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Detection of Absorbance of CuSO_4 , CrCl_3 , $\text{CrCl}_2 \cdot 2\text{H}_2\text{O}$ and NiCl_2 Solution Samples using Home Made Visible Spectrophotometer

Sewmehon Mengistie

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Bahir Dar University

College of Science

Department of Physics

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by

Sewmehon Mengistie

November, 2022

Bahir Dar, Ethiopia

Bahir Dar University

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Solution Samples using Home Made Visible Spectrophotometer**

A Thesis Submitted to the Department of Physics, In Partial Fulfillment of the
Requirements for the Degree of Masters of Science in Laser Physics

by

Sewmehon Mengistie

Advisor: Getasew Admasu (Ph.D.)

November, 2022

Bahir Dar, Ethiopia

Declaration

This is to certify that the thesis entitled “**Detection of Absorbance of CuSO₄, CrCl₃, CrCl₂.216 and NiCl₂ Solution Samples using Home Made Visible Spectrophotometer**”, submitted in partial fulfillment of the requirements for the degree of Master of Science in Laser and Spectroscopy physics of Department of physics, Bahir Dar University, is a record of original work carried out by me and has never been submitted to this or any other institution to get any other degree or certificates. The assistance and help I received during the course of this investigation have been duly acknowledged.

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Date

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Bahir Dar University
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Approval of Thesis for Defense

I hereby certify that I have surprised, read, and evaluated this thesis titled “**Detection of Absorbance of CuSO₄, CrCl₃, CrCl₂.216 and NiCl₂ Solution Samples using Home Made Visible Spectrophotometer**” by Sewmehon Mengistie prepared under my guidance. I recommend the thesis be submitted for oral defense.

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Bahir Dar University
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Approval of the Thesis for Defense Result

As members of the board of examiners, we examined this thesis entitled “**Detection of Absorbance of CuSO₄, CrCl₃, CrCl₂.216 and NiCl₂ Solution Samples using Home Made Visible Spectrophotometer**” by Sewmehon Mengistie. We hereby certify that the thesis is accepted for fulfilling the requirements for the award of the degree of “Master of Science in Laser physics.

Board of Examiners

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Abstract

This study aimed to design a simple and low-cost visible spectrophotometer for the detection of the absorbance of colorful metallic solutions. The designed instrument has a tungsten white light bulb source, a light dependent resistor (LDR) as a sensor, a diffraction grating for light dispersion, a converging lens to collimate the light, filters of different wavelengths, a digital voltmeter, and an ammeter as a detection element. To maintain a constant light intensity, a commonly used power supply was set at 6 V for the light source bulb. The instrument was designed to have a simple and compact size of 55 cm × 12cm × 22 cm in length, width, and height, respectively.

The performance of a newly designed spectrophotometer has been tested on solutions of copper sulfate (CuSO_4) and chromium chloride (CrCl_3) at different filter wavelengths. The results show a linearly dependent absorbance as a function of concentration for these four filters' wavelengths (blue, red, yellow, and yellow-green). The R squared values were in the order of 97 % with an error of 3 % and 98 % with an error of 2 %, respectively. This is consistent with the theoretical R-squared values and the commercial UV-Vis spectrophotometer, with minor differences. The absorbance as a function of the wavelength measuring capacity of the instrument is also tested with copper sulfate (CuSO_4), chromium chloride ($\text{CrCl}_2 \cdot 216$), and nickel chloride (NiCl_2) solutions at a constant concentration. The R^2 values of these graphs were in the order of 98 % with an error of 2 %, 97.7 % with an error of 2.3 %, and 92 % with an error of 8 %, respectively.

The shapes of the graph of absorbance as a function of wavelength for these samples have a close similarity with the absorbance graphs of the Commercial UV-Vis spectrophotometer (DR6000) with only small differences. It is believed that this newly designed spectrophotometer has a low cost and can be used for the detection of colorful solution samples in low-income countries like Ethiopia.

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Thirdly, I would like to thank Dr. Addis Mekonen for his eager cooperation by giving his own prepared copper nanoparticle to test the effectiveness of the measuring capability of this home-made visible spectrophotometer even if it is not used for such purpose due to its nature of insolubility in water.

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Abbreviations

BFO	Bismuth iron oxide
BFCO	Cobalt doping bismuth iron oxide
BPV	bulk photovoltaic
cm	centimeter
FTIR	Fourier-transform infrared spectroscopy
nm	nanometer
PCE	power conversion efficiency
S-Q	Shockley-Queisser
XRD	X-ray powder diffraction
UV-Vis	Ultraviolet–visible spectroscopy

Chapter One: Introduction

1.1. Background of the study

The optical spectrum is a set of wavelength (or frequency) ranges that are created by a light source. Each optical spectrum can be absorbed, emitted, reflected, or have any other property when it interacts with matter. To study the properties of light-matter interaction, an apparatus known as a spectrophotometer can be used. A spectrophotometer is an instrument that uses the principle of light-matter interaction. It enables us to characterize the chemical and physical properties of substances in terms of their capability of absorbing and transmitting light from a sample as a function of wavelength. The spectrophotometer was originally designed to estimate vitamin content in US military rations. Later, it became one of the most widely used measuring instruments of all time in various fields of experimental science. Nobel laureate chemist Bruce Merrifield described the spectrophotometer as "the most important instrument ever developed toward the advancement of bioscience" [1,2]. The instrument is commonly applied in the physical, chemical, and medical sciences. It is commonly used in the fields of biochemistry, chemistry, physics, biology, biochemistry, material and chemical engineering, clinical applications, industrial applications, material science, and more [3]. Many of these applications require highly precise and reliable data, which often requires the implementation of complex and expensive spectrophotometric systems. This crucial instrument can have different types depending on the range of the optical spectrum it uses and its mode of operation. Depending on the range of wavelengths of the light source, spectrophotometers can be classified into two different types. UV-Vis A spectrophotometer is a device that measures light in the ultraviolet (185-400 nm) and visible (400-700 nm) ranges of the electromagnetic spectrum. An infrared (IR) spectrophotometer is also another type of spectrophotometer that uses light in the infrared range (700–15000 nm) of the electromagnetic spectrum. Spectrophotometry is also known as atomic absorption spectroscopy or atomic emission spectroscopy, depending on how it operates. The atomic absorption spectrophotometric method uses the phenomenon of absorbance of the electromagnetic (EM) spectrum and the atomic emission when used for measurement; spectrophotometry makes use of the electromagnetic spectrum's emission. The principle of ultraviolet-visible (UV-Vis) spectrophotometry is the measurement of light absorption as it passes through a material and provides information about the electronic transitions that happen

