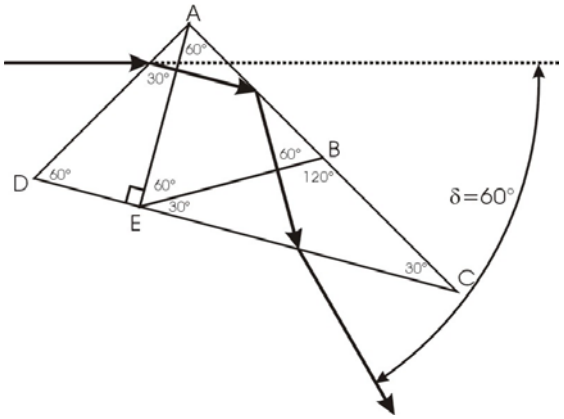

Figure 1: Configuration and ray-tracing in an Abbe prism.

Using the ray-tracing theory, it can be shown that if a monochromatic ray with wavelength λ_1 is injected, with given tilt, in one of the prism's facets, it crosses symmetrically - orthogonal to the AE facet – the ADE prism and is 30° reflected from the AB facet. A total deviation of 60° is obtained after crossing the BEC prism. Hence, the overall system can be thought as a 60° prism (ADE combined with BEC) with the ray propagating in the minimum deviation condition and a second prism that confers the Abbe prism the 60° deviation. With a small rotation of the Abbe prism, the former wavelength is no longer transmitted at 60°, but it is replaced by a second wavelength, λ_2, that matches the conditions for minimum deviation. This behavior causes this type of prisms to be called "constant deviation prisms".

3 DESIGN

After the Abbe prism principles have been described, a new compact detector, shown in Fig. 2, is presented. A hollow Abbe prism is connected to input/output reservoirs. Two multimode optical fibers with PDMS biconvex lenses at the end are defined. With the refractive indices of PDMS and air 1.41 and 1.00, respectively, the distances between the lenses and the optical fibers where chosen so as to have parallel beams at the surface of the prism. The tilt of the optical fibers in relation to the Abbe prism is chosen in order to have maximum intensity for the excitation wavelength (λ=460 nm) when the prism is filled with buffer solution (phosphate buffer, pH 7.4, 10 mM, n=1.334).

The expected behavior of the hollow Abbe prism as the fluorescein concentration, diluted in phosphate buffer, increases, is the following: for low concentration no appreciable RI change of the solution should occur. In this regime, only a decrease of the excitation wavelength peak, without shifting its wavelength, should be observed due to the absorption of light by fluorescein. At high fluorescein concentrations, together with this absorption effect, a change of the RI of the fluid inside the prism is produced, causing a change of the optical path. In turn, this causes the modification of the collected wavelengths at the output optical fiber. Since the prism is designed for the excitation wavelength (460 nm), the high absorption, together with the modification of the optical path will cause a high increase of the sensitivity for high fluorescein concentrations.

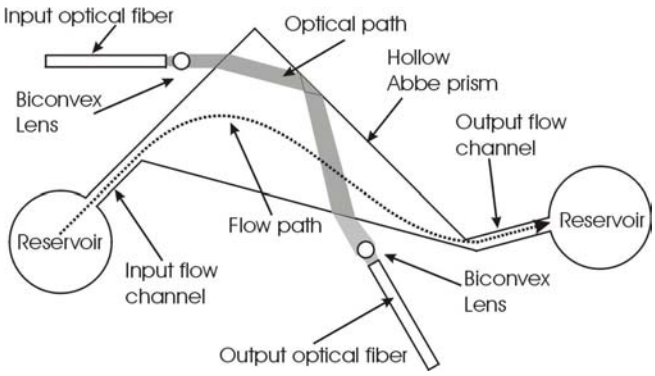

Figure 2: System for fluorescence detection with a hollow Abbe prism (not to scale).

For correct working of the lenses, the optical fiber has to be placed at a given distance from the lenses. Stoppers at the channels fix the distance at which the optical fiber should be placed to obtain parallel beams. Knowing the refractive index of PDMS and air, and designing the radii to be R_1=160 μm and R_2=-R_1, the distance between the fiber and the lens should be S_0=200 μm.

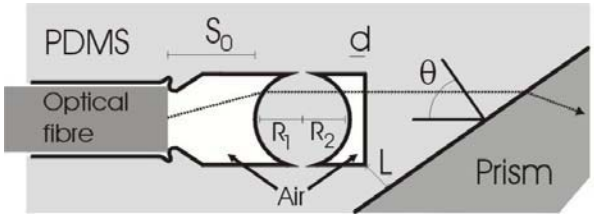

Figure 3: Detailed schematic of the optical fiber channel, the lenses and the prism. Fiber optics are stopped at a distance S_0 which, considering the RI of PDMS and air, together with the curvature of the lens (R_1 and R_2), allows having parallel beams at the biconvex lens output. Lens and channel are tilted an angle θ to have propagation with minimal deviation through the prism. An air gap, with a minimum distance d, separates the PDMS bulk region from the PDMS lens. L remains for the distance between the air gap and the prism in order to avoid leakage.

