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Designing and building a 3D printed low cost modular Raman spectrometer

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ABSTRACT

Raman spectroscopy is broadly used in both industrial and academic communities, including nanotechnology, biology, materials engineering, physics, pharmaceutical industry and environment protection monitoring. Most of these devices are worth thousands of dollars and usually require a PhD-level assistant to operate them, hence they are usually not available for college and undergraduate students. The present paper describes an affordable, easy-to-use, Raman spectrometer that can be built with commercially available electronics and optics and 3D printing. A Crossed Czerny Turner spectrometer optical configuration has been built with a linear CCD module driven by a Nucleo F401RE and Raspberry Pi. Raman spectra of Rhodamine B liquid was obtained. We propose that this system could make it easier for universities to build and use an affordable, effective, easy-to-use, Raman spectrometer for educational purposes. Introducing this technology to students with an affordable, easy-to-use, device as early as possible would help universities and colleges to draw attention to studying science. Further research into the development of affordable and effective Raman spectrometers has the potential to pave the way for broadening the application of this technology in more innovative ways in the daily lives of future residents of smart cities.

Keywords: Raman spectroscopy; Raman spectrometer; 3D printing; experimental innovation.

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INTRODUCTION

Raman spectroscopy is an optical analytical technique that allows investigation of the symmetric molecular vibrations of a material by measuring the frequency and intensity of scattered light. This molecular level information represents the fingerprints of the molecular bonds in the sample under investigation. The main reason for this project was to build an affordable, modular, easy-to-build, and effective Raman spectrometer for laboratory and educational purposes.

Nowadays the increasing availability of low-cost optics and consumer electronics make it possible to build an affordable and easy-to-use Raman spectrometer. Nevertheless, in the last few years, little attention has been paid to building one.

Several authors reported low-cost custom-made Raman spectroscopy devices (Kiselev, Schie, Aškrabić & Krafft, Popp 2016; DeGraff, Hennip, Jones, Salter & Schaertel 2002; Fitzwater, Thomasson, Glinski 1995; Galloway, Ciiolkowski, Dallinger 1992; Sanford & Mantooth 2001; Comstock, Gray 1999; Mohr, Spencer, Hippler 2010; Gnyba, Smulko, Kwiatkowski & Wierzba 2011). More recently, an effective and affordable Raman spectrometer for under $5,000 was built (Mohr, Spencer & Hippler 2010).

In this paper, the authors describe an affordable, adequate, Raman spectrometer device in detail and discuss the reasons for the methods chosen to build a low-cost one. 3D printing technology was chosen to build the mechanical structures of the Raman spectrometer; for image processing, single-board computer (Raspberry Pi), CCD detector and consumer electronics (Nucleo F401RE) were used; the excitation source is a 532nm green light laser. The design allows for

2413-9505 /

flexibility and further changes in the system, and collects the Raman spectra of the sample under investigation after several exposures.

BACKGROUND

Raman spectroscopy devices use a monochromatic light as an excitation source which is shined upon the matter under investigation. This results in two distinctive types of scattered light. One of these is called Rayleigh scattering and it is known as elastic scattering. In this type of scattering, there is no energy transfer between the incident light and the molecules, or atoms, of the matter. Thus, the scattered photon will have the same energy as the incident light (Gnyba, Smulko, Kwiatkowski & Wierzba 2011). The second type is called Raman scattering. When light scatters from a matter, a small percentage of the scattered light has a frequency that is different from the frequency of the incident light (Amer 2010). This is an inelastic scattering of photons on vibrational modes of molecular bonds (Long, D. A. 2002; Smith, E. & Dent, G. 2005).

Raman effect occurs when a photon is scattered with a change in its energy due to an interaction with the vibrational modes in the sample, hence this energy change is related to the vibrational properties of the sample (Jiménez-Sandoval, S. 2000). This information can provide fundamental molecular information on vibrational frequencies and molecular shapes (DeGraff, Hennip, Jones, Salter & Schaertel 2002).

Raman spectroscopy is named after its inventor, Chandrasekhara Venkata Raman, an Indian Professor of Physics at the University of Calcutta who, with his colleague K. S. Krishnan, was the first to realise this combinatorial scattering of light and published a paper on this technique (Raman & Krishnan 1922). Independently of their colleagues in India, a week earlier, two Russian professors, G. S. Landsberg and L. I. Mandelstam at Moscow State University, reported the same phenomenon (Landsberg & Mandelstam 1928).