within the sample. With these measurements, one can then determine the concentration of a sample using Beer's Law, which relates the absorption of light with its concentration as it travels through materials. UV-Vis's spectrophotometry can also allow for these electron transitions to be characterized as indirect or direct and if it is allowed or forbidden [4]. In visible spectroscopy, the absorption or transmission of a certain substance can be determined by the observed color. For example, a sample solution that absorbs light in all visible ranges (i.e., no transmittance of visible wavelengths) is black in color in theory. On the contrary, if all visible light wavelengths are transmitted (i.e., not absorbed), the sample solution is white. If a solution sample absorbs red light (700 nm), it appears green because green is the complementary color of red. In practice, visible spectrophotometers use a prism to select a certain range of wavelength (to filter out other wavelengths) so that a single wavelength of light is passed through a sample solution. This spectrophotometer only works in the range of visible light (420-650nm); hence it has been termed as a visible spectrophotometer [5].

Generally, spectrophotometry is widely used for quantitative analysis in various areas. Any application that deals with chemical and physical properties of substances or materials can use this technique. Although the spectrophotometer has such many applications, its accessibility is limited for developing countries like Ethiopia. This is due to its high cost and shortage of skilled manpower to operate and maintain the instrument. So, this thesis report can contribute its own part to minimize the problem by designing a homemade spectrophotometer for the detection of absorbance of colorful solution samples in the visible spectral region. Any colorful solution sample, whose spectrum is known, will be detected using this designed home-made visible spectrophotometer. This instrument was developed by using easily accessible materials. Its cost is very low as compared to the commercial one and its working principle is easily understandable.

1.2. Statement of the Study

Now a day, general spectrophotometers are sold at a high cost. This high cost becomes a problem for schools, universities and other organizations in developing countries like Ethiopia. The working principles of UV-Vis Spectrophotometer are hidden for most pupils since its mathematical and overall activities are completed inside the box. This is also another problem to ran and use the instrument correctly. For this reason, designing a simple, portable and low-cost spectrophotometer in our home and using it for laboratory activities is one way of solution to

overcome the cost problem. Due to this, the thesis is concerned to solve such problem by designing simple and low-cost homemade visible spectrophotometer for the detection of absorbance of colorful solution samples in the visible spectral region.

1.3. Objective of the Study

This thesis proposal has its own general and specific objectives.

1.3.1. General Objectives

- To design an optical absorption visible spectrophotometer for the detection of absorbance of colorful solution samples in the visible spectral region.

1.3.2. Specific Objectives

- To understand the basic working principles of a spectrophotometer.
- To design a home-made spectrophotometer with available resources in the department.
- To test the optical absorption measurements on the home-made visible spectrophotometer and compare with the measurement of commercial UV-Vis spectrophotometer absorbance spectrum.

1.4. Significance of the Study

In reality, Spectrophotometer is an instrument that enables us to measure absorbance and transmittance of a sample as a function of wavelength by using the principles of light-matter interaction. Spectrophotometric systems are widely used in many fields such as physics, materials science, chemistry, biochemistry, bio molecular chemistry, medicine, engineering and more. These applications require highly precise and reliable data which often leads to the implementation of complex and expensive spectrophotometric systems. Although the spectrophotometer is useful for the study of the optical characterization of materials and so many applications, it is expensive and also has complex instrumentation to use. Its high cost makes the instrument not easily accessible to many organizations. Of course, the cost may not be a problem for developed countries, but it becomes a serious problem when we come to developing countries like Ethiopia. So, this new designed simple and low-cost home-made visible spectrophotometer can simplify the problem and enables the developing countries to have an access of such instrument.

1.5. Organization of the Thesis

The thesis is organized in to five chapters. Chapter one describes about the background, Statement, Objective and significance of the study; Chapter two talks about theoretical aspects of the spectrophotometer and its working principles; chapter three briefly tells about the methods and the materials used to design visible spectrophotometer, Chapter four contains result and discussion parts and Chapter five is the final part of this thesis which comprises the conclusion and outlook part of this thesis.

Chapter Two: Literature Review

2.1. Definition and Parts of UV-Vis Spectrophotometer

Spectrophotometer is an instrument which is used to measure the degree of absorbance of light at certain wavelengths. It is the study of interaction of electromagnetic radiation with matter. It is also a powerful tool which is applicable for quantitative and qualitative analysis of samples and is the most essential of all the instrumental methods of analysis. Based on the optical beam, spectrophotometers are two types: single beam and double beam. A single beam UV-Vis spectrophotometer measures the relative light intensity of the beam before and after a test sample is inserted. A double beam spectrophotometer compares the light intensity between two light paths, one path containing a reference sample and the other path contains the test sample. The main parts of spectrophotometer are light sources, monochromators, detectors and display unit. The light source serves different types of wavelengths of optical spectrum. The light sources can be deuterium, halogen, xenon, and tungsten lamp, etc... A spectrometer acts as a wavelength dispersion element. The heart of the spectrometer is the monochromator whose function is to disperse the light according to their wavelengths [6]. The monochromator contains collimating and focusing mirrors on addition to gratings and slits. The monochromator forms an image of the entrance slit in the exit slit plane at the wavelength present in the light source [7]. There are two configurations for the monochromator: Fastie-Ebert and Czerny–turner configuration [8]. The detectors function as a tool to quantify the intensity of light that is transmitted through the substances in the sample. There are a lot of detectors used in spectrophotometers such as photomultiplier tube, charge coupled devices and photodiode. They are used to convert the transmitted light (photons) to current or voltage for further processing [9]. The signal received from the detector is the low current signal which requires amplification and signal conditioning so as to reject different types of noise. Modification of spectrophotometer instruments can be done by improving and updating the main part of the spectrophotometer, such as using a pointer light as a light source [10] and Light emitting diodes (LEDs) [11–13] and using Arduino and digital cameras as a detector [14–17]. In addition, different types of spectrophotometer instruments have been developed such as UV-Vis [18–21], Flame photometer [22,23] and spectrophotometer simulations using computer programs and augmented reality [24,25]. UV-Vis spectrophotometer uses light spectrums in the ultraviolet range (185 - 400 nm) and visible range (400 - 700 nm).

