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Optical sensor based on periodic array of resonant nanopillars for real time monitoring

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ABSTRACT

Here we present the real time response of a recently reported optical transducer, a periodic array of resonant nanopillars (R-NP). The signal of the sensor was obtained with a common spectrophotometer by measuring light transmission through the R-NP surface. A flow-cell, connected to a peristaltic pump, was employed to load different refractive index (RI) solutions in order to assess R-NP bulk sensitivity and resolution (limit of detection, LoD). Besides spectra, real time sensograms at constant wavelength were recorded. Excellent resolution and fluidic behavior were found, indicating the suitability of the R-NP system for real time biosensing applications.

The results were also compared with other optical sensor (LSPR, localized surface plasmon resonance). Both sensors were assayed with the same optical and fluidic setup, in order to evaluate the differences between transducers in the same conditions. Bulk sensitivity and resolution parameters resulted 4 and 5 times better respectively for R-NP compared to LSPR.

1. Introduction

In the last decades, different kind of optical sensors have been developed and applied to label-free detection of a wide range of target analytes. This field is continuously evolving towards improvement in several technical issues like new transducer research, miniaturization of devices, cost-effective developments or simplification of optical interrogation methods [1–7].

Recently, a new kind of transducer based on periodic arrays of nanopillars has been described [8–12], and has proven a great potential for high yield biosensor development. Particularly, resonant nanopillars (R-NP) composed by two Bragg reflectors (SiO_2/Si_3N_4) and a central cavity of SiO_2 showed very interesting results, achieving resolutions close to $1{\cdot}10^{-5}$ RIUs (refractive index units) [12,13]. In these works, optical interrogation was performed by registering reflectivity with high-resolution FT-VIS-NIR spectrometer, and high capacity for biosensing has been proven. Eight sensing arrays were measured on the same chip [13]. In present paper, we use also a R-NP based transducer, but a new interrogation method – based in the use of common spectrophotometer and light transmission measurements – was employed. In addition, this is the first time real time response of R-NP is registered, by measuring signal changes at a constant wavelength. For this approach, we employed a fluidic system that has been previously used as flow-cell in a LSPR biosensor [4]. At present work, we changed the LSPR sensing surface by the R-NP transducer, and the obtained sensing parameters were compared. Results of the R-NP system confirm the high quality of this novel optical transducer.

On the other hand, we demonstrate that the system "spectrophotometer + sensing cuvette" can be employed for the interrogation of different optical transducers in transmission mode, making it suitable for the acquisition of spectra and real time sensograms.

2. Materials and methods

2.1. Chemicals

Sodium chloride (NaCl, >99%) was supplied by Sigma-Aldrich (St. Louis, MO). Aqueous NaCl solutions were prepared dissolving

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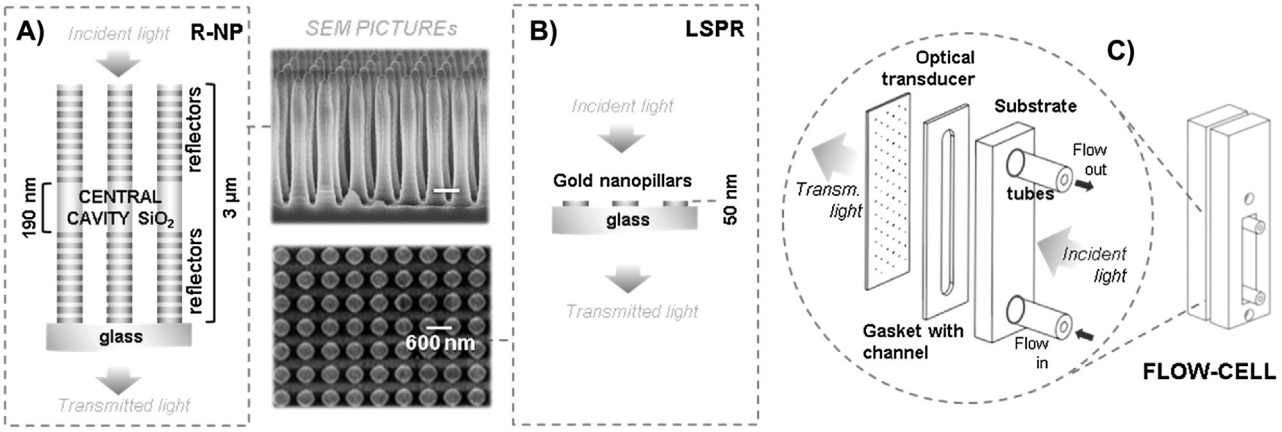

Fig. 1. Scheme of the R-NP (A) and LSPR (B) optical transducers. Representation of flow-cell for optical interrogation (C).

