Continuous measurement with three-in-one plasmon sensor in sucrose solutions

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Abstract— Surface plasmons are a revolution in nanophotonics because they permit the transport of energy along metal-dielectric interfaces, something which is of vital importance in sensors which measure refractive indexes. The aim of this research has been the design and optimization of several surface plasmon resonance (SPR) sensors to measure the refractive index of a solution of sucrose and water (BRIX degree) in real time. The optimization and design of these sensors was made from the theoretical modelling of the reflectance curves, which has allowed us to determine which the most favourable configuration is. The sensors proposed are modulated in intensity and they have the advantage of not having mobile parts, so, their lifetime is longer.

We propose a plasmonic sensors (three-in-one) for measuring different sucrose concentrations. As the refractive indexes vary very little with the concentration, we have designed a three-in-one sensor to cover all concentrations from 0-80% BRIX degree. The proposed sensors operate by ranges of refractive indexes (low, medium and high). They are made up of a hemispherical prism and a layer of gold following the Kretschmann configuration. The sensor working in the low zone has a sensitivity of between 22.95 and 4.64RIU^{-1} and a resolution of between 4.3x10^{-4} and 8.7x10^{-5}RIU, the one used for the medium zone has a sensitivity of between 21.05 and 3.89RIU^{-1} and a resolution of between 5.1x10^{-4} and 9.5x10^{-5}RIU, and the one for the high zone has a sensitivity of between 19.60 and 4.64RIU^{-1} and a resolution of between 4.3x10^{-4} and 1.0x10^{-4}RIU.

Index Terms— Concentration of sucrose, nanosensors, optical sensor, soft drinks, surface plasmon resonance.

INTRODUCTION

The Plasmonics is a field of high growth, part of nanophotonics, based on the study and applications of the interaction of the conduction electrons of metal-dielectric interfaces of nanometric thickness with electromagnetic radiation, with the result of this being superficial plasmons, whose properties have motivated many studies throughout their history and which have many applications in various fields of science (Physics, Medicine, Chemistry, Biology, etc.).

Different types of sensors have been proposed in the literature for the measurement of the concentration of sucrose in solutions, for instance, in [1], a multifunctional sensor for measuring concentrations of ternary solutions with NaCl and sucrose used in an osmotic dehydration process has been proposed. The sucrose concentration is determined from the combination of three solution parameters: temperature, ultrasonic velocity and electrical conductivity.

Turton et al. [2] developed a sensor to measure sucrose in alcoholic beverages using Love-mode acoustic waves. Measurements were made with sucrose solutions of up to 50%. The behaviour of this sensor is linear for low viscosities, but the sensitivity is reduced for higher viscosities due to the non-Newtonian behaviour of the fluid.

There is another sensor based on Fresnel reflection with optical fiber to identify the sucrose or ethanol content in samples extracted from a container. The error of the sensor is less than 3wt% for the sucrose and less than 5.1vol% for the ethanol. The concentration of solute varied from 0 to 50wt% for sucrose solutions, and from 0 to 100vol% for ethanol solutions at 22ºC [3].

In the paper [4], the authors reproduce the Pieris rapaes butterfly optical nanostructure and use it as a sucrose sensor in the range of 0g/L to 250g/L with a refractive index increase from 1.333 to 1.36. The parameter measured was the reflectance in function of the concentration of sucrose due to the light scattered

(Mie scattering) on the artificial butterfly wing structure.

In 2014, Gorma et al., [5] designed and optimized an integrated hybrid surface plasmon biosensor and simulated the results for a medium with refractive indexes between 1.33-1.34. It had a very high sensitivity, of around 3000nm/RIU and a resolution of 3.34 10^{-6}RIU.

In the study [6], the authors carried out the fabrication and checking of a sensor to measure sucrose with onion membranes modified with invertase and gold chloride. The sensor was based on formation of invertase-induced nano-gold clusters and particles within the membrane and in the fluorescence phenomenon. The linear fitted between the sucrose concentration and the fluorescence intensity is $R^2 = 0.952$. The lower limit to detection is $2x10^{-9}$M and the dynamical range (where the sensor has a linear behaviour) is $2.25x10^{-9}$ to $4.25x10^{-8}$M.