2.2. Working Principles of UV-Vis Spectrophotometer

In the present day, spectrophotometry is a well-known and a well-established measurement method of both physical and chemical quantities. It is one of the most widely applied physicochemical techniques which are remarkable for its sensitivity and precision. It is also an experimental technique that is used for measuring the concentration of solutes in a specific solution by detecting the amount of light absorbed by those solutes. This technique is useful because certain compounds will absorb different wavelengths of light at different intensities. For example, spectrophotometry has been used in determining absorbance, film thickness and semiconductor band gap [26–28]. Spectrophotometry which uses the visible region of electromagnetic spectrum from 380nm to 780nm is often called colorimetry. Colorimetry is a technique of analysis based on comparing the unknown color intensity with that of standard solution. The applicability of spectrophotometric technique for the determination of traces of metals, nonmetals and organic compounds is one of the main reasons for the wide utilization of photometric methods of analysis. In spectrophotometric analysis, the intensity of light spectrum, passed through an absorbing sample which is placed between the light source and the detector, is measured as a function of wavelength. The graph between the transmitted or absorbed intensity of light and its wavelength is called the absorption spectrum which is a characteristic of the absorbing component and forms the basis for qualitative analysis. The absorbed or transmitted intensity of light is also a measure of concentration, and this provides the basis for the quantitative analysis. When a beam of light is incident on matters, several types of interactions such as reflection, refraction, diffraction, interference, absorption etc., may occur. Of these interactions, absorption in which certain wavelengths are selectively removed by matter is of unique importance. A ray of light can either be absorbed by some materials or simply pass through the materials without being affected. When a molecule absorbs light, energy is transferred from the light ray to the molecule. If the frequency of the electronic and magnetic fields of the light ray matches the frequency at which molecules will vibrate, then light will be absorbed. If the frequency does not match, the light will pass straight through the molecule unaltered. Inert molecules whether solid or liquid appear colored due to the way they modify light illuminating the object. Thus, different objects absorb some wavelengths and reflect others. For example, if a white light passes through a blue solution, it absorbs all colors except blue. The techniques of spectrophotometry are to measure the intensity of light across the UV-Vis spectrum as it passes through a sample and a blank. A blank is a solution filled in a cuvette that

does not absorb any of the wavelengths of light. It allows for a full spectrum to be measured. Typically, white light passes through a slit and proceeds to pass through a sample. The transmitted light diffracts through the diffraction grating and has a picture of the spectrum taken by a camera.

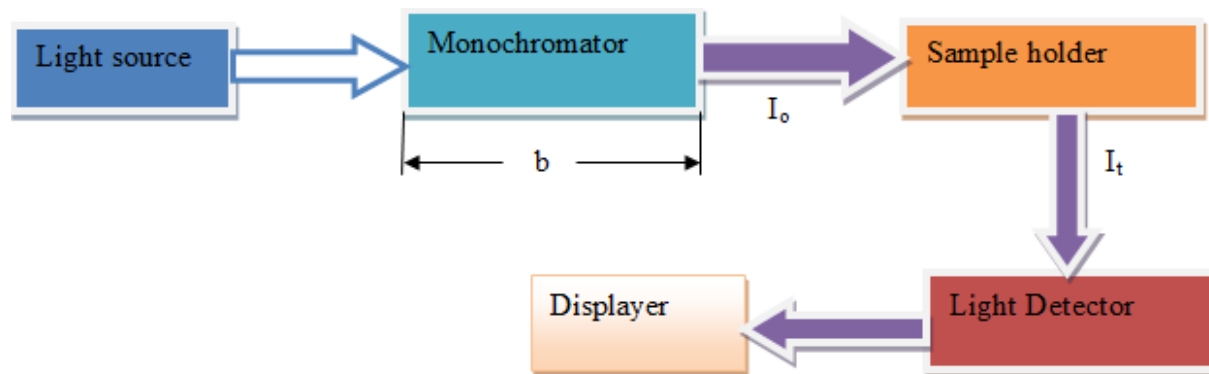


Figure 2. 1: Diagrammatical representation of a spectrophotometer.

The spectrophotometer measures the absorbance of a sample according to Beer Lambert law. It is a quantitative method which uses the two well-known laws to quantify absorbed light in a substance. These two fundamental laws governing the fraction of incident radiation absorbed by matter are

I. Lambert's law and Beer's law

Lambert law proposed that the portion of light absorbed by a medium is independent of the incident light intensity; and Beer stated that the absorption of light is directly proportional to both the concentration of the absorbing medium and thickness of the medium (path length) and allow the measuring sample of different path lengths.

$$\text{Absorbency} = \text{concentration} \times \text{path length}$$

II. Lambert's Law

The relationship between the absorption of light and the path length of the absorbing substance was first formulated by Bouguer (1729) and later by Lambert (1760) which is mathematically expressed in the logarithmic form as

$$\log T = \log \frac{I_T}{I_0} \quad (2.1)$$

Where, ‘ I_0 ’ is radiant power of incident radiation, ‘ I_T ’ is radiant power of transmitted radiation, ‘ T ’ is transmittance.