BASIC THEORY

Raman scattering is a quantum phenomenon; however, it can be described with classical electromagnetic theory. The following expressions are taken from a book written by Maher S. Amer et al. (Amer 2010). A monochromatic light wave with an oscillating electric field in the x-direction interacts with a molecule. The amplitude of the electric field (E_X) can be expressed as:

$$E_x = E_x^0 \cos(2\pi\, v_0\, t) \qquad (1)$$

where E_x^0 is the maximum amplitude of the electric field, and v_0 is the frequency of the incident light. The normal vibration mode of a diatomic molecule with a vibrational frequency of v_v, can be expressed as:

$$q_v = q_v^0 \cos(2\pi\, v_v\, t) \qquad (1.1)$$

Once the incident light interacts with the molecule, the light will polarise the electrons of the molecule. This, as a result, induces a dipole moment (μ):

$$\mu = \alpha E \qquad (1.2)$$

Where α is the polarizability tensor. Because of the oscillation, light is emitted at three different frequencies. These three different frequencies can be expressed as a Taylor series in (q_v) as:

$$\alpha = \alpha^o + \left(\frac{\partial\alpha}{\partial q_v}\right) q_v \qquad (1.3)$$

Substituting 1 and 1.2, the dipole moment can be expressed as:

$$\mu = E_x^o \alpha^o \cos(2\pi\, v_o t) + E_x^o \left((\frac{\partial\alpha}{\partial q_v})_0 \right) q_v^o \{\cos[2\pi(v_o - v_v)t] + \cos[2\pi(v_o + v_v)t]\} \qquad (1.4)$$

The three frequencies are $v_o, (v_o - v_v), (v_o + v_v)$. One of these frequencies is the same as the frequency of the incident light v_o , which means that there is no energy exchange with the material: this is the Rayleigh scattering. The two other modes experience a frequency shift from molecular vibrations $(v_o \pm v_v)$, hence a change in energy occurs equal to the energy of the molecular vibration: this is the Raman scattering. The light that is scattered with lower energy $(v_o - v_v)$ is called Stokes scattering, and the other scattering with higher energy $(v_o + v_v)$ is called anti-Stokes scattering (Amer 2010). The Raman scattering is generally weak and is surpassed by fluorescence.

DESIGN CONSIDERATIONS AND INSTRUMENTATION

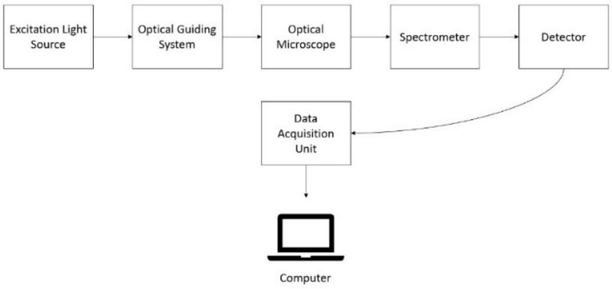

Fig. 1: Instrumentation of a typical dispersive Raman spectrometer

Raman instrumentation is a very dynamic and complicated field. Valuable information on modern Raman spectroscopy can be found in different publications (Smith & Dent 2005; Delhaye, Barbillat, Aubard, Bridoux and Silva 1996; Barbillat, Turrell, Corset 1996; Adar, Lewis, Edwards, Dekker 2001).

The instrumentation of a typical dispersive Raman spectrometer is given in Fig. 1. The excitation light source is a laser; usually either a monochromatic laser or a tuneable dye laser. Two of the most important parameters of a laser are its wavelength and its power. The laser excitation wavelength is relevant to what one can examine with the device at hand. Even though the Raman signature is independent of the excitation wavelength, different excitation wavelengths come with advantages and disadvantages depending on the sample under investigation (Zhou, P., B&W Tek Inc.). For instance, with long wavelengths, the excitation efficiency is lower and heat absorption is high, which may cause damage in a biological specimen.