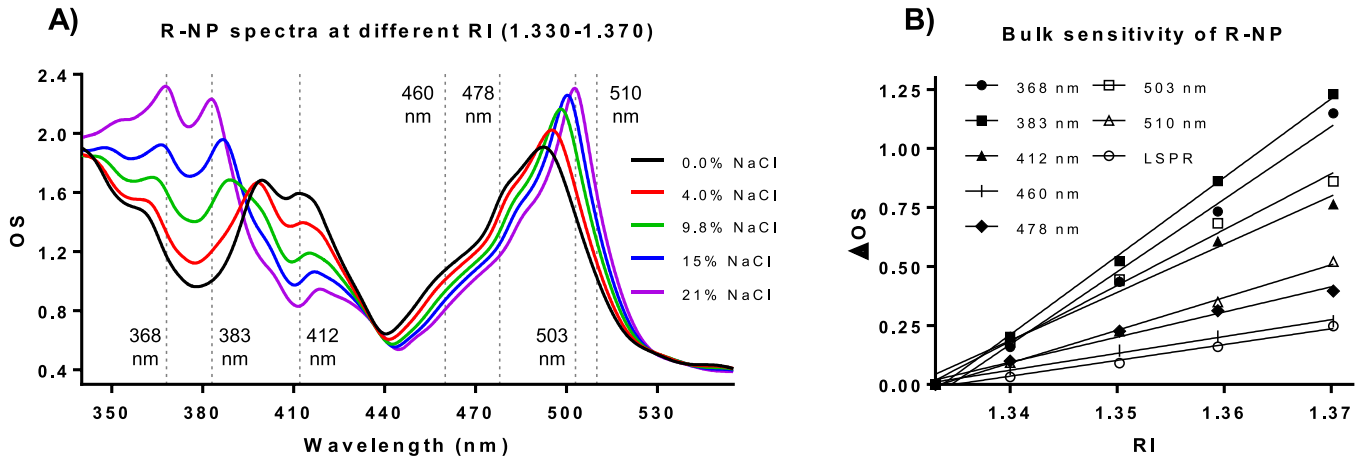

Fig. 2. A) Spectra of R-NP surface at different RI (1.330–1.370). B) Sensitivity (ΔOS vs. RI) of the R-NP system at different wavelengths. Sensitivity of a LSPR transducer is also included (λ = 840 nm) [4].

different amounts of NaCl in ultrapure water in order to obtain different refractive index (RI) solutions.

2.2. Sensor and holder set-up

The period of R-NP is 600 nm, the height is 3 μm and the diameter is in the order of 350 nm. R-NP are composed by a SiO_2/Si_3N_4 multilayer stack, with 18 pairs of reflectors and a central cavity (SiO_2) of 190 nm, placed on a glass substrate (see Fig. 1A). The fabrication process has been previously reported [14]. For LSPR surface, the period was the same, and height and diameter of gold nanopillars were 50 and 440 nm, respectively [4]. See Fig. 1B.

The employed flow-cell and interrogation system, has been recently published [4]. Briefly, a silicone gasket is placed between the sensing surface and a transparent substrate creating a microfluidic channel (8.5 μL). This system is placed inside two taps with *standard cuvette* shape, suitable for the cuvette compartment of common spectrophotometers, (see Fig. 1C). Optical response was registered with spectrophotometer JASCO-V670. Spectra – Optical Signal (OS) vs. wavelength (λ) – was measured at 200 nm/min, with data pitch 0.1 nm and bandwidth 2 nm.

The OS value is calculated as logarithmic ratio of the intensity of transmitted (I) and incident (I_0) radiation through the RNP transducer (-log (I_0/I)).

Sensograms (OS vs. time) were measured at fixed wavelength, (383 and 503 nm for R-NP and 840 nm for LSPR), with data pitch 0.5 s and bandwidth 2 nm. By means of a peristaltic pump, solutions with different RI were loaded in the flow cell.

2.3. Bulk sensitivity and resolution

Spectra at different RI were measured in order to choose the most sensitive wavelengths. The spectra are shown in Fig. 2A. For it, different RI solutions were loaded in the system. For bulk sensitivity assessment, linear regressions were constructed by representing the increment of optical signal (ΔO.S.) vs. RI (RIUs), at different wavelengths (see Fig. 2B). The slope of the curves (variation of signal per unit of RI, OS/RIU) represents the sensitivity of the system for each wavelength.

Real time response (sensograms) of the solutions was recorded by flowing the sequence: 0.0, 4.0, 9.8, 15, 21, 15, 9.8, 4.0 and 0.0% of NaCl (five min each, at 50 μL/min). The corresponding RI values are: 1.3330 (0.0%), 1.3400 (4.0%), 1.3502 (9.8%), 1.3594 (15%) and 1.3702 (21%). These sensograms are included in Fig. 3A.

The resolution (LoD) was defined as the minimal RI change which generates a detectable signal (OS = 3·N, where OS is optical signal and N is noise). The noise (fluctuation of OS) was assessed from sensogram when a solution is flowing (Fig. 3B). The resolution value (RIUs) was calculated dividing the value of detectable signal (OS) by the achieved R-NP sensitivity (slope, OS/RIU).

To evaluate the response at low RI changes, solutions with lower amount of NaCl –0.00, 0.01, 0.02, and 0.03%- were also tested in the