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Photodiode-Based Spectrophotometer

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Abstract

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A spectrophotometer device supplies a light source at a stable frequency, and has a sensor that is capable of reading the light frequency after it has hit or been through a material. The device can identify the chemical elements, measure the concentration of it in a solution or identify impurities in it.

The goal of this thesis work was to learn the process of designing a spectrophotometer after studying the principles that regulate spectrometry and, in the end, measure its range. This thesis is a learning tool to better understand the concept, the studies and finally the device.

This thesis presents the process to design and test a portable spectrophotometer. The device is a proof-of-concept and will stay permanently at the stage of prototype. The measurements were done with a multimeter.

The results showed that the white LED is sufficient to the measurement of the of the concentration of the solution. The RGB LED did not produced results that are considered reliable enough but some of measurements showed that a different colour LED can enhance the sensitivity of the device at specific frequencies. The possibility of calibrating the device it with a camera could make a difference.

Keywords: Spectrum, Spectrometers, Spectroscopy.

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List of Abbreviations

Abs:	Absorption coefficient
ADC:	Analog Digital Conversion
FIR:	Far-Infrared Region
FT:	Fourier-transform
LED:	Light Emitting Diode
MIR:	Mid-Infrared Region (IR or MIR)
NIR:	Near-Infrared Region
Op-Amp:	Operational Amplifier
Raman:	Raman Spectroscopy
VIS:	Visible region in spectrum
UV:	Ultraviolet

1 Introduction

Spectroscopy is the branch of studies in science that investigates the interaction between matter and radiated energy. It is the theoretical approach to the balancing of matter in atomic and molecular levels. It investigates how electromagnetic waves interact with matter.

Spectrometry uses complex instruments with accuracy that cannot be replicated in school laboratory. The design of a spectrometer requires a team with several people to be accurate and efficient.

Nevertheless, a good result for simple tasks can be achieved by a simple device. The spectrophotometer design process requires a knowledge of operational amplifiers, LEDs, sensors, and measurement devices.

The thesis goes through the history of the science of spectroscopy. Different kinds of spectroscopy are analysed. The main goal of this work was to produce a device that could project a light at a specific frequency and could measure it after it has been through a substance. During the phases of production and testing, particular care was put on the LEDs and the light detector part of the device. Still all the different components of a spectrophotometer were studied and considered for future builds.

At the beginning of the project, the collection of data was relegated to an Arduino system of the new generation. It can be connected to a 5V battery pack and has Bluetooth® Low Energy and Wi-Fi connectivity options. The ADC is handled by a simple program to use with the Arduino Cloud system.

The collection of data was in the end left to the multimeter.

2 Spectroscopy Theory

To study spectrophotometry a student should have a clear understanding of spectroscopy.

The study of Spectroscopy investigates how electromagnetic waves interact with matter. It analyses the light and its spectrum, studies mechanisms like absorption and emission. [1.]

2.1 History of Spectroscopy

In 1666 Sir Isaac Newton, observing the diffraction of light by a prism, discovered that what was considered white light, is the combination of several colours. [2.]

Around the 18^{th} and 19^{th} century, the theories of a correlation between the light and elements came to form. In the 19^{th} century Fraunhofer and Wollaston in separate experiments observed dark lines placed in the same position on the spectrum of the Sun. It was the beginning of spectral analysis. Those lines were later attributed to specific elements that are present in the Sun's atmosphere. [3; 4.]

In the 1860 Bunsen and Gustav Kirchhoff are considered the first ones to perform a real quantitative spectroscopy and mainly discovered that different elements have a unique spectrum. They used spectroscopy as a tool for elemental analysis. [5.] [6.]

Helium was first discovered in the spectrum of the Sun in 1862 even before it was synthesized on earth and discovered on earth some 20 years after it was discovered in the Sun spectrum. [7.]

The idea of light being formed of electromagnetic waves was demonstrated by Maxwell in "A Dynamical Theory of the Electromagnetic Field" in 1865. [8.]

Spectroscopy has been very closely tied to scientific advancement regarding the relation between light and materials.

In the beginning of the 20th century Planck solved the black-body radiation problem. This event is considered the birth of quantum mechanics. The solution was based on spectroscopic observations of thermal radiation from black bodies. Quantum mechanics is still the fundament of all the understanding of how light and matter interact. [9.]

In 1905 Einstein concluded that each wave of frequency *f* is associated with a finite packet of photons with energy *hf* each, where *h* is Planck's constant.[10.]

In 1913 The Bohr model of the hydrogen atom explained the spectral lines and their location. [11.]