The propagation through the ADE and BEC prism with minimum deviation also requires the correct tilt of the optical fiber. Considering that the prism is filled with phosphate buffer, a tilt θ=54.8° with respect to the prism wall assures that light is propagating through the prism with minimum deviation. Biconvex PDMS lenses are differentiated from the bulk PDMS by a small air gap, with d=30μm. Since light emerging from the lenses has parallel

beams, which have orthogonal incidence to this air gap, no ray deviation is produced at this gap. To avoid leakage, a security distance, fixed to be L=140 μm is left between the closest corner of the air gap and the Abbe prism.

4 FABRICATION

The microfluidic device was fabricated by casting of PDMS (Sylgard 184 elastomer kit, Dow Corning, Midland, MI, USA) against a master made of EPON SU8 (SU8-25, MicroChem Corporation, Newton, MA, USA). In order to obtain a sufficient height that allows a hassle-free insertion of the optical fibers we used a two-step spin-on process for SU8. After spin-coating and drying the negative photoresist was exposed to UV light through a mask. A post-exposure bake was followed by developing the SU8 in propylen glycol methyl ether acetat (PGMEA, MicroChem Corporation, Newton, MA, USA). The resulting master exhibited a height of 250 μm. A prepolymer of PDMS was prepared by mixing the curing agent and the elastomer base in a 1:10 ratio (v/v) and degassing in a vacuum chamber. After pouring the prepolymer over the master the PDMS was cured on a hotplate at 60°C for 1 hour. Prior to bonding the PDMS slab to glass the elastomer was peeled off from the mold and holes were punched out with a stencil for access to the fluidic channels. We used a bonding procedure based on a surface treatment in an oxygen plasma **[9]**. Both the PDMS slab and the glass were put in a barrel etcher (Surface Technology Systems, Newport, UK) and exposed to an oxygen plasma. Immediately after plasma oxidation the two surfaces were brought in contact and the fluidic system was irreversibly sealed. The total volume of the prism is 1.579 μL.

5 CHARACTERIZATION

The fabricated device can be seen in Fig. 4: light with the excitation wavelength, emitted from an SLED (λ=460 nm), is injected into a multimode waveguide with a core diameter of 200 μm. The optical fibre is placed at the input channel. Readout comprises an identical optical fibre that collects all the light emerging from the prism at 60° and that is connected to a spectrometer (P.117, STEAG MicroParts, Dortmund, Germany) with a spectral resolution of 12 nm and a time of integration of 2.5 sec.

Air entrapment at the unstable upper meniscus can affect the overall properties of the Abbe prism. Although it could be solved by liquid injection at low speed **[10]**, for simplicity and measurement repeatability, the positioning of an auxiliary channel on this meniscus, directly connected to the output port, allows a faster and more homogeneous hollow Abbe prism filling.

Two different regimes can be observed in Fig. 5: For low concentrations (Fig. 5a), only a decrease of the collected intensity as the fluorescein concentration increases can be observed. Since the maximum peak remains fixed at the excitation wavelength, it can be concluded that, in this region, there are no significant changes on the RI of the solution. Light emitted from the fluorescein is emitted with arbitrary directions and with a wavelength (510 nm) for which the Abbe prism has not been designed. Hence, fluorescence (or light at the emission wavelength) is mainly not collected by the output fibre optics. For high fluorescein concentrations (Fig. 5b), in addition to this absorption effect, a shift of the output intensity peak is obtained. This shift is due to a variation of the RI of the solution inside the hollow Abbe prism. As a result, the optical path is modified and the excitation wavelength no longer fulfils the minimum deviation condition, resulting in a sharp decrease of the measured intensity.

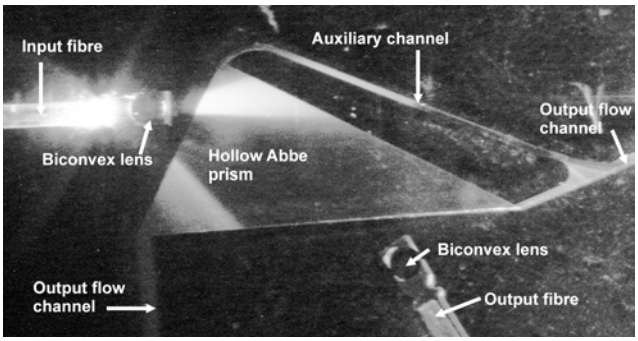

Figure 4: Picture of the system used, displaying the input/output PDMS biconvex lens, the optical fibres, the fluidic channels and the Abbe prism filled with buffer solution + 100 μM fluorescein.

From the previous data and considering the Lambert-Beer law, the absorbance as a function of the fluorescein concentration was calculated. As can be observed in Fig. 6, two regions can be clearly distinguished. For low concentrations, the behaviour of the prism fullfills the Lambert-Beer law, allowing to determine the limit of detection (LOD) to be 4.7±0.1μM. The observed abrupt change on the linear behaviour cannot be explained by only considering fluorescein absorption, since in this region, the RI shift also has to be considered. Due to its non-linear behaviour, no LOD can be calculated, and only the smallest fluorescein concentration (112.5 μm) that gives significant signal of the emission wavelength can be determined.