Another kind of sensor is based on microwaves [7]. Essentially, it is a coplanar waveguide loaded with a split ring resonator for measuring the sucrose concentration. The relationship between the transmission coefficient and concentration is almost linear ($R^2 = 0.991$).

In 2017 a high-sensitivity sucrose sensor based on a standard erbium-doped fiber ring laser incorporating a coreless fiber [8] was developed. It measures the sucrose concentration in aqueous solutions. The device is able to measure concentrations of sucrose between 0-60% with a average sensitivity of 0.57nm/%. A single-mode-coreless-single mode is part of a crystal fiber Mach-Zehnder interferometer and this optical fiber is submerged in a sample of sucrose concentration. In the same year, a sucrose sensor by means of functional Cu foam material was constructed. As with the former sensor, it is necessary to take a sample of the total mixture [9].

In 2019, an etched fiber Bragg grating based sensor was designed [10]. The foundation of the sensor is to detect evanescent fields to detect the refractive index. It has been validated with mixture of water/sucrose which, depending on the proportion of each, changes the refractive index.

In the specific case of soft drinks, a flow system based on a multi-commutation approach for the determination of glucose and sucrose employing enzymatic reactions was developed in 1999. The measures were made with samples extracted from a tank and the results were not in real time. The system was able to measure 30 samples per hour [11]. In the same year, a method to measure glucose concentration in soft drinks was developed [12]. It was again necessary to extract a sample. The base of the method was the glucose oxidase-catalyzed oxidation of the analyte. The limit of detection was 10mmol/l (1.8ppm). This method measures the glucose concentration in soft drinks, after their elaboration.

Ilaslan et al. developed a fast, low cost technique based on Raman spectroscopy combined with chemometric methods, to quantify glucose, fructose, and sucrose in commercial soft drinks [13]. It is a very rapid method for evaluating the quantitative analysis of glucose, fructose, and sucrose, but it is only able to measure in already elaborated soft drinks. It is necessary to extract a sample of the liquid.

Specifically for cola drinks, there is a sensor to determine sucrose and phosphate simultaneously [14]. It is based on the schlieren effect. The device does not measure in real time since it needs 171s to measure, the sample has to be prepared and it does not measure directly in the tank. Moreover, the sensor only measures from 1 to 12Brix.

In the field of surface plasmon resonance (SPR) sensors, an optical fiber based localized surface plasmon resonance sensor using graphene oxide encapsulated Au nanoparticles were manufactured as a sucrose sensor [15]. The cladding of the optical fiber is substituted by graphene oxide encapsulated Au nanoparticles. This optical fiber is submerged in the sucrose solution. The problem of this sensor is that the absorbance depends on time. It has checked with 5 refractive indexes (5 concentrations) with a refractive index of the analyte (sucrose) varying from 1.3395 to 1.3790. The resolution of this sensor is 8.7×10^{-5}RIU.

Some authors have proposed an SPR to measure the concentration of sucrose with water [16]. This is made up of a titanium/silver thin film on indium-tin-oxide coated glass. According to the authors, silver improves the sensor. Moreover, the roughness of the top layer influences sensor sensitivity when the roughness increases (the thickness decreases) and thus the sensitivity also decreases. This sensor works in angle interrogation.

Another SPR sensor based on hetero-core structured fiber optic was applied to measure the refractive index of sucrose solution and fruit juices (tomato, grapefruit, melon, orange, etc.) [17]. It is constructed with a short length of single-mode fiber optic inserted and spliced in a multi-mode fiber optic. The hetero-core structured fiber optic SPR sensor was made with silver deposited on the hetero-core part. The sensor uses the Kretschmann configuration.

The consumption of sucrose is increasing every day in the food industry and it is important to know the sugar content in these types of drinks. 99.5% of the sugar present in these drinks is sucrose [18], therefore, it is essential to have some measurement system for this sucrose. Moreover, sucrose is an essential substance for human beings, but excessive consumption is harmful to health.