Indian physicist C. V. Raman in 1928 published the results on the studies of the inelastic scattering of photons by matter. Only more than 30 years later with the invention of the laser and 40 years the CCD will be possible to make the Raman spectroscopy to have feasible timing. The initial light source was a mercury lamp, the spectra was recorded on photographic plates. It took several hours to a few days to obtain a result. [12.]

2.2 Application of Spectroscopy

A full specific list of applications would be extremely lengthy. It is important to mention the fundamental applications. It can confirm the identity of a substance. It is possible to identify the components of a compound. It can determine if there is a contaminant and what is the percentage of it in a product.

A list of the fields where spectroscopy is a fundamental instrument:

- In chemistry is used to investigate the atomic structure of substances.
- In medicine to study the components in bodily functions.
- Astronomy is mostly based on the study of light to identify the fabric of the universe.

- Biology uses it for tissue investigation.

2.3 Light and Electromagnetic Spectrum

It is known that light is an electromagnetic wave. It is mainly characterised by its wavelength. In the visible spectrum the wavelength determines the colour, as shown in figure 1.

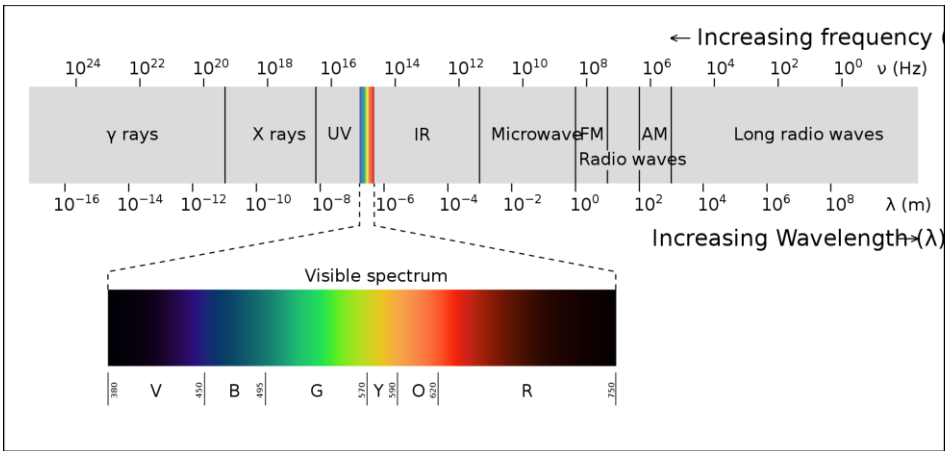

Figure 1. Electromagnetic Spectrum. Reprinted from Wikipedia [13.]

The wavelength (λ) is the distance between two crests in the electric and magnetic field oscillations as shown in figure 2.

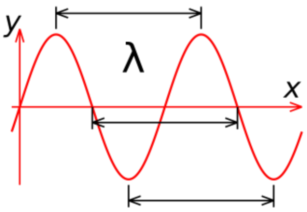

Figure 2. Wavelength. Reprinted from Wikipedia [14.]

Light carries energy. Energy and wavelength are related by a simple inverse relationship.

Energy per photon

$$E = hV = hc/\lambda \quad (1)$$

E= Energy
h= is the Planck constant
V=Frequency
c= speed of light
λ= wavelength of the light

Energy is equal to Planck's constant(*h*) times the frequency(*V*) or Planck's constant(*h*) times the speed of light(*c*) divided by the wavelength(λ) [15.]

The radiation of very short wavelengths will have very high energy, the radiation of very long wavelengths will have very low energy.

An important property of light is its Polarization. As shown in figure 3 It is mainly the direction of the electric field vector so polarization can be important because all the spectroscopic processes depend on polarization and most optical components in optical instruments used have some polarization dependence as well.

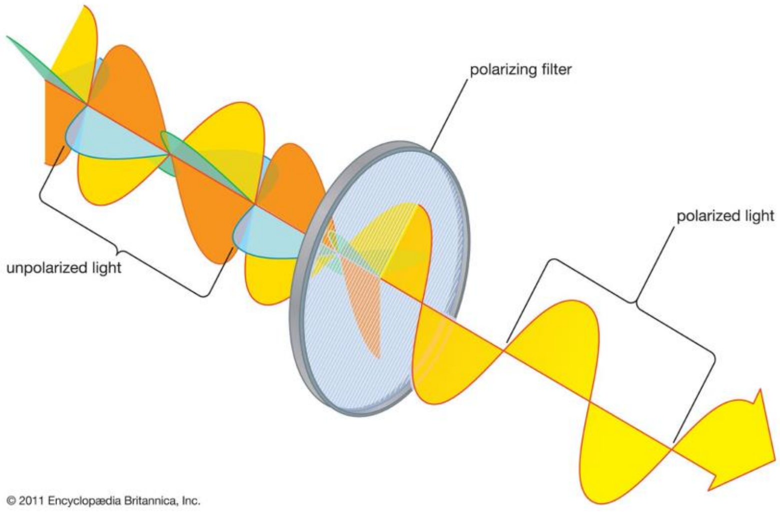

Figure 3. Polarization. Reprinted from Polarization [16.]