III. Beer's Law

Beer in 1852 studied the effect of concentration of the constituent in a colored solution by using the light transmitted through or absorbed by the solution [29]. He found similar relationship between transmission and concentration as Lambert had found between transmission and thickness of the absorbing medium. The intensity of monochromatic light decreases exponentially as the concentration of the absorbing medium increases arithmetically. This may be written in the form of

$$\log T = \log \frac{I_T}{I_0} = -k c \quad (2.2)$$

Where, ‘ k ’ is proportionality constant and ‘ c ’ is the concentration.

The two laws are combined using a new proportionality constant ‘ a ’ and written as

$$\log \left(\frac{I_T}{I_0} \right) = -a b c \quad (2.3)$$

Where, ‘ b ’ is the path length and ‘ a ’ is a constant. The amount of light (photons) passing through the cuvette and into the detector is dependent on the thickness of the cuvette and the concentration of the sample. Once we know the light intensity passing through the cuvette, we can relate it to transmittance (T). Transmittance is the amount (fraction) of light that passes through the sample. This can be calculated by using the equation;

$$\text{Transmittance } (T) = \frac{I_t}{I_0} \quad (2.4)$$

Where, I_T it is the light intensity after passing through the cuvette and I_0 is the light intensity before passing through the cuvette. Transmittance and absorbance are related by the expression;

$$\text{Absorbance}(A) = -\log \left(\frac{I_t}{I_o} \right) = -\log T \quad (2.5)$$

Where; absorbance (A) is the amount of light (photon) that is absorbed. From the above equation, the unknown concentration of the sample can be determined by using Beer-Lambert Law. The ratio of I_T/I_0 is known as transmittance (T) which is dimensionless quantity. As ' abc ' is a logarithmic quantity, it is a pure number. The above equation can be written as

$$A = abc \quad (2.7)$$

The constant 'a' is called absorptive if the concentration is expressed in grams/litter. If the concentration is expressed in moles/litter, the constant 'a' is replaced by ' ϵ ' called molar absorptive. Thus,

$$A = \epsilon bc \quad (2.8)$$

Equation (2.8) is a fundamental law which is governing the absorption of all types of light spectrum and applies not only to solutions but also to gases and solids as well.

2.3. Deviations from Beer's Law

There are no exceptions that are known for the linear relationship between absorbance and path length at a fixed concentration of absorbing medium. With constant path length (b) deviations from the direct proportionality, between the measured absorbance and concentration, will occur as a result of chemical and instrumental factors. The confirmation of Beer's law is tested experimentally by plotting a graph between absorbance and concentration. A straight-line plot passing through the origin indicates the confirmation to Beer's law. A non-linear plot tells the

deviation from the Beer's law. Deviations are termed positive and negative depending on the observed curve which is above and below the theoretical straight line respectively. The Beer's law is fundamentally a restricted law which is valid and obeyed at low concentrations; but the law does not hold good for higher concentration solutions [30]. The deviations may be attributed to so many factors such as reflection of radiation by the test solution, pure solvent, transmission, unable to obtain monochromatic radiation, fluorescence or scattering, change of structure of the colored species with concentration, reaction of electrolytes with the colored complex when present in large amount, temperature, ionization, association or dissociation of colored solutes in solution and inert salts when present even in very low concentration due to the influence of electrostatic interactions [31]. It is still possible to apply spectrophotometry in quantitative analysis with marked success provided that necessary precautionary measures are taken to control the experimental conditions.

2.4. Ring Bom's Plot

There is an uncertainty in measurements due to the error of the instrument even in the range of measurement which is confirmed to Beer's law. A small random error in the reading of percentage transmittance (T) causes a large relative error in concentration when transmittance 'T' is either very small or very large. Practically, the range of concentration can be determined spectrophotometrically by the help of Ring Bom's plot.

In Ring Bom's plot, a graph is drawn between percentage absorbance against logarithm of the concentration in which 'S' shaped or 'sigmoid curve' is obtained [32]. The term absorbance is defined as

$$\text{Absorbance}(A) = 1 - \text{Transmittance}(T) = 1 - T \quad (2.9)$$

$$\% \text{ Absorbance}(A) = 100 - \% \text{ Transmittance}(\%T) \quad (2.10)$$

Deviation from Beer's law leads Ring Bom to arrive at the conclusion that the accuracy is greatest at the point of steepest slope (inflection point) of the curve between the power output (P) and the logarithmic concentration (log C). Thus, if a system obeys Beer's law the inflection point

occurs at 36.8 % transmittance or 62.2 % absorbance and the corresponding absorbance value is 0.4343. The Ring Bom's plot has two advantages. Firstly, it shows the concentration range in which the analysis error is minimum; this is the concentration range corresponding to the nearly linear portion of the Ring Bom's plot and secondly the accuracy of the analysis at any concentration range can be easily evaluated.

If the system under study does not obey Beer's law over the entire range of concentration, the Ring Bom's plot takes a sigmoid shape. The curve will have a different slope and the inflection point appears at a value other than 63.2% absorbance. However, the curve still shows the optimum concentration range and the relative analysis error can be calculated from the slope of the curve.

2.5. Sensitivity of a Spectrophotometer

Sensitivity is defined as the least amount of the determined species that can be obtained spectrophotometrically under experimental conditions. It refers to the calibration curve obtained between concentration and absorbance values. The sensitivity is numerically expressed as molar absorptive

$$\epsilon = \frac{A}{bc} \quad (2.11)$$

Where 'A' is absorbance, 'b' is the path length expressed in cm and 'c' is concentration in moles per liter. The units for molar absorptive are liter per mole per cm. Quantum mechanically, molar absorptive (ϵ) value cannot exceed 1.5×10^5 . Spectrometric methods with $\epsilon > 1 \times 10^4$ are referred to as sensitive while those with $\epsilon < 1 \times 10^3$ as less sensitive. Sensitivity is sometimes expressed in terms of specific absorptive. Specific absorptive is defined as the maximum absorbance of a 1% solution over a 1 cm path length measured with a spectroscopy. It is denoted by 'a' and is given by the expression

$$a = \frac{(\text{absorbance} \times \text{dilution factor}) \times 10 \text{ mg/mL}}{\text{concentration (mg/mL)}} \quad (2.12)$$