For the purpose of this project, three of the most widely used excitation wavelengths were considered: 532nm, 785nm, and 1064nm. Close to the ultraviolet range, the excitation efficiency is high and heat absorption is low, but this comes with disadvantages. A wavelength closer to the ultraviolet range can cause electronic excitation within the molecules, thus changing the chemical bonds (Kiselev, Schie, Aškrabić & Krafft, Popp 2016). Secondly, fluorescence, which is a well-known nemesis of the Raman effect, is likely to occur. The longer the excitation wavelength, the more light is absorbed by the sample and the more it is heated. There are methods to minimise this: rotating the sample and lowering the laser power are two of them, but they come with costs, that is adding complexity to the design as well as increasing the measurement time and worsening the signal-to-noise ratio (Zhou, P., B&W Tek Inc.). But, one of the significant advantages of using a near infrared laser light is the reduction of fluorescence. However, scattering intensity decreases with the fourth power of the excitation frequency according to the Rayleigh law. It is well known that only one Raman scattered photon occurs for every 10^6 photons. Therefore, even low fluorescence can cause problems and block the Raman signal from reaching the detector (Tuschel 2016). It's also important to know that Raman scattering intensity is proportional to the laser power and depends on the fourth power of the excitation frequency. Thus, shorter wavelength provides better Raman signal (Kiselev, Schie, Aškrabić & Krafft, Popp 2016).

532nm laser excitation provides good sensitivity which is one of the features needed for a low-cost Raman spectroscopy design. Raman signals excited by a 532nm laser are distributed in the visible range, where the response is best for most of the silicon-based CCD chips and has the benefit of covering the full range from 65cm^{-1} to 4000cm^{-1}. For these reasons, we have chosen a 150mW 532nm wavelength green laser module with thermoelectric cooling. The Raman scattering at 532nm is 16 times better than at 1064nm, and scan time is much longer at higher wavelengths compared to 532nm (Zhou, P., B&W Tek Inc.). A 532nm laser line filter has been used and placed in front of the 3D-printed laser mount. This makes sure that only 532nm wavelength light enters the system from the laser source. These filters only transmit the relevant wavelength and block the other frequencies that may occur due to instability in a laser source. (https://www.semrock.com/filter-types-for-raman-spectroscopy-applications.aspx).

Optical microscopes are used to focus the laser light on the sample to collect the backscattered light and send it to the spectrometer. A 100X microscope objective is used for this purpose.

OPTICAL CONFIGURATION

The next important part of a Raman spectrometer is the spectrometer. Rayleigh scattered light should be rejected with a notch filter, or an edge filter or a monochromator. Notch filters are quite expensive for a low-cost Raman spectrometer project. Edge filters are relatively cheap in comparison and have the advantage of allowing observation of Raman lines closer to the Rayleigh line. In this case, one might need two edge filters; one with a short-band pass region and the other with a long-band pass in order to take two different regions of the spectrum. Mark Johnson et al. (https://hackaday.io/project/1279-ramanpi-raman-spectrometer) has built a cheap, open source Raman spectrometer using a motorised system with two different edge filters on, which takes several exposures. The images are stacked afterwards for evaluation purposes. His design is truly clever, and ingenious. The reader is encouraged to check his design.

With inspiration from his design, we carried out several exposures alternating between two edge filters. The results from each filter were evaluated and images were stacked together. Edge filters with relevant wavenumbers were not available at a cheap price, hence edge filters with wavelength numbers close to 532nm have been purchased. A 550nm optical long-pass filter and a 522nm optical short-pass filter have been used.

The spectrometer consists of a group of gratings and mirrors that can be arranged in different ways. The function of a spectrometer is to receive the light and reflect it onto several optics in order to finally shine this reflection on the CCD image detector, which reads the spectral components and digitises the signal and finally sends it to a computer for displaying and analysing purposes. There are different configurations for a spectrometer. The first consideration for this project was the Fastie-Ebert configuration. Even though this configuration is an inexpensive-to-build, simple, and commonly used design, it has limitations in terms of providing good image quality (http://www.thespectroscopynet.eu/?Spectrometers:Monochromator:Fastie-Ebert_configuration).