2.4 Spectroscopy

Once the spectroscopy was considered the science of light in the visible spectrum, modern spectroscopy extends through the whole electromagnetic spectrum. This thesis is strongly based on the behaviour of the photons in relation to the mass.

In figure 4 the absorption mechanism is visualized as a packet of photon that is absorbed by the mass puts the electrons in an atom in an elevated energy state. In an atom where the electron goes to a lower energy state there is an emission of photons. The amount of energy exchange is given by the structure of the photon and electrons.

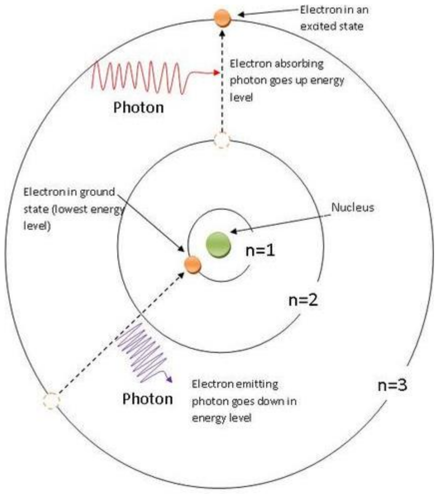

Figure 4 Emission and Absorption. Reprinted from: Principles of Structural Chemistry [17.]

Nowadays the mechanisms of particles as electrons, protons and ions are studied by spectroscopy. [18.]

2.5 Infrared Spectroscopy

The most used spectroscopic method is the infrared spectroscopy sometimes called vibrational. It is a valid method of investigation for solids, liquids, and gases. IR spectroscopy is based on the principle that the absorbtion of IR frequencies is correlated to a specific molecular structure and vibration. The instrument has a light emitter that works in the infrared section of the light spectrum. The IR spectrum is usually graphed by assigning a percentage of Transmittance or Admittance to the vertical axis and the frequency on the horizontal axis. When the frequency is measured in wavenumbers, the units is the reciprocal centimetre (cm^{-1}). If it is measured in wavelength the unit is the micrometre or micron(μm).
In the electromagnetic spectrum the infrared range is split into three sections: the near-, mid- and far- infrared according to their distance from the visible range.

The near-IR range goes from 4000 cm^{-1} to 14,000 cm^{-1}, or 2,500 nm to 700 nm, the mid-IR range from 670 cm^{-1} to 4000 cm^{-1}, or 15 µm to 2.5 µm. Last the far-IR 670 cm^{-1} to 10 cm^{-1}, or 1000 µm to 15 µm. These ranges are called regions and each of them exert a different behavior of the molecular bonds. The are different modes for the molecule to transition: translational, vibrational and rotational; in IR-spectroscopy mostly produce vibrational transitions. The different vibrational modes can be classified by their characteristics in the movement. When the bonds are subjected to a variation in the interatomic distance is called *stretching* vibrations and can be symmetric (marked with V_s) if they vary the distance at the same, asymmetric (marked with V_{as}) if they are not. When is the angle of the bond that is subjected to an alteration in degrees, these are called bending vibrations, four different vibrational modes are observed: scissoring (marked with δ) and rocking (marked with ρ), when there is an in-plane bending. Wagging (marked with ω) and twisting (marked with τ), when there is an out-plane bending. The different motions in vibrational modes are shown in figure 4. [19.]