The unit for specific absorptive 'a' are $\text{ml gm}^{-1}\text{cm}^{-1}$ and its value corresponds to the absorbance of 1 $\mu\text{g/ml}$ (1 ppm) solution in a cuvette with an optical path length of 1 cm. Mostly, the sensitivity is expressed in terms of sensitivity index (S) given by Sand ell [33]. According to sand ell, sensitivity is the amount of absorbing species in microgram per milliliter (ml) of the solution (1 ppm) with an absorbance of 0.001 for a path length of 1 cm. The units of 'S' are $\mu\text{g cm}^{-2}$ and is given by

$$S = \frac{10^3}{a} \quad (2.13)$$

Where; 'a' is specific absorptive. The major drawback of the absorbency detection in micro volumes is that the optical path length. It decreases through the sample as the sample volume decrease and this affects the sensitivity.

Generally, different methods are available to study optical properties of various types of materials in small size. For example, scanning/transmission electron microscopy (SEM/TEM) are techniques used to obtain high-resolution (down to sub-nanometer) optical information of Nano materials (NMs); Atomic force microscopy (AFM) gives Nano-scale resolution in the vertical (z axis) dimension; and X-ray diffraction (XRD) gives information on the NMs atomic structure; all these techniques can only be used on dry samples (powders) [34,35]. Methods suitable for the characterization of NMs in liquid media include field flow fractionation (FFF), which enables the differentiation of large molecules, aggregates, and particles based on their size; Dynamic light scattering (DLS) and Nano-particle tracking analysis (NTA)—these two methods are widely used to determine the size distribution profile of particles using Brownian. Ultraviolet-visible spectrophotometry (UV-Vis spectrophotometer) allows NM characteristics assessment such as size, aggregation state, and refractive index by a simple absorption measurement [35–37]. Although all these methods allow NM characterization, their performance is dependent on instrument setup, instrument-based differences, complex methodology of sample preparation, and the user's level of expertise. Moreover, most of the methods do not allow real-time monitoring of NM size, sample integrity, or differentiation between dispersed or aggregated particles [38]. UV-Vis's spectroscopy is a widely used method that provides non-invasive and fast real-time evaluation of NM size, concentration, and

aggregation state. Additionally, it is a simple and inexpensive process with minimal sample preparation, which makes this method an essential tool that is extensively used in various laboratories within many disciplines and markets [36–38]. UV-Vis works by measuring the transmittance of optical radiation of a wavelength between 180 and 1100 nm through a liquid sample. The UV and Vis optical spectrum covers the wavelength range for the ultraviolet (170 nm to 380 nm), visible (380 nm to 780 nm), and near-infrared (780 nm to 3300 nm) [37,39]. The wavelength of light spectrum passing through the sample cell is measured; the intensity of light incident to the sample is designated as I_0 , and the intensity of the light emerging on the other side is represented as I_T [37]. The Beer-Lambert law studies the relationship between absorbance (A) as a function of concentration (C) of a given sample, extinction coefficient (ϵ) of a given sample, and the two intensities [37]. Absorption measurements can be gathered at a single wavelength or over an extended spectral range; the measured light transmittance is used to calculate an absorbance value by the Beer-Lambert law equation of (2.8). The absorbance can also be calculated as the ratio between the intensity of a reference sample ' I_0 ' and the unknown sample ' I_T ', as described in equation (2.5) [37]:

Therefore; depending on path length (l), Nano-particle concentration(c) and extinction coefficient (ϵ), absorbance (A) can be obtained. So, UV-Vis's spectrophotometry is a useful instrument to characterize metal Nano-particles that they possess bright color which is visible by naked eye. By this technique, in addition to quantitative measurement, qualitative information about the Nano-particle can be obtained.

Like the commercial UV-Vis spectrophotometer, the Bear Lambert's law is a crucial law to calculate the measured absorbance values of the designed homemade visible spectrophotometer. This law correlates absorbance (A) with molar absorptivity (ϵ ; L/mol.cm), path length of light in the sample (b; cm), and concentration (C; mol/L) by using equation (2.8) [40].

Then, when the above relation in equation (3.1) involves the light transmittance (T) and light intensity (I), the absorbance A is defined using equation (2.5) [40]. Since transmittance is related with current and voltage by using the following formula;

$$Transmittance(T) = \frac{I}{I_0} = \frac{V_{sample} - V_{zero}}{V_{solvent} - V_{zero}} = \frac{I_{sample}}{I_{solvent}} \quad (2.14)$$

$$A = -\log_{10}(T) = -\log_{10}\left(\frac{I}{I_0}\right) = \log_{10}\left(\frac{I_0}{I_T}\right) \quad (2.15)$$

$$A = -\log_{10}(T) = -\log_{10}\left(\frac{V_{sample} - V_{zero}}{V_{solvent} - V_{zero}}\right) = \log_{10}\left(\frac{V_{solvent} - V_{zero}}{V_{sample} - V_{zero}}\right) = -\log\left(\frac{I_{sample}}{I_{solvent}}\right) \quad (2.16)$$

Where; $V_{solvent}$ is the voltage measured with a blank sample (distilled water), V_{sample} is the voltage measured with the sample solution, V_{zero} is the voltage reading when the light source is off (in the dark signal), I_{sample} is the current measured with the sample and $I_{solvent}$ is the current measured with a blank sample (distilled water).

However, when equation (2.14) and (2.16) are used to calculate transmittance and absorbance, the characteristics of the detector (LDR) should be considered since its displayed voltage readings do not have a direct relationship to the light intensity like that of the intensity detected by the detector in a commercial UV-Vis spectrophotometer. This is due to the special characteristics of the LDR.