The second configuration is the famous Czerny-Turner configuration. The Czerny-Turner instrument consists of an entrance slit, focusing mirror, collimating mirror, diffraction grating and finally a CCD detector. The light gets into the system through an entrance slit and reflects onto a collimating mirror. This collimating mirror reflects the light onto diffraction grating, and then is diffracted towards a focusing mirror. This second mirror focuses the beam from the diffraction grating onto a CCD linear array detector. The Czerny-Turner optical configuration is a commonly used design because of its compact nature, consisting of two concave mirrors and diffraction grating (https://www.laserfocusworld.com/articles/print/volume-49/issue-05/features/spectrometers--miniature-spectrometer-designs-open-new-applicati.html; http://bwtek.com/spectrometer-part-4-the-optical-bench/)

There are two subtypes of the Czerny-Turner Configuration: Uncrossed and Crossed. For this project we used Mark Johnson's spectrometer design for the Crossed Czerny-Turner configuration. His work is open source and, again, we encourage you to check his work on the hackaday website (https://hackaday.io/project/1279-ramanpi-raman-spectrometer).

After the optical configuration and the laser wavelength are determined, the next consideration is for CCD imaging and data processing.

We found Esben Rossel's open source CCD imaging design, which is another low-cost solution (https://tcd1304.wordpress.com/tcd1304-pcb/).

We have used his PCB design for CCD imaging configuration and his software for data processing.

Nucleo F401RE and Raspberry Pi are used for signal digitisation and image processing. Finally, Raspberry Pi is connected to an LCD screen where the results are displayed through a graphic.

Figure 2 shows our overall system design.

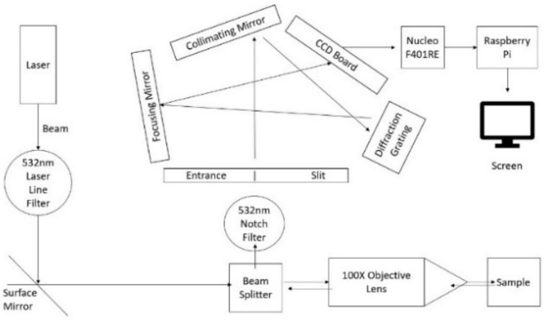

Fig. 2: Overall system design

After the decisions were made on these subjects, the design of the system was started. We have used OpenSCAD, a computer-aided design (CAD) software, for the drawings of each mechanical part of the system and Autodesk 3DS Max for illustrations.

CAD drawings were then converted into STL file format to be 3D printed with a MakerBot Replicator 2 3D printer. Fig. 3, Fig. 4 and Fig. 5 are illustrations of the system. These illustrations were made using Autodesk 3DS Max.

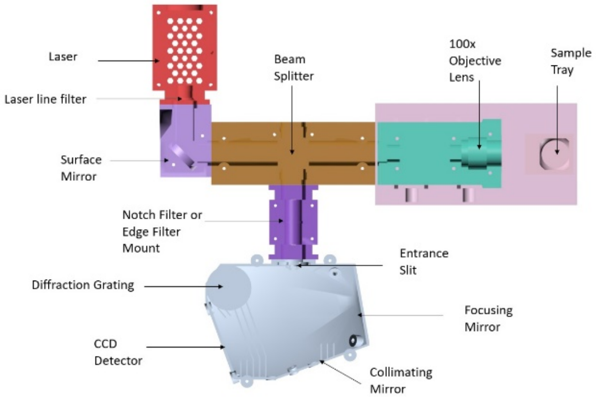

Fig. 3: Top view illustration of the Raman spectrometer system

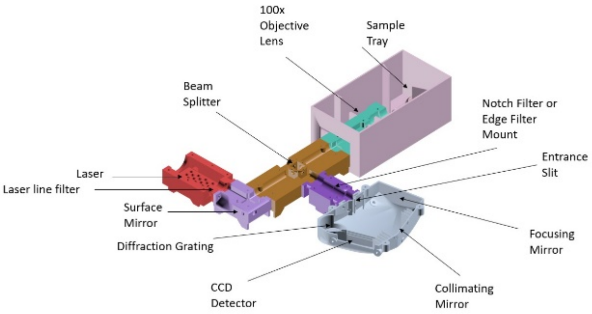

Fig. 4: Raman spectrometer system with each part labelled and assembled

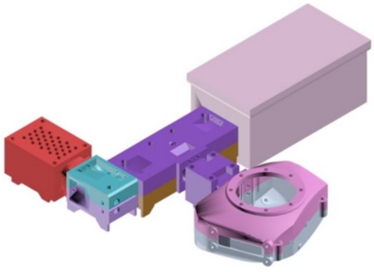

Fig. 5: Raman system with covers closed

Fig.4 and Fig.5 show what the system looks like when the 3D-printed parts are attached to each other and also demonstrate where each optical component is. Parts are assembled using M4x16mm screws.