Sensors based on the resonance of surface plasmons are mainly used to measure the refractive index of a substance, and its principle of operation is based on achieving the excitation of the surface plasmon by coupling the wave vector of an evanescent wave and the plasmon itself.

From reviewing the papers mentioned above we can confirm that there is as yet no sensor available that is capable of measuring sucrose concentrations in real time without extracting a sample. When a sample is extracted, it is no longer real time, and it is physically impossible to extract a dissolution sample from a specific point in a tank, because when extracting the sample, it will become mixed with other parts of the liquid in the tank. Therefore, we have designed a plasmonic sensor made up of three (three-in-one) sensors that measure the refractive index (and therefore the concentration of sucrose in water) in the range of 0-80BRIX in a continuous way and at any moment during the dissolution.

The decision to design three sensors (three-in-one) was taken with the aim of optimizing the design of each of them in different ranges of the refractive index of the solution. We have designed each sensor to have varying thicknesses of the layers of materials that make up the sensor or by even adding or removing layers of the same or other materials, and we have been able to maximize the sensitivity of each sensor in every range of sucrose concentration, thus making these sensors applicable in the manufacture of different soft drinks where either one sensor or another can be used according to the area of concentration in which it is going to operate, and thus ensure a greater reliability in the sucrose content.

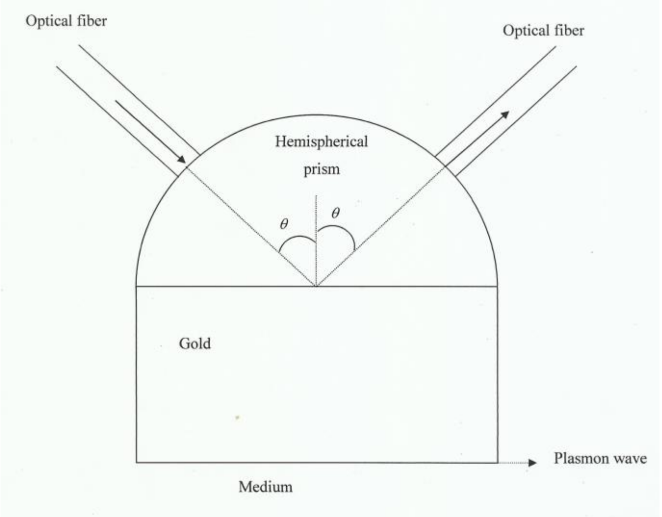

Fig. 1. Scheme of the first plasmonic sensor. On the left side of the hemispherical prism, the optical fiber transports the incident radiation and on the right, another optical fiber collects the reflected radiation. SPPs are also, shown propagating through the gold-medium interfaces.

The sensor we propose has an excellent application for determining the concentration of sucrose in soft drinks, sugary drinks, syrups, sweetened condensed milk, etc. Moreover, in the production process, the mechanism for pouring liquid (water or any other liquid) and adding sucrose could be connected with our sensor which in real time is able to determine the concentration of the sucrose.

DESIGN OF THE PLASMONIC SENSOR

The operational principle is based on the SPR [19].

We have design three-in-one sensors to measure the refractive indexes modulated in intensity based on the resonance of surface plasmon polaritons (SPPs). We have used the Krestchmann configuration [20] (with and without dielectric layer) with different materials and thicknesses in each sensor. These sensors work by ranges of sucrose (high, medium and low) in the solution). They can be considered as three sensors in the same device, (see Fig. 1).

The medium is a solution of sucrose and water which refractive index is in [21]. All data are obtained at 20ºC, 1atm and are measure with a wavelength of 589nm (sodium D line).

The used parts of the sensor in the Krestchmann configurations are:

- Laser emitting polarization P at 589 nm (sodium D line) with S polarization, SPPs is not excited, for this reason, it is used the P polarization [22].
- Prism: hemispherical prism SF10 with refractive index Np = 1.72803 [23].
- Gold: golden layer with refractive index Nm = 0.18559 + 2.8638i [24].

The detector is used to collect the light reflected in the interface glass-gold. Optical fibers transport the light from the laser to the hemiprism and from the hemiprism tothe detector.