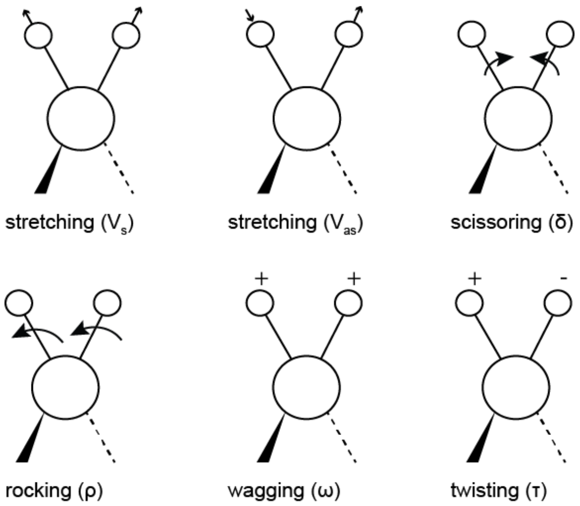

Figure 5. Vibrational modes in molecules

2.6 Raman Spectroscopy

Raman spectroscopy observes inelastic light scattering and uses lasers for excitation. The basic setup can be observed in figure 6. The laser hits a dichroic mirror and is then reflected to the sample, the Raman light produced by the shifted energy can pass through the mirror where the laser is blocked. Typically, the signal is very weak in a Raman experiment and the laser signal would overwhelm the light. In the final stage the light goes through a coupling lens and then into the spectrograph.

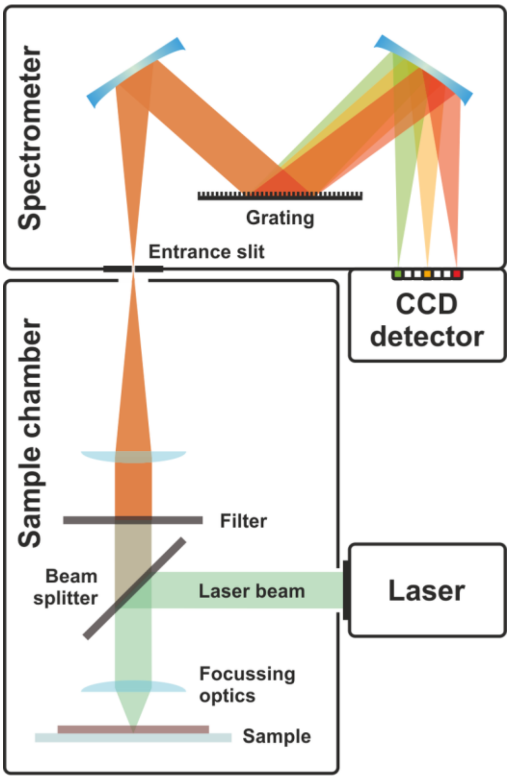

Figure 6 Raman spectrophotometer. Reprinted from: Wikipedia[20.]

Raman is very widely used for material identification or probing if materials are contaminated. [20.]

2.7 Luminescence

In luminescence processes the high energy state might be produced by a biological or chemical process or an electrical process. The luminescence like fluorescence or phosphorescence excites first an atom with incoming light and then light of different wavelengths is emitted.

2.8 Transmittance and Absorbance

The concepts of lights transmittance and absorbance are fundamental for the understanding of the Beer-Lambert Law.

Absorbance is the capacity of a substance to absorb light of a specified wavelength. Transmittance is the relation between the light absorbed by a substance and the light that comes through.

If a light at a specific frequency is transmitted through a solution (figure 5) with an incident intensity of I_0 and a transmitted intensity of I.

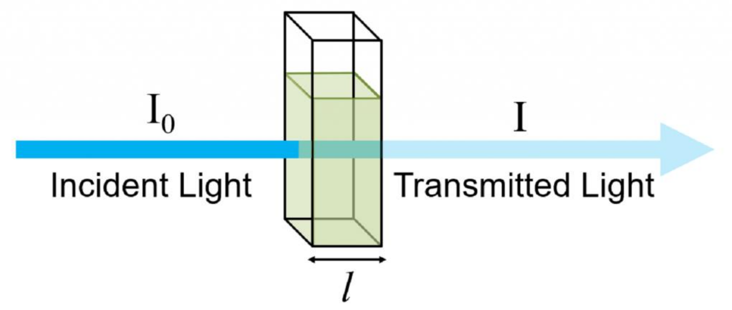

Figure 5 Transmission of light through a sample solution in a cuvette. Reprinted from Edinburgh Instruments [21.]

The transmittance T of the solution in the cuvette is given by the formula

$$T = \frac{I}{I_0} \qquad (2)$$

T: transmittance

I: incident light

I_0:transmitted light

$$A = \log_{10} \frac{I}{I_0} \qquad (3)$$

$$A = \log_{10} T \qquad (4)$$

2.9 Beer-Lambert Law

Beer-Lambert law

In Beer-Lambert the Absorbance is calculated.

$$A = \varepsilon * l * c \quad (5)$$

A: is the absorbance of the solution (no units)

ε: is the molar absorptivity or the molar extinction coefficient (L/mol.cm),

l: Path Length (cm)

c: Concentration (mol/L)

[21.]