DATA COLLECTION

TCD1304DG was used for CCD imaging purposes. It is a sensitive linear image sensor with 3648-pixel number. An evaluation board is needed for both driving and reading the data from linear CCD. STM32 Nucleo F401RE MCU is sufficient for this role. F401RE board has a Cortex-M4 processor running at 84MHz. The power-in pin on TCD1304DG was connected to the F401RE's 3V pin. CCD output is analog, therefore, an ADC (Analog-to-Digital Converter) is a necessity. Nucleo F401RE has 12-bit ADC capable of 2.4 Msps and is suitable for our application. ICG (Integration Clear Gate), fM (master clock), SH (shift gate) and OS (output signal) must be connected to the Nucleo F401RE for data processing and collection. Finally, the ground connection to the Nucleo F401RE must be made on one of the ground pins. More information on the connections can be found on Esben Rossel's blog. Nucleo F401RE is connected to Raspberry Pi 3 with a USB cable. Once the programmed .bin file is copied into the Nucleo, the Nucleo F401RE should start blinking with a 2Hz frequency.

In addition, the required environment settings must be applied on the Raspberry Pi command line for the operating and usage of the GUI (Graphical User Interface). After these settings and connections are complete, CCD should be ready to collect data.

CCD outputs data when ICG and SH pulses match up. Our CCD was clocked at 500kHz which requires a minimum ICG period of 14776. The user must keep the ICG period to multiples of the SH-period to ensure the CCD timing requirements are met. If the ICG-period is too long, then the CCD will introduce a lot of dark current due to the long exposure from the laser light, thus, resulting in an increase in temperature. A TTL (Thermoelectric cooler) can be attached to the CCD with additional design changes in the system. Esben Rossel et.al 2017 designed a CCD with TTL (https://erossel.wordpress.com/page/3/)

A sufficient TTL would help to reduce the dark current by decreasing the temperature below 0^{0}C. Reader is encouraged to visit Esben Rossel's blog which is regularly updated.

Fig. 6 is a picture of the assembled Raman Spectrometer.

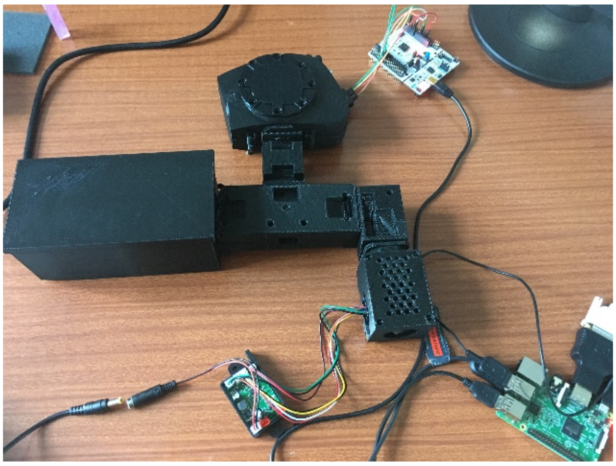

Fig. 6: Assembled 3D printed Raman spectrometer

TEST RESULTS

Our Raman spectroscopy device achieved good performance and provided adequate results. The Raman spectra obtained of liquid Rhodamine B ($C_{28}H_{31}ClN_2O_3$) is shown on Fig. 8. The CCD heat and laser induced fluorescence are the main sources of the background noise which can either be reduced with a thermoelectric cooler (TTL module), which can be incorporated into the design of the CCD, or by using a baseline of dark current and then subtracting it from the results. There is also interference signal coming from the vial tube, which contains Rhodamine B solution. Several exposures were taken and stacked together to catch the full Raman spectra of Rhodamine B in liquid form.

ADC-conditioning circuit was not built for the device, thus the spectrum is upside down in Fig.7. A simple ADC-conditioning circuit can be built to avoid the upside down-view. On Fig.7 an exposed pixel gives a low value and an unexposed pixel gives a high value in the Raman intensity axis. The Y-axis is turned upside-down on SigmaPlot software to evaluate the results of the spectrum.