An electronic system processes the response of the detector to set up the signal and give us the reflectance in each measurement. The sensor is connected to a PC to show the measurement results of the refraction index in real time and, therefore, the concentration. The software has programmable alarms to warn if the solution is above or below the desired concentration. Of course, data can be sent to remote points, so, it is not necessary for the operator to be in the place where the measures are being taken.

We have divided the BRIX degrees into three zones. The low refractive index zone, from 1.3333 to 1.3855, that corresponds to 0 to 30-35BRIX. The thickness of the Au layer is 49nm and the incidence angle is 59.7º, the medium refractive index, from 1.3820 to 1.4414, that corresponds to 30-35BRIX until 58-60BRIX. The thick of the Au layer is 48nm and the incidence angle is 64.4º, and finally, the high refractive index, from 1.4345 to 1.4940, which corresponds to 56-58BRIX to 80 BRIX approximately. The thickness of the Au layer is 45.8nm and the incidence angle is 70.5º.

RESULTS AND DISCUSSION

1 Sensor for low sucrose concentrations

This sensor is designed to work with sucrose solutions that have a refractive index of between 1.3333 and 1.3855 (from 0 to 30-35BRIX). To achieve optimum sensor sensitivity and resolution, the sensor has been designed so that the reflectance curves shown in Figure 2a have the maximum possible separation between them and also cover a wide range of reflectance values. This has been achieved for an angle of incidence of 59.7º and a thickness of the gold layer of 49nm.

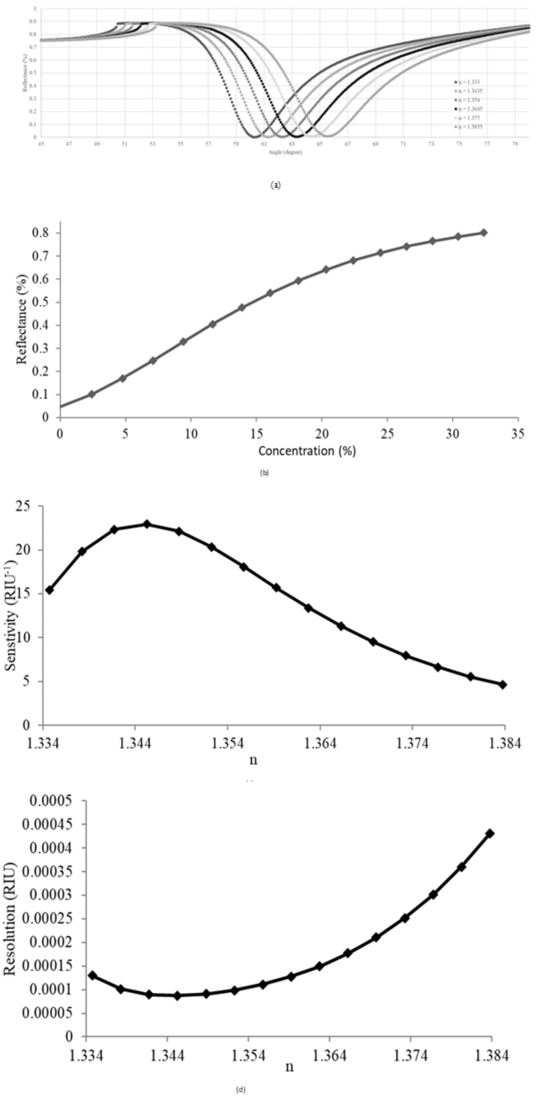

Fig. 2. (a) Reflectance curves as a function of the angle of incidence of the light in the prism for solutions with a low refractive index (low sucrose concentration), (b) Reflectance vs. % sucrose concentration in the case of the sensor designed for solutions with a low refractive index (low sucrose concentration), (c) Sensor sensitivity for solutions with a low refractive index (low sucrose concentration), (d) Sensor resolution for solutions with a low refractive index (low sucrose concentration).