3 Optical Spectrophotometers

Spectrophotometers use different technologies to measure different sections of the spectrum. Due to the physical work principles, it is impossible to make an instrument that could measure the whole spectrum. As shown in Figure 6 optical spectrometers have the same structure, there is a light source, a complex system of lenses, slits and mirrors that alter the frequency of the light at the end there is the detector.

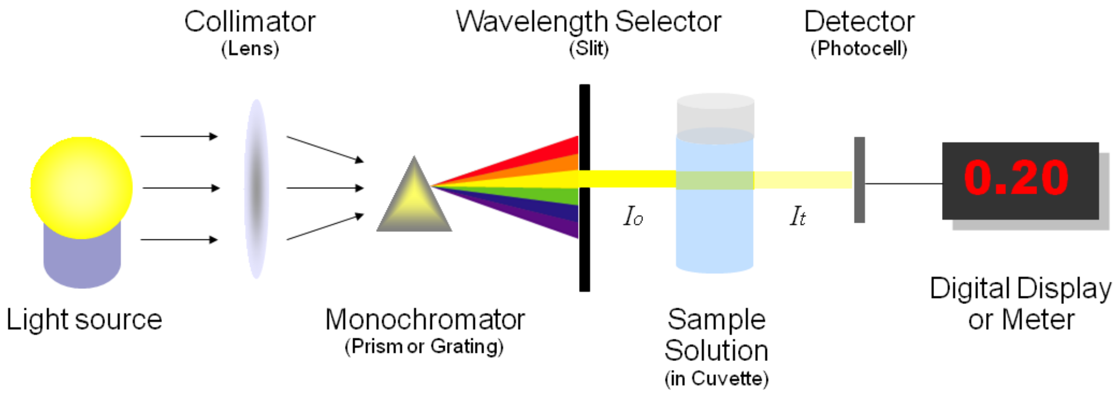

Figure 6 Basic structure of spectrophotometers. Reprinted from Libretexts [22.]

Spectrometers for absorption measurements are traditionally divided in small sections of the light’s spectrum:

- ultraviolet/visible (UV/VIS) (175-750 nm) range
- near-infrared (NIR) (0.8-2.5 μm) range
- mid-infrared (MIR) (2.5-25 μm) range
- far-in-frared (FIR) (25-1000 μm) range

3.1 Dispersive Spectrometers

These instruments have components that disperse the light, usually at infrared wavelength. This component can be a diffraction grate or a prism. Dispersive Spectrometers are divided into two categories: the monocromator and the dispersive.

3.1.1 Monocromators

A monocromator has a pair of slits, one at the entrance of the system and one at the exit. It uses only one detector. Light that passes through a monochromator and gets reflected by a rotating chopper mirror working as a disperser.

3.1.2 Spectrographs

In a spectrograph the dispersive element is fixed: a prism or a diffraction grate. There is no slit at the exit and there is an array of detectors, giving the possibility of a bigger range of wavelength but having a higher cost.

3.2 Fourier-Transform Spectrometers

The Fourier-Transform are based on a different principle. A component called interferometer uses the superposition principle to extract information. They are named after the Fourier transform that is used to interpret the data after detection. [22.]

4 Implementation and Analysis

This thesis analyses the process to design and test a portable spectrophotometer. The device is a proof-of-concept and will never be produced in series. The output voltage of the device was recorded initially with an Arduino and by the end of project with a multimeter. While testing, the gain of the operational amplifier required adjustment to ensure the amplifier will operate in the linear region.

4.1 Portable LED spectrophotometer

At an early stage of the thesis there was an investigation of several papers regarding spectrophotometers with a portable setup. The one that was more suitable as a starter for a thesis was “A practical portable photometer using LEDs as inspection light source”.[23]

Analysis of a Led Spectrophotometer.

A decision was made to follow only the LED module in detail because it was not in the goals of the thesis to go deeper in the use of LEDs, only a few modifications on the values of the resistances. On the module of Photodiode and LED the same Op-Amp, OP27, was used. A much simpler design was used for the photodiode’s module, following different methods. The original circuit was using two op-amp as transimpedance amplifier for the photodiode.

4.2 Hardware

The device consists of two different modules, one for the LED and a second one for the photodiode. The two modules are going to be separated to give the possibility to supply power in two different ways. The led module is a simple circuit with the possibility to change the LED to variate the frequency of the light. The module of the Photodiode has A positive 5V and a negative 5V to supply the OP-Amp.

4.2.1 Components

A list of components was redacted. Not all of them were used in the final prototype. The following list includes the most used components.

Sensor: BPW 21 OSRAM Silicon Photodiode for the Visible Spectral Range. [24.]