Fig. 8 shows the Raman spectrum that is obtained after several exposures were taken with different edge filters. Data were stacked together when a sufficient amount was collected.

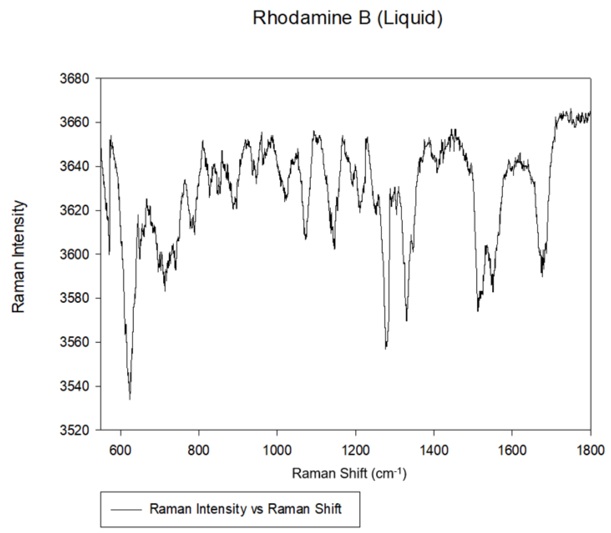

Fig. 7. Upside down Raman spectrum of Rhodamine B liquid

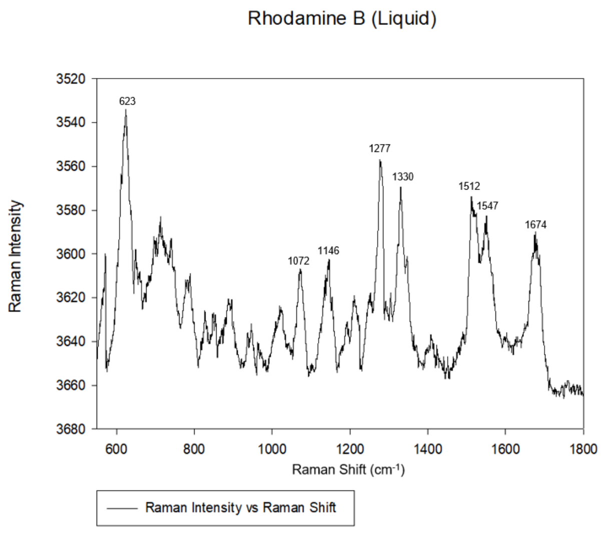

Fig. 8. Raman spectrum of Rhodamine B liquid

Experimental results have slightly different peak positions than literature values. The theoretical calculation found in the literature is based on an isolated molecule in the gas phase which may be the reason behind this (Prabhaharan, 2014). Our design is only suitable for liquid form, hence we investigated a liquid sample. A group of researchers have investigated the solid phase Rhodamine B powder (Zhang, J. T., Li, X. L., Sun, X. M., Li, Y.D. 2005). They also had different peak positions than the theoretical calculations and concluded that this might be due to theoretical calculations being based on an isolated molecule in the gas phase.

623cm^{-1} peak is related to the xanthene ring puckering mode (Yang et al 2013). Other peaks between 1000cm^{-1} and 1700cm^{-1} on the spectrum are related to C-H, C-C, and C=C bonds (Zhang, J. T., Li, X. L., Sun, X. M., Li, Y.D. 2005). Comparison between theoretical and experimental peak positions is given in Table. 1.

Tab. 1. Theoretical and experimental Rhodamine B peak positions

DFT (cm^{-1})	Liquid (cm^{-1})	Liquid (cm^{-1})
613	623	Xanthene ring puckering
1195	1146	C-C Bridge Bands
1278	1277	Aromatic C-H Bending
1355	1330	Aromatic C-C Bending
1504	1512	Aromatic C-H Bending
1525	1547	Aromatic C-H Bending
1644	1674	Aromatic C-C Bending & C=C stretching

LIST OF MATERIALS AND EQUIPMENT

The following optics, electronics and machinery were used for building the Raman spectrometer. The prices are roughly based on each relevant company's website before custom clearance, taxes and additional shipping costs etc.