Figure 2b represents the reflectance vs. sucrose concentration. We have fitted the data [21] of the refractive indexes and the concentration and the polynomial obtained is % concentration = $2793n^3 - 12791n^2 + 19906n - 10422$. This polynomial adjustment allows the concentration to be expressed based on the refractive index. Figure 2b shows the reflectance as a function of the concentration, so that, finally, we are able to obtain the concentration from the reflectance given by the sensor.

To calculate the sensitivity (Fig. 2c), which is the change in reflectance per unit of change of refractive index, we have to take into account that the reflectance is not linear with the refractive index.

As in this sensor the relation between reflectance and refraction index is not linear, we can apply the definition of sensitivity between each pair of values [25] of Figure 2b.

In Figure 2c, we can check that at about n = 1.34, the sensitivity is maximum (around 23RIU^{-1}) and at about n = 1.38 minimum (around 5RIU^{-1}) where we have assumed the resolution of the photodetector to be 0.2% [26] (assumed for all the sensors in this research).

Figure 2d shows the resolution of the sensor in the range of low sucrose concentration. It varies between 4.3x10^{-4} and 8.7x10^{-5}RIU, i.e., we are able to detect to the fourth or fifth decimal place.

2 Sensor for medium sucrose concentrations

This sensor allows us to measure sucrose concentrations from 30-35BRIX to 58-60BRIX (refractive index from 1.3820 to 1.4414). The optimal design has been achieved for an angle of incidence of 64.4º and an Au layer of 48nm thick. Figure 3a shows the reflectance curves.

Figure 3b shows the reflectance vs. sucrose concentration for solutions with a medium refractive index (medium sucrose concentration). The sucrose concentration of the solution can be obtained from the reflectance value.

Figure 3c shows the sensitivity vs. average refractive index of the sucrose solutions.

The maximum sensitivity is around 21RIU^{-1} for n = 1.39 and the minimum is around 4RIU^{-1} for n = 1.44.

The range of resolution in the medium range of refractive indexes is between 5.1x10^{-4} and 9.5x10^{-5}RIU, as shown in Figure 3d.

3 Sensor for high sucrose concentrations

In the case of solutions with high sucrose concentrations, we have designed the sensor so that it can measure sucrose concentrations from 1.4345 to 1.4940, which corresponds to 56-58BRIX to 80BRIX approximately. The thickness of the Au layer is 45.8nm and the incidence angle is 70.5º. This sensor has been optimized so that in the mentioned BRIX range it has the maximum sensitivity and resolution possible. The reflectance curves are shown in Figure 4a.

Figure 4b shows the reflectance vs. sucrose concentration for solutions with a high sucrose concentration. In a similar way to the previous cases, by means of this curve the sucrose concentration of the solution can be obtained from the measurement of the reflectance recorded by the detector.

Figure 4c shows the sensitivity vs. the average refractive index of the sucrose solutions. The maximum and minimum of sensitivity are 20 and 4.6RIU^{-1}.