Op-Amp: OP27 Low noise high speed precision operational amplifier. [25.]

Arduino MKR WiFi 1010. [26.]

Several capacitors and resistances of different values.

LEDs of different values.

4.2.2 Design

In schematic in figure 7 the placement of the photodiode in relation with the op-amp

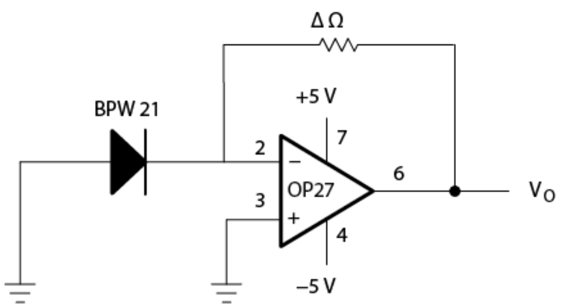

Figure 7. Photodiode module.

In figure 8 The two modules are connected for measuring the capacity of the photodiode. The LED and the photodiode are in the same box. The box will be shut close to prevent the ambient light from deviating the results. Two power suppliers are used instead of batteries to be able to control the circuit with different values.

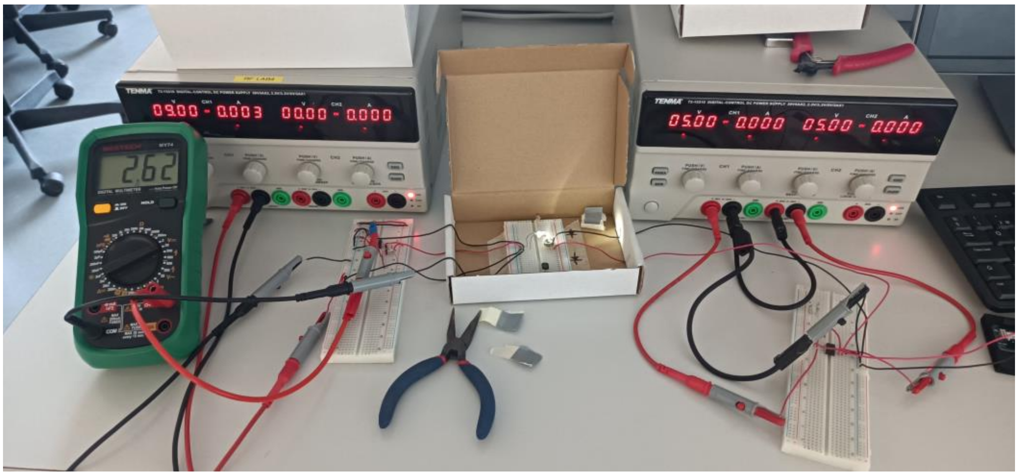

Figure 8.Different modules connected.

After the results a second setup was implemented. The difference was the use of different modules for the light, the cuvette and the sensor. In the light module, a slit made of razor blades was put in after the light source as is possible to see in figure 9.

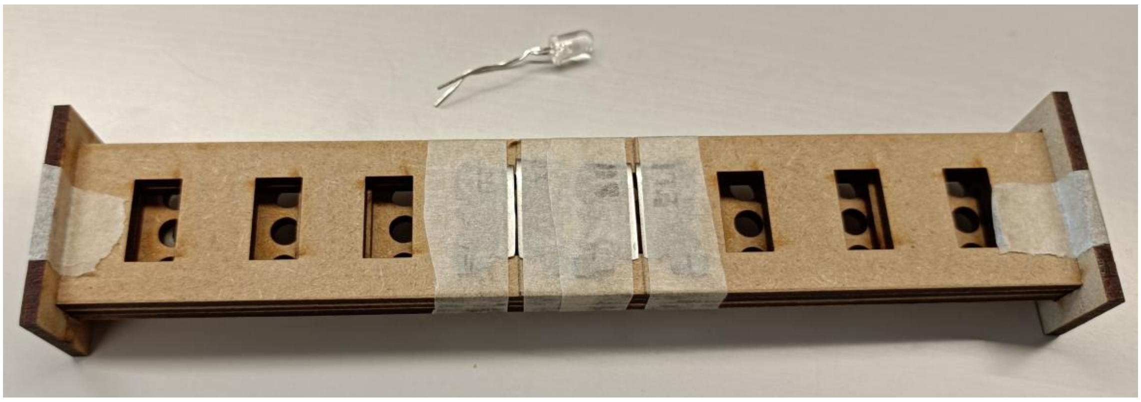

Figure 9. Case for LEDs with razorblade as slit.