The LDR has a high resistance in the dark and a low resistance when it gets light. As a result, voltage reading of a sample is greater than the voltage reading of a solvent (a blank). Due to this, the transmittance is greater than one and the absorbance becomes negative. So to correct this, ignored the minus sign in the logarithmic equation or use the given logarithmic equations by inverting each calculated values of the transmittance since the voltage dropped on the LDR has inverse relationship with the transmitted light. This means that if the voltage dropped on the LDR increases, the amount of the transmitted light will be decreased. For current output, however, it is possible to equation (2.14) and (2.16) directly since the amount of current reading is directly proportional with the transmitted light.

The commercial UV-Vis Spectrophotometer gives information about absorbance of a substance by displaying its absorbance value on the displayer directly although its detector detects the transmitted light as a voltage output. The instrument contains an internal processor to do the necessary mathematics which is immediately converting voltage reading to absorbance.

However, in the home made spectrophotometer, the absorbance value can readily be calculated with a calculator or spreadsheet.

Chapter Three: Materials and Methods

3.1. Materials for the Homemade Spectrophotometer

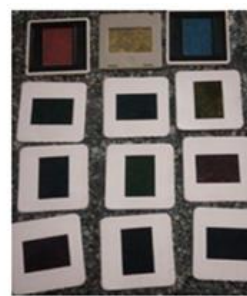
The homemade spectrophotometer was designed using the following materials found in the department of physics at BDU: These include a tungsten lamp, a diffraction grating, a lens, a detector (a light-dependent resistor, or LDR), a displayer (a voltage meter and an ammeter), a sample holder (a cuvette), and Styrofoam. The solution samples used for calibration in this thesis are copper sulfate (CuSO_4), chromium chloride (CrCl_3 and $\text{CrCl}_2 \cdot 2\text{H}_2\text{O}$), and nickel chloride (NiCl_2). They are all Aldrich Sigma in various proportions. Figures 3.1 and 3.2 show photos of the materials used to build the homemade spectrophotometer.



Light dependent resistor (LDR)



Diffraction grating



Filters



Converging lens



Digital voltmeter

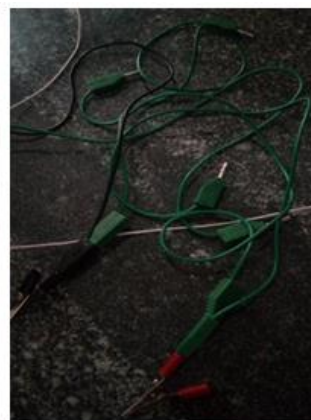
Figure 3.1: Light Dependent Resistor (LDR), diffraction grating, converging lens, filters and digital voltmeter.



Holder



Bulb



Wires



Analogue micro Ammeter



Power supply (2 V – 12 V)



Cuvette (sample holder)



Samples of $\text{CrCl}_2 \cdot 216$, NiCl_2 and CuSO_4 solution

Figure 3.2: Holder, Bulb, wires, Analogue micro Ammeter, power supply (2 V-12 V), Cuvette (sample holder) and samples of solutions.

3.1.1. Light source

This homemade visible spectrophotometer uses a power supply (0–12 V) and an EP-10 tungsten lamp (0.45 A, 6 V) as a light source. A tungsten lamp is made of a tungsten filament and halogen gas. A low pressure of inert gas (typically argon) fills the free space inside the glass bulb that surrounds both the tungsten filament and the halogen gas. Tungsten lamps are small, bright, and relatively inexpensive. The light emitted from these bulbs contains the entire visible light spectrum and beyond. Because of its inherent characteristics, tungsten light contends for the title of most versatile light source.

3.1.2. Converging lens

A converging lens, also called a convex or positive lens, is thicker in the middle and thinner around the edge. The parallel light rays that pass through a convex lens converge at their principal focus. Convex lenses are used in a microscope, magnifying glasses, cameras, the correction of hypermetropia, etc. The angle at which the rays of light enter the convex lens depends on the distance of the object from the lens. This homemade visible spectrophotometer uses a convex lens to collimate the light coming from the tungsten light source.

3.1.3. Diffraction grating

A diffraction grating is an optical device that is made of a surface ruled with close, equidistant, and parallel lines for the purpose of resolving light into spectra. A grating can be a transmission mode or reflection mode grating, depending on whether it is transparent or mirrored. Reflection gratings are further classified as plane or concave. The concave reflection grating is a spherical surface ruled with lines that are the projection of equidistant and parallel lines on an imaginary plane surface. The advantage of a concave grating over a plane grating is its ability to produce sharp spectral lines without the aid of lenses or additional mirrors. This makes it useful in the infrared and ultraviolet regions since these radiations would be absorbed on passage through a lens.

A diffraction grating is used to disperse a beam of light of various wavelengths into a spectrum of each individual wavelength. Gratings provide exceptionally high spectral line resolution. The resolving power (R) of an optical instrument is the ability to separate closely spaced lines in a spectrum and is equal to the wavelength λ divided by the small change in wavelength ($\Delta\lambda$)

between two wavelengths that can be detected, i.e., $R = \lambda/\Delta\lambda$. Thus, this homemade visible spectrophotometer uses a transmission diffraction grating (100 lines per millimetre).

3.1.4. Filters

Filters are monochromatic devices that may be made of colored glasses, plastics, gelatin, or sometimes a colored liquid in a glass cell. They are mostly placed after the light source to modify the light. Colored filters allow a single wavelength of light to pass by blocking the unwanted wavelengths. In this thesis, blue, yellow, yellow-green, and red plastic filters are used to calibrate the homemade visible spectrophotometer.

3.1.5. Cuvette and sample holder

The card box was used to make the cuvette holder. It has a width of 1 cm, a length of 1 cm, and a height of 4.5 cm. On both sides of the card box, a small hole was made where the light was incident on and coming out of the sample. The hole on both sides of the sample holder box was used to focus a light beam in a small area (1 cm in diameter) in front of the light sensor (see Figure 3.3).

A cuvette is a small, transparent, rectangular vessel that comes in a variety of materials, quality standards, and dimensions. Cuvettes are designed to hold samples for spectroscopic measurements. A light beam is sent through the cuvette to evaluate its absorbance, transmittance, fluorescence emission, fluorescence polarization, or fluorescence lifespan.