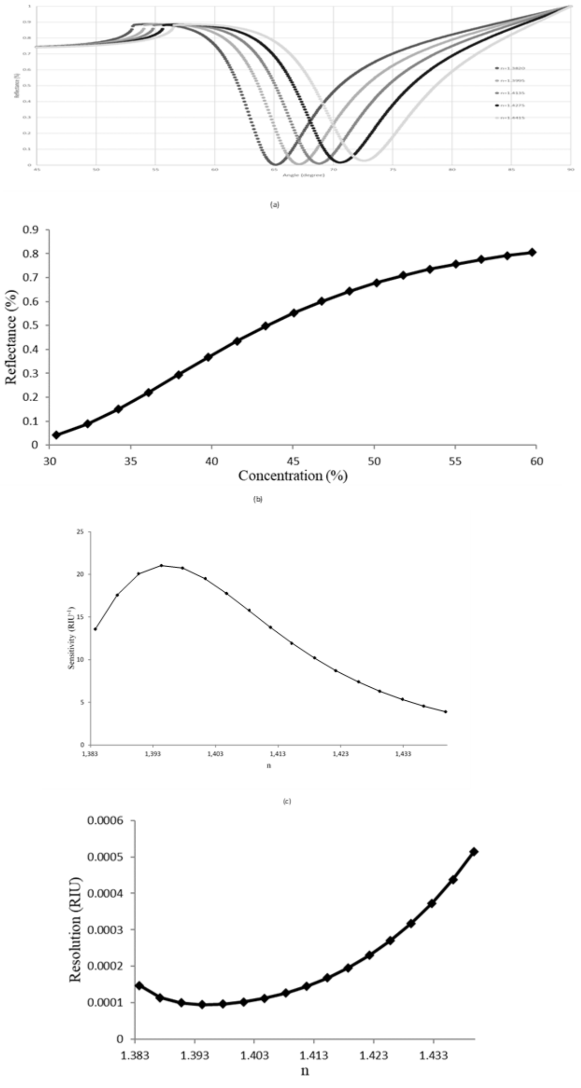

Fig. 3. (a) Reflectance curves as a function of the angle of incidence of the light in the prism for solutions with a medium refractive index (medium sucrose concentration). (b) Reflectance vs. % sucrose concentration in the case of the sensor designed for solutions with a medium refractive index (medium sucrose concentration). (c) Sensor sensitivity for solutions with a medium refractive index (medium sucrose concentration). (d) Sensor resolution for solutions with a medium refractive index (medium sucrose concentration).

The maximum and minimum of sensitivity are 20 and 4.6RIU^{-1} as can be checked in Figure 4c.

The sensor resolution is shown in Figure 4d. The extreme values of the resolution are 4.3x10^{-4} and 1x10^{-4}RIU (more resolution in the lower refractive indexes of the range).

Finally, it should be noted that these sensors have been designed so that there is a small area of refractive indexes in which the end of one sensor and the beginning of the other overlap, so that if it is decided to install the three together they are able to operate without any type of problem (and problems are also avoided in the limits of the intervals), and in the area where they overlap they would be programmed so that the sensor with the best resolution would automatically act.

We can now compare our sensor with other sensors proposed in the literature. Thus, in [15] a sensor based on optical fiber using graphene oxide encapsulated Au nanoparticles for sucrose sensing is proposed. The authors of this paper work with sucrose solutions with refractive indices in the range of 1.3395 to 1.3790. This range corresponds to our sensor designed for low refractive indexes. This sensor [15] has a sensitivity of 2.449ΔA/RIU (ΔA indicates the change in absorbance) and a resolution of 8.7x10^{-5}RIU. In the low refractive index range, our sensor has a maximum sensitivity (23RIU^{-1}) greater than sensor [15] and a similar resolution (8.7x10^{-5}RIU). Moreover, our three-in-one sensor covers a larger measuring range of sucrose concentration. The sensor we propose also has the advantage that its manufacturing process is simpler than in the case of sensors that incorporate nanoparticles.

Our sensor is based on intensity interrogation. It is not necessary to rotate the prism during the measurement process Intensity interrogation is perhaps the simplest technique and involves a fixed input wavelength whose reflected intensity varies according to the interaction between the surface plasmon wave and the analyte [5]. Therefore, our sensor allows us to instantly measure the concentration of the sucrose, unlike those sensors which are based on an angular interrogation, in which it is necessary to rotate the prism [16]. It also has the advantage that it does not require the use of a spectrometer, such as sensors based on spectral interrogation [8], [10], [15], [17], [18], [25], [27].

