Fluorescence-based optical fiber sensor for liquid sample analysis in silica glass well

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ABSTRACT

Liquid samples are often of interest for various analytical purposes, requiring rapid, precise, portable, and easy-to-use equipment. Then, we proposed a portable system for fluorescence spectroscopy was developed, integrating an optical fiber sensor into a single-well plate. A proof-of-concept of the system was demonstrated using two different fluorescence samples: chlorophyll and cyanobacteria culture. The manufacturing method employed was ultrafast laser-assisted etching (ULAE), and the optical fiber was custom-mounted for tip-endcap integration. The fluorescence analysis demonstrated an enhancement in the signal of the liquid sample through the integration of the fiber capture system in the detector.

Keywords: fluorescence, fiber sensor, ULAE, well

1. INTRODUCTION

The analysis of liquid samples is a common task in laboratory analytical work. The methods used can be divided into three main categories: separation methods, microscopy, and spectroscopic techniques [1]. Additionally, extraction methods are often employed to enrich the content of liquid samples and facilitate the use of these techniques. Separation methods such as distillation, evaporation, and filtration are time-consuming and require a high level of expertise. Microscopy imaging, on the other hand, requires constant manipulation and typically involves expensive equipment. In contrast, spectroscopic techniques such as absorption, fluorescence, or Raman analysis offer faster systems without the need for complex sample preparation steps, although conventional equipment remains bulky and costly. However, some miniaturized systems have already been proposed. However, some miniaturization systems have already been proposed [2]. In this sense, high performance liquid chromatography has an on-chip version. This is a separation method for identification and quantification of the sample components.

In this work, a miniaturization of a fluorescence spectroscopic system is proposed for liquid sample analysis, aimed at improving both portability and ease of use. The system incorporates an optical fiber sensor, which is specifically designed to collect the fluorescence light emitted by the liquid sample upon excitation. This integration allows for more efficient light collection, enhancing the sensitivity of the system.

2. PROPOSED DESIGN

The conventional one-well plate configuration was modified to implement optical spectroscopic analysis in a rapid and user-friendly manner. The integration of an optical configuration in a small space was conceived by integrating fiber optics for the detection of the emitted light. Then, the distribution of multiple fibers along the well wall was considered. In particular, a square well with 4 fibers in the centre of each side of the well was intended. Then a custom one-square-well plate was designed to allocate fiber entries in each side center. Figure 1 shows the proposed design whose dimensions specifications are included in Table 1.

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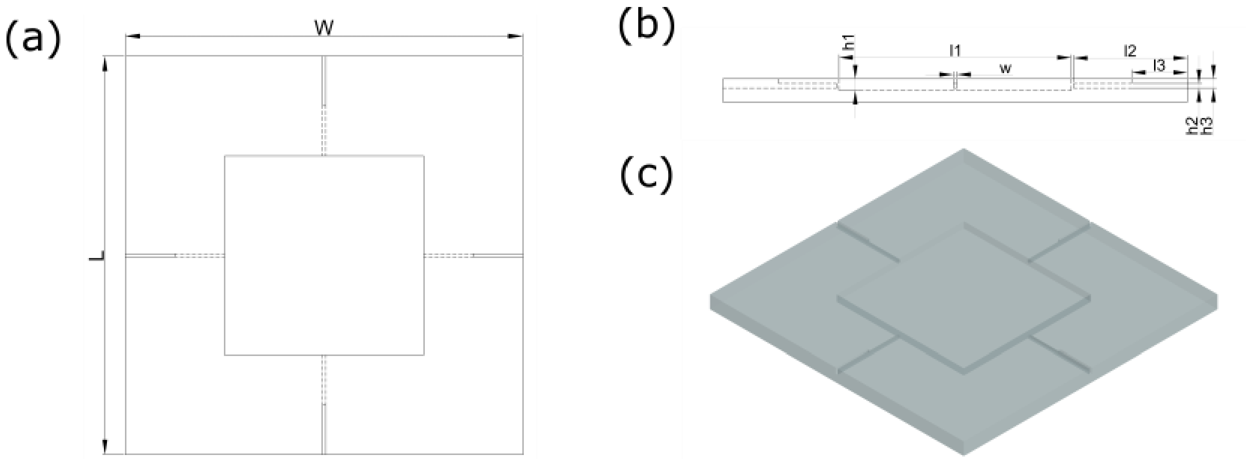

Figure 1. Details about design parameters. Top view (a) and side view (b) include labels whose values are given in Table 1. 3D view is provided in (c) and (d) correspond to a photo of the first prototype.

