Design and implementation of an experimental Raman spectrometer

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Non-invasive medical diagnosis has become popular due to the possibility of detecting illnesses in vivo and in real time this technique, often referred to as "optical biopsy" it comprises several optical techniques such as thermography, diffuse reflectance spectroscopy, optical coherence tomography and Raman spectroscopy among others. Particularly Raman spectroscopy is an optical technique based on the inelastic scattering of light that can detect disease markers, this technique has been successfully used to detect several types of diseases, however the high price of a Raman spectrometer makes it difficult for the medical community to adopt its use as a common diagnostic procedure. In this work, an experimental Raman spectrometer was designed and fabricated from low-cost readily available components. The system was characterized and the Raman spectra obtained was compared to commercial systems. Results show that it is possible to fabricate an experimental Raman system with the desired optical configuration and with some improvements in the selected commercial components, it could be useful for non-invasive medical diagnosis.

Keywords: Raman spectroscopy; electronic instrumentation; laser technology.

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1. Introduction

Raman Spectroscopy is a technique based on an optical phenomenon first reported by C. V. Raman [1], in which a beam of light inciding on a sample generates inelastic collisions between the photons of the incident beam and the molecules of the sample, producing scattering with a frequency-shift due to the gain or loss in energy by the inelastic collisions. These frequency-shifts are known as Raman shifts and give detailed information about molecules that are Raman active [2-5]. Every Raman active molecule has a characteristic Raman spectrum which is unique to that molecule and can be considered a fingerprint for that molecule. This technique has a wide range of applications in different fields. For instance, in the case of medical diagnosis the technique has several advantages over the traditional biopsy since it provides results in real time, it makes sample preparation unnecessary and it is nondestructive [6]. Although a Raman spectrometer can be used for medical diagnosis depending on its sensitivity, specificity and low cost [7,8], currently these devices are not commonly used by the medical community mainly because their extremely high prices and their size, which is too large to carry it around a hospital. In this work, a methodology of design for an experimental Raman spectrometer is proposed and then validated following an implementation with commercial components.

2. Methods

The design presented in this work is based on previously proposed designs [9,10], that consists on a light source, a waveguide for excitation and detection, a detector and a PC for data processing. The major opto-electronical components of the proposed Raman spectrometer were from Ocean Optics:

the calibration source, the light source and the optic probes. Also, some optical-mechanical components used were from Thorlabs: the light source and its driver, a notch filter to reject the pump wavelength and two micro-positioners for fixing this optical filter. For the data acquisition, the software Ocean Optics Spectra Suite was used and the data processing was performed using the MatlabTM software (The MathWorks Inc., Natick, Massachusetts, USA). The light source used was a single mode fiber pigtailed NIR diode laser Thorlabs LP785SF-100 with a pump wavelength of 785 nm and optical power regulable from 0-100 mW, its principle of operation is ruled by the Eq. (1) and for the optical-electrical characterization of the diode laser, an optical power meter Newport was used.

$$P(I) = \begin{cases} 0.5I - 40, & \text{if } 80 \leq I \leq 310. \\ 0, & \text{if } 0 \leq I \leq 80. \end{cases} \quad (1)$$

where I is the current measured and P is the optical power obtained. This principle of operation is shown in the Fig. 1.
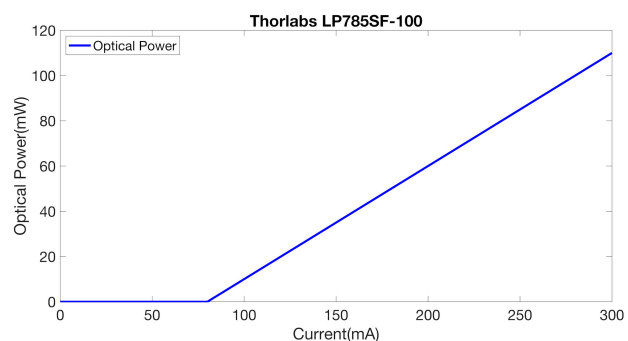

FIGURE 1. Spectral range of the spectrometer Ocean Optics $USB4000 - VIS - NIR$.

TABLE I. Commercial spectrometer configuration.