The confirmation of the RI changes for high fluorescein concentrations is confirmed with the help of Fig. 7. As can be observed, in the pure absorption regime, there is no shift of the excitation wavelength, in accordance to the non-variation of the designed optical path. Conversely, for fluorescein concentrations higher than 100 μM, a shift of the excitation and emission wavelengths collected at the output fibre optics is observed. This shift is caused by the modification of the optical path inside the prism, or, in other words, due to a shift of the RI.

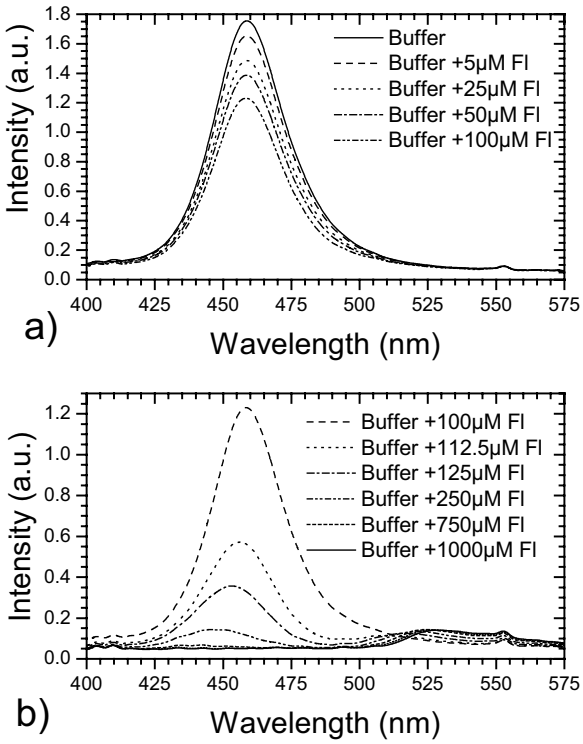

Figure 5: Output intensity as a function of the wavelength for different fluorescein concentrations in phosphate buffer a) at the pure absorption region, b) at the absorption-RI shift region.

6 CONCLUSIONS

A PDMS-based hollow Abbe prism for detection based on absorption and RI shift has been presented. By the implementation of biconvex lenses and self aligned channels for optical fibers, the external parts of the setup have been reduced to a connected SLED and a spectrometer. Two different working regions can be distinguished: at the pure absorption region (fluorescein concentrations below 100 µM), the Abbe prism matches the Lambert-Beer law, with a calculated LOD of 4.7±0.1 µM. Conversely, for higher concentrations, the RI shift inside the prism cannot be neglected, since it causes a modification of the optical path. As a result, a sharp deviation of the linear behavior is observed. Due to the non-linear behavior, no LOD can be calculated for this region. However the minimum fluorescein concentration detectable, by ways of measuring the emission wavelength, is 112.5 µM.

7 ACKNOWLEDGEMENTS

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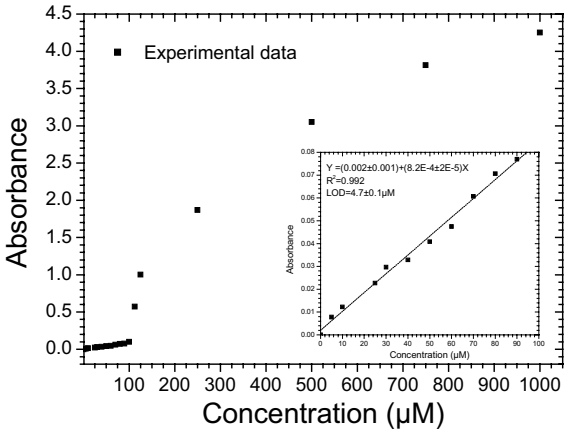

Figure 6: Absorbance as a function of the fluorescein concentration, Inset: Linear region, where the Lambert-Beer law applies.

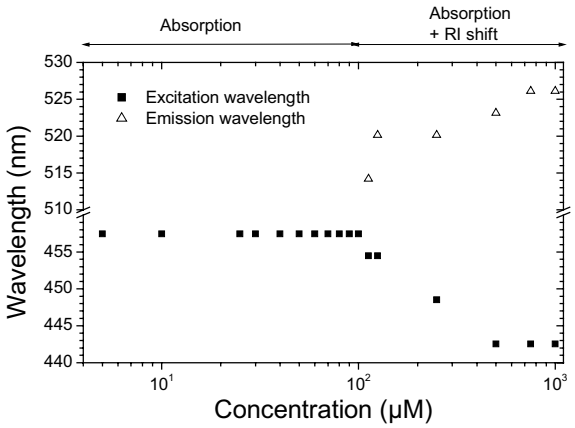

Figure 7: Transmitted excitation and emission wavelength as a function of the fluorescein concentration.

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