Table 1. Dimensions of the design according to labels in Figure 1. Units are indicated in brackets.

Global sizes (mm)		Lengths (mm)		Heights (µm)	
W	20	l1	10	h1	500
L	20	l2	4.9	h2	200
w	0.155	l3	2.5	h3	400

3. MANUFACTURING PROCESS

3.1. Custom well plate

The fabrication method was Ultrafast Laser-Assisted Etching (ULAE) [3]: a two-step technique of substrative laser manufacturing. The material selected was fused silica and a commercial glass of dimensions 20x20x1 mm from FOCtek was used. Initially femtosecond laser pulses are focused on the glass inscribing the contour of the design to modify the material in those localized points and then the glass is chemically etched, where the pristine material etching is 3 orders of magnitude lower than in the processed points. Particularly, this inscription was extended in size 100 µm from the glass size to ensure identical inscription conditions and the laser inscription parameters were 9 kHz for PRR and 350 nJ for pulse energy. Moreover, a condition of low pulses regime ensures a polarization independent inscription [4]. Then the glass was etched in a NaOH solution (5%w), maintained at 85±2 ºC and magnetically stirred at 500rpm during 5h. Finally, it was cleaned using distilled water to remove all the extracted parts and any glass fragments.

3.2. Custom alignment structure

A custom endcap fiber was fabricated to insert the fibers from the well together. The fibers were in a stainless stain capillary fixed in a resin mount for screw it in the fiber connected to the spectrometer. The capillary was 0.8mm ouside diameter and 40 mm length while the resin piece was printed and then washed and cured using FormaLabs products.

3.3. Complete device assembly

Once fiber channel access in the glass was verified a 100/140 µm fiber (NA = 0.28, from Corning) was fixed using optical adhesive (Norland 601, from Thorlabs) inside each on them. In this prototype there were three free access. Then, all the fiber ends were coupled together in the custom endcap which finally was connected to a fiber directed to the spectrometer.

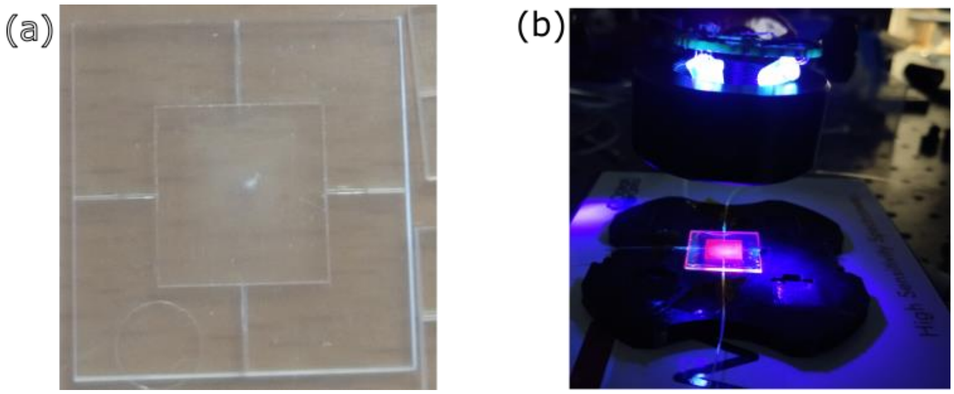

Figure 2. (a) Silica glass well with four channels for the fiber sensor assembly before ULAE manufacture. (b) Fluorescence-based optical fiber sensor during measurements.

4. PROTOTYPE VALIDATION: RESULTS AND ANALYSIS

4.1. Validation criteria.

A proof-of-concept of the proposed design was evaluated using two different liquid samples. First, a fluorophore was selected: chlorophyll extraction; then, a liquid sample of a pure culture of cyanobacteria: *Dolichospermum crassum* specie, labeled as UAM502 in the Department of Biology at the Autonomous University of Madrid who provided the sample. Both samples were chosen as reference material due to their well-established behavior in fluorescence spectroscopy: chlorophyll require UV excitation with emitted fluorescence in visible range (around 680 nm) and UAM502 exhibits fluorescence emission in the red spectrum, near the excitation point.

The system configuration was maintained for both cases: the excitation light source was positioned above the well, and the emitted light was directed to a commercial spectrometer (Maya2000Pro-series from Ocean Optics). The excitation light source was focused onto the liquid sample and the emitted fluorescence was captured using the fibers that were coupled to the spectrometer using the custom endcap connected to a commercial fiber of 600 µm (from Ocean Optics). Here below are presented the fluorescence measurements of each case obtained when using a single fiber, a couple of them or three fibers.