Specifications	Value
Wavelength Range	693 nm
Slit	25 um
FWHM	1.42 nm
Grating	600 gr/mm
Pixel Resolution	7.5
Detector Elements	3648
Blaze	500 nm
Input Focal Distance	42 mm
Output Focal Distance	68 mm
SNR	300:1

For the system waveguide, the optical fiber pigtailed to the diode laser was connected via a matching sleeve to a multimode optical probe $R200-7-VIS-NIR$, used as waveguide for the light source excitation and for the detection of the resulting back-scattered light.

A critical point on the Raman spectrometers is the filtering of the pump wavelength by using a notch filter with the same wavelength of the pump wavelength. In this work a notch filter $NF785SF-33$ was fixed between two micropositioners $PAF-SMA-5-B$ coupled to the optical probe used for the backscattered detection. Finally, for the detection of the Raman signal, an optical fiber connected to the micropositioner was connected to a general-purpose commercial spectrometer $USB4000-VIS-NIR$ (Table I).

The main parameters needed to customize a commercial spectrometer as a Raman spectrometer are: the slit, the grating, the optical resolution and the spectral range. The slit is defined as the input aperture of the spectrometer. In this part, the incident beam from of light incoming from the optical probe is transmitted inside the spectrometer divergently to a collimator and subsequently diffracted and focused onto a CCD for detection. From the slit diameter and the pixel resolution, it is possible to derive the slit diffractive angle of the spectrometer by using the Young equation, resulting in an angle of $13.27°$. The grating is an optical periodic array of diffractive elements impressed on a refractive surface, whose main purpose is to modify the magnitude or phase of a beam of light. The capacity of the grating to diffract a beam of light is ruled by the grating Eq. (2):

$$\sin\theta_i + \sin\theta_d = \frac{m\lambda}{d} \tag{2}$$

Where λ is the wavelength of the incident beam of light, m is the order of diffraction. In this work, the selected spectrometer has a Blazed Grating, defined as a concentration of a limited zone designed to obtain a mayor efficiency of a specific wavelength by the Eq. (3):

$$m\lambda = 2d\sin\theta_B \tag{3}$$

Where θ_B is the blaze angle and is the angle formed between the face of the groove and the grating plane. The blaze angle of the spectrometer was fixed at $13.65°$ and configured to have a maximum efficiency at 500 nm with a groove period of 1.66 um. For the optical resolution or Full Width at Half Maximum ($FWHM$), defined as the capacity of the spectrometer for differentiating two bands, it is needed that the parameters of the slit and the grating were efficiently configured because a) greater the groove frequency of the grating, greater the optical resolution, but resulting in a lesser spectral range. To determine the optical resolution of the spectrometer, first it is necessary to derive the dispersion by the wavelength range and the number of detectors of the spectrometer following the Eq. (4):

$$D = \frac{R}{N_D} \tag{4}$$

Where D is the dispersion, R the optical range and ND is the number of detectors. With the dispersion it is possible to calculate the optical resolution with the Eq. (5):

$$O_R = DP_R \tag{5}$$

Where O_R is the optical resolution and PR is the pixel resolution. Because the grating and the slit were fixed, the resulting values for this work were a dispersion of 0.189 nm/pixel and an optical resolution of 1.42 nm. Finally, the spectral resolution, defined as the separation between two spectral bands in function of the incident light wavelength is ruled by the Eq. (6):

$$\Delta_{1,2} = \lambda_0 - O_R \tag{6}$$

Where Δ_1 and Δ_2 are the differences between the incident light beam wavelength λ_0 and the optical resolution.

3. Results

Before the Raman detection, a wavelength detection characterization of the spectrometer was performed in order to obtain the detection percentage of the wavelength selected as a light source. For this task, a white-light tungsten lamp Ocean
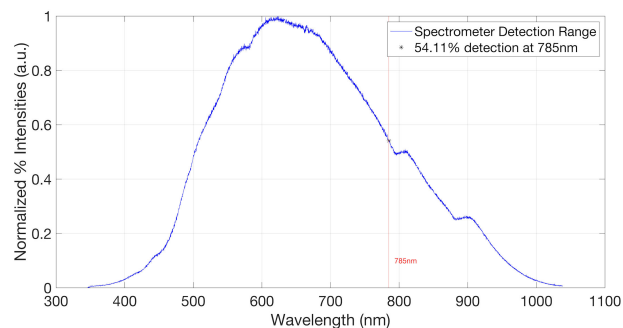