Tab. 2. Equipment list

Component	Price ~ $
MakerBot Replicator 2	$3,000
150mW Focusing 532nm Green Laser module with TTL	$90
1.75mm 900gr MakerBot PLA	$40
STM32 F401RE Nucleo-64	$13
Raspberry Pi 3	$40
TCD1304DG CCD + PCB	$10
12.5 Cube Beam Splitter 532nm	$95
100µm x 3mm, Mounted, Air Slit	$111
532nm CWL 12.5mm Diameter Bandpass Filter	$125
525SP Optical Short-Pass Filter	$20
550LP Optical Long-Pass Filter	$12
50mm x 100mm FL Protected Aluminium Concave Mirror	$42.50
20mm x 80mm Focal Length Spherical Mirror	$37.50
1200 Grooves/mm 25mm Square VIS Holographic Grating	$140
Newport 10D20ER Mirror	$60
TOTAL COST	**$3,836**

Total cost may be cheaper if a cheaper 3D printer is used or if outsourced 3D printer services are used.

CONCLUSIONS

This paper outlines designing and building a low-cost, easy-to-use Raman spectrometer device. The presented device is convenient for analysis of liquid solutions. Several exposures must be taken and stacked together for a full spectrum. The device is not yet ready for professional analysis but can be a useful tool for undergraduates for experimental and educational purposes, to introduce students to one of the most important techniques in the spectroscopy literature.

We conclude that an affordable and easy-to-use Raman spectrometer can be built with low-cost equipment. The most expensive parts of the system are optics and 3D printer. An addition of a notch filter for better performance will increase the cost but can be purchased and added into the system. As aforementioned, one needs to take several measurements, provided with a reasonable exposure time, to obtain the Raman spectrum of the liquid sample under investigation. The key problem is reducing the noise caused by the laser, container, CCD, heat, and the walls of the 3D printed parts.

Raman spectroscopy, without a doubt, is a useful tool in research and many industrial fields. However, it is not yet commonly known and used by the public. An affordable Raman spectrometer has the potential to pave the way for many applications in daily life of our connected world. Raman spectrometer may become an affordable device for public to purchase; just like what we see happening with 3D printers these days.

Introducing Raman spectroscopy to students during their pre-college and/or undergraduate era has the potential to draw children's and students' attention to science and gain their interest. Educating undergraduate students in relevant science and engineering areas with an easy-to-use, low-cost Raman spectroscopy could help universities educating their students in physics laboratory lectures with this technology, without causing any damage or repair costs to professional equipment.

Paving the way in education has the potential to increase the possible applications of Raman spectroscopy in the future by educating students with this technology at early stage. This will raise visionary students for the future of the world.

Eventually, making Raman spectrometers cheaper and better is likely to lead to Raman spectrometers that are affordable and as effective and accurate as today's expensive versions.

This would lead to more applications and wide spread usage of Raman spectrometers, especially in the future smart cities, where many elements of the city will require monitoring and quality checks. The Raman spectrometer has the potential to be a part of smart city citizens' daily life.

This prototype may pave the way for further improvements at CERN IdeaSquare (http://ideasquare.web.cern.ch/) for a more robust 3D printed Raman Spectrometer. A 3D printed low-cost Raman spectrometer can be further improved and used at CERN IdeaSquare for innovative ideas and for demonstrations to students and hackers who are visiting IdeaSquare, who might use it for innovative ideas that they develop at IdeaSquare. This device may also help to educate the students who are visiting CERN IdeaSquare and draw their attention to molecular physics. A 3D printed low-cost Raman spectrometer also has potential to be used at workshops and laboratories at CERN IdeaSquare, with further improvements in the device for solid phase investigations and perhaps better imaging and data analysis, particularly at Light Laboratory.

There are not too many researches which make spectrometers both cheaper and more effective. Hence, these devices are not accessible to undergraduate students or experimental innovators who could benefit from using Raman spectrometers. A department like CERN IdeaSquare has the right potential for paving the way in this area, with scientists and innovators collaborating at IdeaSquare as part of a hub for experimental innovation and ideas for improvements on a low-cost, effective, 3D-printed Raman Spectrometer.

SAFETY

Laser safety glasses must be worn whilst building and operating the device.

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