The results demonstrated fluorescence emission patterns for both samples showing intensity enhancement when combining collection. Thus, the design was validated and its potential for liquid sample analysis in fluorescence spectroscopy was confirmed.

4.2. Pure chlorophyll extraction sample

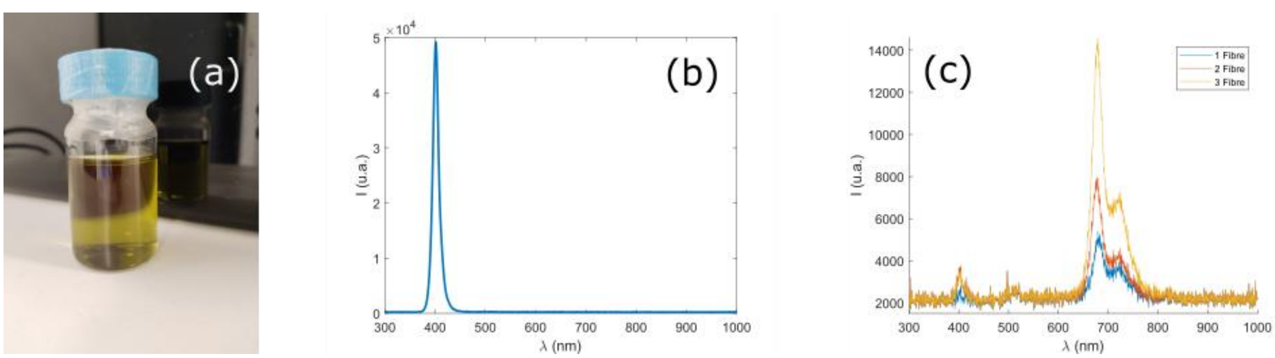

Figure 3. (a) Photo of the sample of chlorophyll. (b) Spectrum of the excitation light source: ring of 3 UV-LEDs. (c) Emission spectrum from the fluoresce measurement of the chlorophyll sample when validate the optical fiber sensor comparing fiber integration configurations.

4.3. Pure cyanobacteria culture

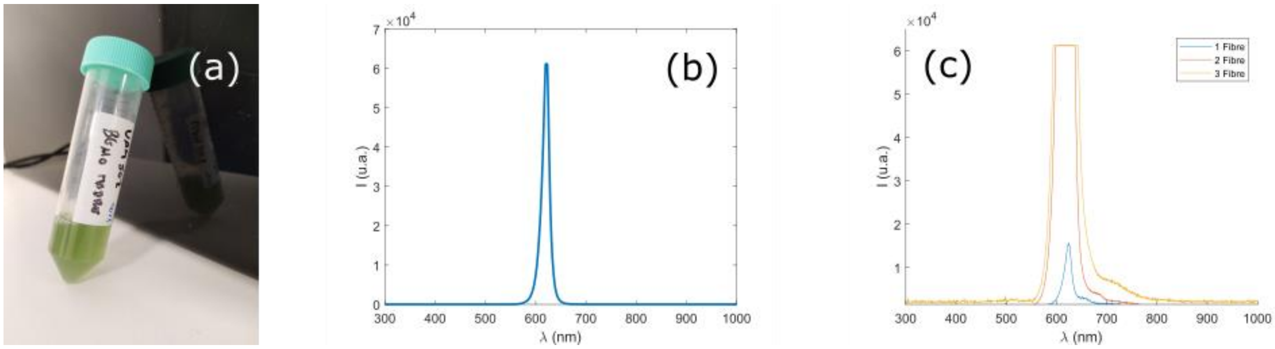

Figure 4. (a) Photo of the sample of chlorophyll. (b) Spectrum of the excitation light source: red superLED. (c) Emission spectrum from the fluorescence measurement of the chlorophyll sample when validating the optical fiber sensor comparing fiber integration configurations.

5. CONCLUSIONS

A device for optical liquid sample analysis was designed and prototyped on a fused silica glass using ULAE technique. The proposed device consists of a well plate with three fibers positioned to face the walls of the well. The proposed design was evaluated for fluorescence spectroscopy analysis using samples of chlorophyll and pure cyanobacteria. The results demonstrated an enhancement in the emission spectra of both liquids when the fibers were coupled together and in the spectrometer.

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