In the sensor module, shown in figure 10, a small tunnel was placed before the active part of the sensor to give no possibilities to non-incident light to shine to the sensor.

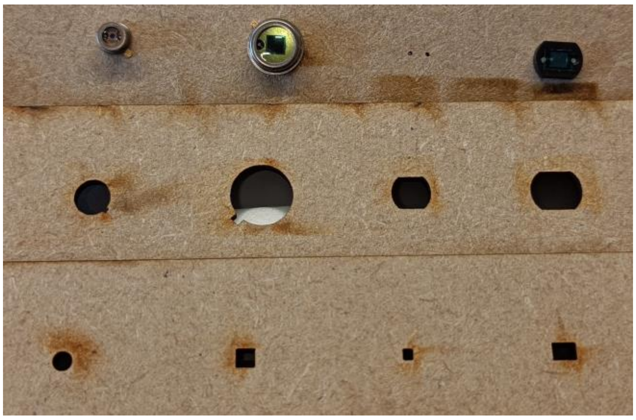

Figure 10. Sensor’s module.

The slit width was 0.7 mm~ and 20 mm hight, the tunnel was deep 3mm and the same area of the active part of the sensor.

In figure 11 is possible to see the assembly of the modules that substitued the box.

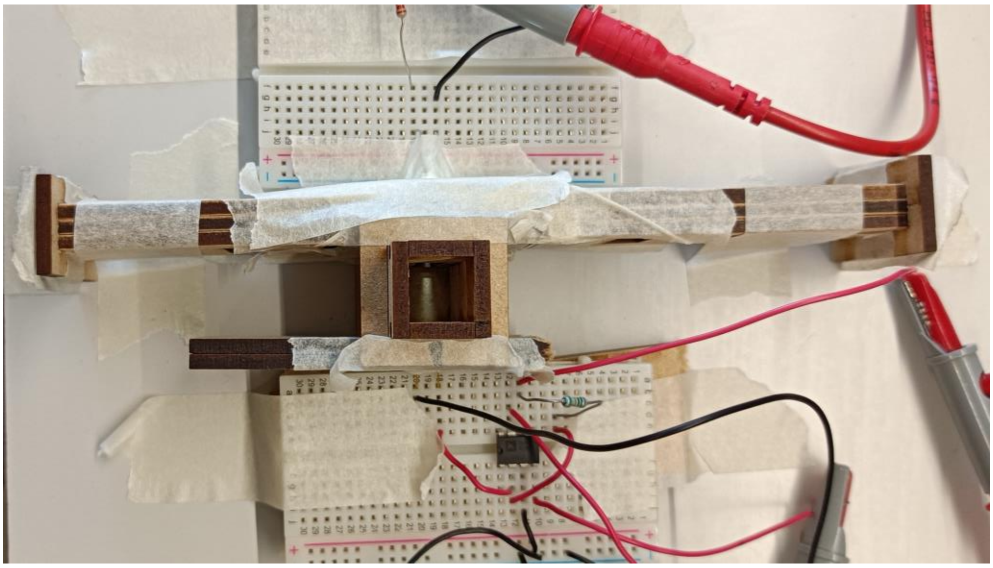

Figure 11. Modules assembled.

In figure 12 the beam of the with LED is visible inside the cuvette module. Projecting fron the slit of the LED module, left of the image, to the tunnel of the sensor.

Figure 12. Cuvette chamber.

4.2.3 Solutions

In figure 13 Samples were prepared by Patric Paxal from the Chemistry laboratory in Metropolia. They contained water and a blue dye agent. Patent Blue V is a sky blue dye used in the food and pharmacological industry. [27.] The samples were having different concentrations. Starting from 1 *Mg/L* to100 *Mg/L*.

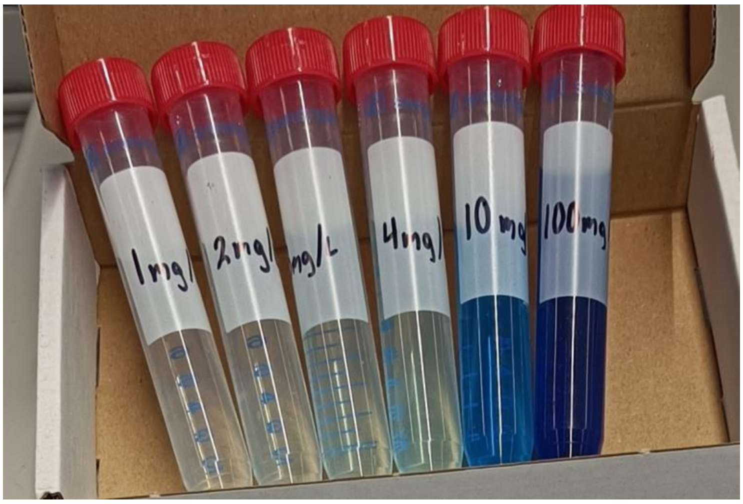

Figure 13. Patent blue V dye samples.