There are mainly two types of cuvettes. One is a two-sided transparent quartz cuvette, and another is a four-sided transparent quartz cuvette. The four-sided one is quite costly compared to the two-sided transparent cuvette. The two-sided cuvette is sealed (not transparent) at one end. It is made of a clear and transparent material, such as plastic, glass, or quartz.

Glass cuvettes are utilized for visible-range measurements ranging from 310 to 2500 nm. In contrast, quartz cuvettes are used for the entire UV and visible spectrum, from 200 to 2500 nm. The width (thickness) of the cuvette defines the length of the light path through the sample, which affects the absorbance value. The path length of cuvettes is used for estimating the absorption coefficient. In this thesis, a two-sided transparent cuvette is used as a sample holder.

It is sealed at one end and made of a clear, transparent material called quartz. This cuvette has a width of 1 cm and transmits light only on its transparent side.

3.1.6. Light Detector

Light detectors are light sensors that are used to simply translate the amount of light incident on them into an electrical signal. They are using it all over the world. Light sensors can be used in a variety of applications, including robots, to detect the current ambient light level, determine how dark or bright an environment is, in traffic lights, and so on.

Since the Light Dependent Resistor (LDR) is one type of light sensor electronic component, this homemade visible spectrophotometer uses the LDR as a light detector. The LDR allows current to pass through it if light is incident on it, and it shows a reverse process if it gets dark. This means it has a high resistance when it is dark and a low resistance when it is exposed to light.

3.1.7. Displayer

The outputs of this homemade visible spectrophotometer are displayed using digital voltmeters (0-20 V) and analogue ammeters (0-500 A).

3.2. Methods

3.2.1. Design a new home-made visible Spectrophotometer

The homemade spectrophotometer was constructed using a standard commercially available setup and resources from a literature review. The instrument was built on a 57 cm by 46 cm Styrofoam base. On the Styrofoam base, four stands were fixed vertically in a straight line in the horizontal direction, with a constant distance gap. The stands are used as holders for materials needed for the design of the homemade visible spectrophotometer. The instrument essentially consists of an electrical power source, the tungsten lamp, the convex lens, the diffraction grating, the filters, the cuvette holder, the LDR detector, and the digital voltmeter and analogue ammeter. This homemade visible spectrophotometer is built using the following steps:

Firstly, the power supply is stationed before the lamp, and the lamp with its holder is put on one of the vertically fixed stands that are next to the power supply. Secondly, the convex lens is

placed on the second vertically fixed stand, which is followed by the third vertically adjusted stand containing the diffraction grating. Thirdly, the filter is placed on the fourth vertically adjusted stand, and a cuvette holder is placed next to the filter. Finally, the LDR detector is put behind the cuvette holder, and the digital voltmeter (in parallel with the LDR) and analogue ammeter (in series with the LDR) are connected to the detector using wires with crocodile clips.

Generally, all components were constructed with easily accessible materials. The whole size of the instrument is 55 cm × 12 cm × 22 cm for length, width, and height, respectively. Figure 3.3 shows a complete schematic illustration of the simply designed visible spectrophotometer.



Figure 3. 3: Overall arrangement of the designed visible spectrophotometer.

3.2.2. Experimental Method and Measurement Procedures

I. Experimental Method

Although designing the instrument and making it work is a quick and easy task, preparing the apparatus for detailed work in a precise manner requires additional time and attention. As a result, the measurements performed on this newly designed instrument are only intended to test the effectiveness of the home-made visible spectrophotometer in measuring the absorbance of solutions at various concentrations and wavelengths. To use the homemade visible spectrophotometer for absorbance measurement, the following measurement methods were used:

Firstly, an EP-10 tungsten lamp (0.45 A, 6 V) was connected to the power supply that was set at 6 V. The lamp produces a broadband light, and this light was directed to the convex lens, which is used to concentrate all the light on a small area. The collimated light was dispersed by the diffraction grating, and this dispersed light was made to pass through the filter. The filter allows passing a single beam of light to focus on the sample. In the sample, a portion of light was absorbed, and the remaining one was transmitted. Then, the transmitted light was detected by the LDR, which was connected to the digital voltage meter and analogue ammeter. The final outputs are voltage and current readings, which are displayed using the voltage meter and ammeter, respectively. These readings were converted to the absorbance and transmittance of the sample analysed.

It is important to note that the LDR has inverse relationships with the transmitted light. So, the transmitted and absorbed light was not read directly from the displayer like a commercial spectrophotometer, but rather it was expressed in the form of a voltage drop on the LDR or the current reading on the ammeter. Here, instead of using the intensity of incident and transmitted light, the output readings of the voltmeter or ammeter are used to calculate the transmittance and absorbance values of the measurements. The voltage and the current produced by the LDR were read using a digital multimeter and an analogue micro-scale ammeter. By a reasonable approximation, the measured voltage is related to the intensity of the transmitted light. So, from the obtained voltage readings, the transmitted and absorbed lights were calculated by using Beer Lambert's law equations of 2.14, 2.15, and 2.16.

The designed visible spectrophotometer is put inside the card box to minimize the interference (the noise) of room light to the sensor measurement. Both the inside and outside of the designed instrument were covered with a thick card box. To increase the spectral resolution, the sensor was put directly behind the crystal glass cuvette cell. Thus, the sensor detects the light only transmitted through the diluted sample of liquid solution. Therefore, when a high concentration of the solution is put into the crystal glass cuvette cell, the sensor detects a low intensity due to the lower transmission of light through the solution.