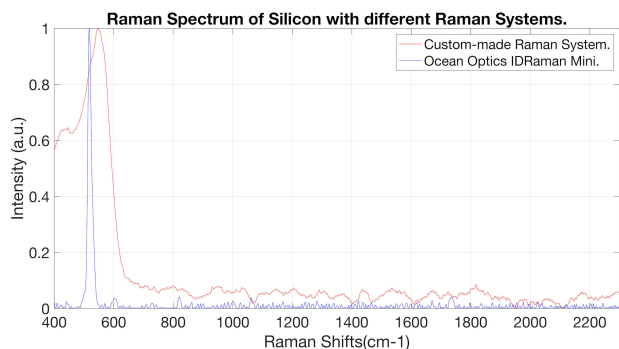FIGURE 2. Spectral range of the spectrometer Ocean Optics $USB4000-VIS-NIR$.

FIGURE 3. Mean Raman spectra of Si between the experimental Raman spectrometer and the commercial system.

Optics $LS-1$ was connected directly to the spectrometer and it could be seen that at 785 nm the percentage of detection of the spectrometer was of 54.11 (Fig. 2).

Following the proposed methodology in the Sec. 2, an optical resolution of 23 cm^{-1} was obtained, therefore, an effective characterization of the system was performed on samples with characteristic Raman bands like the Si, which has typical Raman bands at 300 cm^{-1} and 521 cm^{-1} previously reported and available in the web [11] and was used as reference spectrum in this work. The sample was put under the probe and the integration time was set to 10 s. Five Raman spectra of the Si were obtained from the experimental Raman system and five spectra from an Ocean Optics IDRaman Mini is shown in the Fig. 3, where it can be seen the considerable difference in the width between the spectrums (Fig. 4). With this issue, the possibility of quantitative analysis by comparing the resulted spectra with some
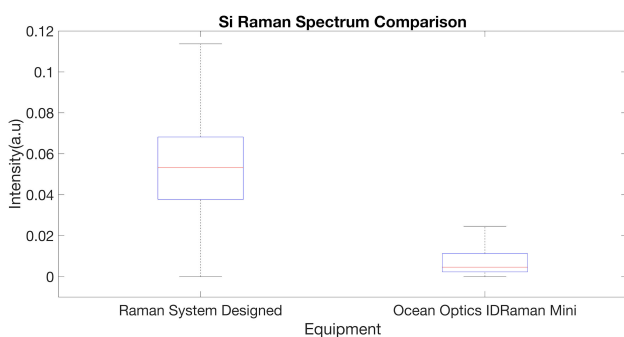

FIGURE 4. Mean intensities of the 521 cm^{-1} band corresponding to Si in both systems.

spectra from a data base is not possible, the possibility of a quantitative analysis, by comparing the resulting pectra with some spectra from a data base is no possible. insstead, it is only possible to perform a qualitative analysis of the spectra by identifying bands.

The configuration of the Ocean Optics IDRaman Mini is the following: a laser diode of 90 mW as a light source, a spectral range of 400-2,300 cm^{-1} and a spectral resolution of 8 cm^{-1}. Compared with this device, the experimental Raman system implemented was considered as a low-resolution system capable of detect the Raman scatterings of the sample under test.

4. Conclusion

Raman spectroscopy is a well-recognized technique used for material characterization, illegal substances identification and recently, as a tool for medical diagnosis. Unfortunately, the prices of the Raman systems are thousands of dollars making them inaccessible for great part of the medical personal, also the largest dimensions of the fixed Raman systems are a drawback for its heavy use. Actually, there are several portable Raman systems, but again, the price and the performance are two problems.

In this work, a methodology of design for an experimental Raman spectrometer is presented and implemented, due to the lack of specificity in the components, the results show a spectrometer with low resolution but capable of the detecting the Raman bands of commonly used materials like silicon. It is from our great interest in a future work to build a multipurpose Raman spectrometer following the same methodology of design described in this work, but with different and more accurate components.

Acknowledgements

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