In figure 14 they were moved to the more appropriate cuvettes for the measurements.

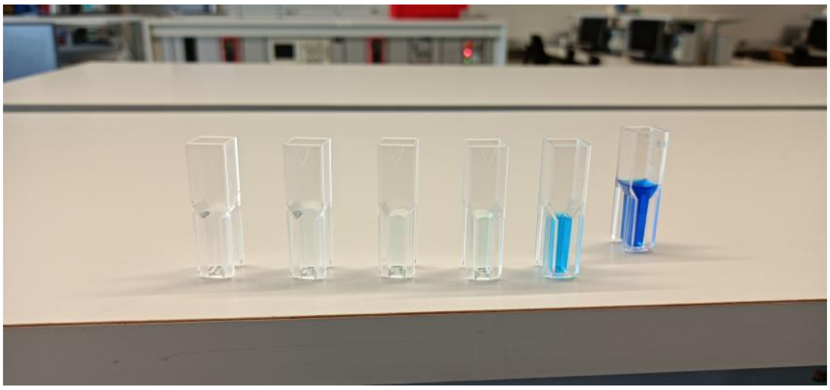

Figure 14. Cuvette for measurements in a spectrophotometer.

4.3 LEDs.

To test if the white led was not the only appropriate choice an RGB Led was tested with different concentrations of the solutions.

4.4 Results.

The results show in table 1, are the measurements of voltage output from the photodiode module at different solution concentration, with the different module in a box without a slit and without a tunnel.

Table 1. Photodiode output voltages.

	Empty cuvette	**100mg/l**	**10mg/l**	**4mg/l**	**3mg/l**	**2mg/l**	**1mg/l**
White LED	1,1V	0,45V	0,78V	1,23V	1,35V	1,21V	1,25V

Blue LED	0,44V	0,68V	0,44V /liar	0,44V /liar	0,44V /liar	0,44V /liar	0,44V /liar
Red LED	1,56V	0,27V	0,87V	2,23V	2,23V	2,39V	2,83V
Green LED	0,67V	0,50V	0,32V	0,64V	0,68V	0,68V	0,68V

The results were measured on a multimeter.

A second set of results were measured when the device was assembled with different modules. The slit for the LED and the tunnel for the sensor is in place. Due to the nature of the prototype the measurements were repeated when the initial state without cuvette was showing a different value from the original.

As it can be seen from table 2 the white LED was giving the best results. The blue led was giving some liar, already at 4mg/l, since the liquid was on a similar frequency of light. In the red light was acting as a filter giving a no voltage result with a concentration of 100mg/l.

Table 2. New set Photodiode output voltages.

Newset	Water in the cuvette	100mg/l	10mg/l	4mg/l	3mg/l	2mg/l	1mg/l
White LED 4,72V no cuvette	**5,16V**	**1,04V**	**2,61V**	**4,80V**	**4,89V**	**4,91V**	**5,13V**
Blue LED 2.00v no cuvette	2.45V	1.66V	2.33V	2.42V	2,43V /liar	2,44V /liar	2,48V /liar
Red LED 2.88V no cuvette	3,33V	0,00V	0,01V	2,57V	2,73V	2,85V	2,98V
Green	1,50V	0,15V	1,08V	1,46V	1,47V	1,45V	1,50V

LED 1,16V no cuvette							

5 Conclusions

The project had three main steps and a single goal.

The first step was to acquire and understand the basic information regarding Spectroscopy, starting with the history of this field of science, how the matter is affected by the absorption and emission of electromagnetic radiation, understanding the light as electromagnetic radiation.

The second step was to learn and understand Spectrometry. Not only the understanding of the relation between electromagnetic radiation and matter, but also the measurement of the radiation itself.

The third step was to build a device that would behave like a spectrophotometer. That final step was never intended to produce an investigation tool that would compete with laboratory equipment. As a proof-of-concept, project was used to learn how to save resources, to make a better planning process for the next project.

The main goal of the thesis project was to use this study to test the skills learned during these years in school, the research and analysis of information, the capacity of implementing concepts, the capacity of collecting the measures and producing a report.

The results were meet with some initial difficulties, being the first set of measurements not reliable. The second set was giving interesting results and a good understanding of the difference in using a more complex device.

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