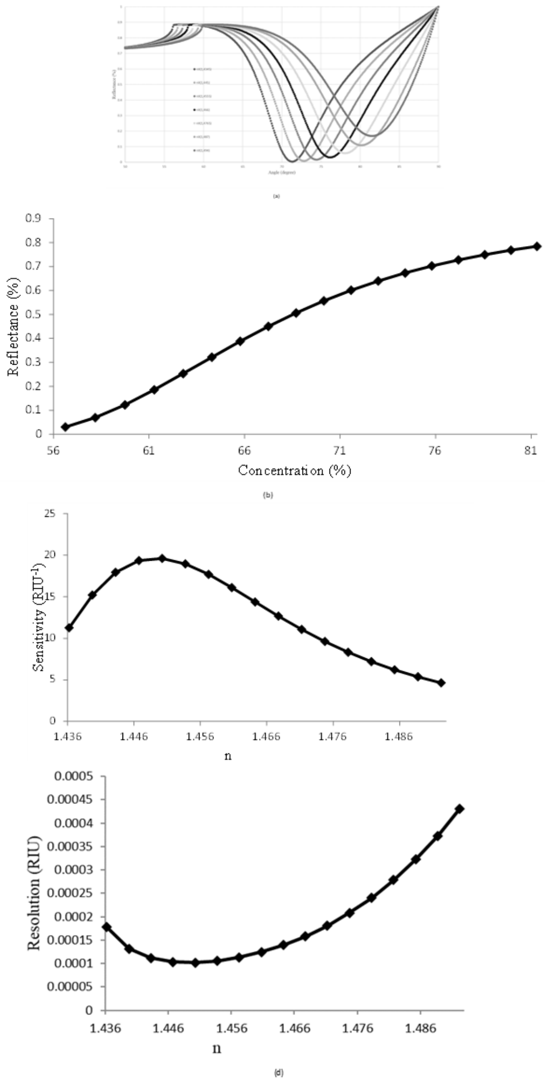

Fig. 4. (a) Reflectance curves as a function of the angle of incidence of the light in the prism for solutions with a high refractive index (high concentration of sucrose). (b) Reflectance vs % sucrose concentration in the case of the sensor designed for solutions with a high refractive index (high sucrose concentration). (c) Sensor sensitivity for solutions with a high refractive index (high sucrose concentration). (d) Sensor resolution for solutions with high refractive index (high sucrose concentration).

Compared with the non-plasmonic sensor, the sensitivity in [4] is at least one order of magnitude lower than that of a plasmonic-based refractive index sensor, therefore, our sensor has better sensitivity. Moreover, our sensor has a higher sensitivity than the microwave sensor based on a ring resonator [15].

In all the studies revised in this paper, the sensors measure small samples prepared for the experimentation or are extracted from a solution tank and in the case of chemical sensor, they waste some chemical products to produce reactions with sucrose, however, our sensor does not waste anything.

Compared to [1] (34Brix), [2] (50Brix), [3] (50Brix), [4] (63Brix), [7] (50Brix), [8] (60Brix), [10] (33Brix), [13] (12Brix), [14] (12Brix), [16] (45Brix), [17] (24Brix), [28] (50Brix), our sensor measures a wider range of sucrose concentration because our sensor is able to measure concentrations from 0-78ºBRIX.

Not all chemical sensors are able to give an immediate response to the sucrose concentration in a solution, there is a time interval needed to produce chemical reactions with the chemical compound used and, therefore, the sensor takes 127s to measure [12].

Unlike other sensors, the sensor we present in this paper is capable of measuring in real time. So, for example, the [11] only measures 30 samples per hour, [29] takes around 7-12min, [30] with the modified electrodes takes 15s and the lifetime is 57 days, [31] has a readout sensor of 8s and [6] of 30s, [32] 1min, and [33] between 1-2min.

CONCLUION

Our sensor is designed in such away that the measurements at the end of the range of one (refractive index/concentration) and the beginning of the next overlap, so we are able to assure the correct measurement of all the concentrations.

This sensor is ideal for applications where a big sensitivity and resolution is necessary.

It is capable of continually measuring concentrations for refractive index from 1.3333 up to 1.4940, i.e. from 0 up to 80Brix. Moreover, the sample does not have to be extracted from the tank where the solution is, we can measure in any part of the tank and know the concentration in every place, even if the concentration is not homogeneous.

The overall system can be equipped with a visual and audible alarm which can be automatically regulate, the pouring of liquid or sucrose to obtain the correct concentration at every moment.

Another specification is that the gauge can be controlled by a PC capable of storing the data in the memory, either printing them or sending them in real time by internet.

Our sensor could easily be implemented in electronic tongues (intelligent tongues) due to its small size and used to distinguish different levels of sweetness with considerable high sensitivity and resolution.

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