II. Measurement Procedures

Some experiments have been done to check the measuring capacity of a specially designed visible spectrophotometer. The most effective validation method would have been to compare

the measurement values of the newly designed homemade visible spectrophotometer with the theoretical values and any commercially available UV-Vis spectrophotometer (DR6000). In the experiment, the relationships between absorbance as a function of solution concentration and absorbance as a function of wavelength were investigated. The measurement capacity of the home-made visible spectrophotometer was evaluated by using copper sulphate (CuSO_4), chromium chloride (CrCl_3 and $\text{CrCl}_2 \cdot 2\text{H}_2\text{O}$), and nickel chloride (NiCl_2) solutions in different concentration ranges and wavelengths. Therefore, absorbance is measured at the red filter (680 nm), the blue filter (450 nm), the yellow-green filter (510 nm), and the yellow filter (580 nm), and these absorbance results are plotted as a function of concentrations and wavelengths. All the absorbance measurements were made by using the following measurement procedures:

Firstly, a 100-ml stock solution of 0.32 M CuSO_4 was prepared by diluting 8 g of copper sulphate (CuSO_4) with standard distilled water. Next, the sample solutions were prepared from a stock solution by using the following equation (3.1):

$$C_s V_s = C_w V_w \quad (3.1)$$

Where, C_s is concentration of the stock solution, V_s is required volume of the stock solution, C_w is concentration of the working solution and V_w is volume of the working solution. So, the prepared solutions were 0.04 mol/L, 0.08 mol/L, 0.12 mol/L, 0.16 mol/L, 0.20 mol/L, etc.

Secondly, the blank sample was put in a sample holder, and the displayed voltage and current outputs were recorded. Thirdly, the blank sample was taken out of the sample holder and replaced by the prepared dilute sample solution. The absorbance as a function of concentration was calculated by combining different concentration ranges with a fixed wavelength at which the absorbance is greatest. These procedures were repeated for each concentration range, and the results were also tabulated. To ensure precision in the measurement analysis, each measurement was conducted repeatedly (at least three times), and the average data was taken as the measured result.

Fourthly, the recorded voltage and current readings were then converted to transmittance and absorbance values using Beer Lambert's law mathematical equations of 2.14, 2.15, and 2.16. Fifthly, the absorbance as a function of the wavelength relationship was checked by using measurements collected by interchanging the blue, yellow, yellow-green, and red filters at a constant concentration. These measurements were made with variable wavelengths at constant concentration.

Finally, the graphs of absorbance as a function of concentration and absorbance as a function of wavelength were plotted using Origin software. The graphs were then analysed, and the extinction coefficients (ϵ), R^2 values, and slopes of the calibration graphs were calculated. These graphs were compared with the theoretical measured values and the results obtained from a commercial UV-Vis spectrophotometer. Here, we can use the voltage readings or current readings as alternative outputs of a home-made visible spectrophotometer.

In addition to absorption by the compound, other processes reduce the intensity of light that transmits through the sample; so it is important to take a "background" reading for the solvent and the cell, which corresponds to I_0 the input part of light intensity, the voltmeter does not read out zero volts when no light falls on the detector, and the correction is made by subtracting the voltage at zero light (V_{zero}) from all readings. These two procedures correspond to the "zeroing" processes of the home-made visible spectrophotometer. However, some older needle-type voltmeters may be set to zero manually, which simplifies the mathematical calculations.

Chapter four: Results and Discussion

4.1. Absorbance as a Function of Concentration

The colors of objects are dependent on the light's interaction with the object. Light can be reflected, transmitted, absorbed, or exhibit any other property when it interacts with matter. This is due to the ability of the compounds or electrons in objects to transmit, absorb, and reflect specific wavelengths of light. In other words, the color of light observed is the one that is not absorbed. The wavelengths of light that are absorbed can be determined by the electrons in a compound [41]. As electrons move from a lower energy level to a higher energy level in an atom, they can absorb energy and become excited. The amount of energy or the wavelength of light that the electrons absorb is determined by the type of atoms found in the compound and how those atoms are bound together. Various types of environments for electrons will also determine how much of a particular wavelength of light can be absorbed, which is reflected in the molar absorptivity of the compound [41]. Since the color of an object is due to the ability of its constituent atoms to absorb light, its color should become more intense as the concentration of electrons in the sample increases. The increase in concentration leads to more electrons can interact with light in the sample which can then absorb more light at a particular wavelength. Thus, there should be a relationship between the concentration of the sample being studied and its absorbance. This relationship is best determined using a wavelength of light in a region of the visible spectrum of light where the maximum absorbance is seen. This wavelength is most sensitive to concentration changes. The purpose of this experiment is to test the measuring ability of the new home-made visible spectrophotometer by using solutions of CuSO_4 , CrCl_3 , $\text{CrCl}_2 \cdot 2\text{H}_2\text{O}$, and NiCl_2 with different concentration ranges.

4.1.1. Copper Sulphate (CuSO₄) samples

Since copper compounds tend to be blue in color [41], it is proposed that CuSO₄ would have a maximum absorbance at a longer wavelength, which corresponds to a color more in the range of red. Once the best wavelength, at which maximum absorbance occur, for studying CuSO₄ is determined, it can be used to examine the relationship between the absorbance and concentration of the sample and use that relationship to calculate desired information for unknown sample solutions. As a result, the performance of the newly designed home-made visible spectrophotometer was evaluated using various concentrations of copper sulfate (CuSO₄) solutions. Tables 4.1, 4.2, 4.3, and 4.4 show the measured results of the calculated absorbance and transmitted values at different concentration ranges of copper sulfate (CuSO₄) solution using voltage outputs. Tables 4.5, 4.6, 4.7, and 4.8 show the measured results of the calculated absorbance and transmitted values at different concentration ranges of copper sulfate (CuSO₄) solution using current output. The measured results are collected by using the new home-made visible spectrophotometer, which interprets the output signals in terms of voltage ($V_{solvent}$ and V_{sample}) or current ($I_{solvent}$ and I_{sample}) from the detector.

I. Plotted graphs of absorbance and transmittance as a function of concentrations using voltage output

Table 4. 1: Transmittance and Absorbance data for a stock solution of 0.32 M CuSO₄ using red filter (at 650 nm).

C [mol/L]	$V_{solvent}$ [V]	V_{zero} [V]	V_{sample} [V]	T [arb. units]	1/T [arb. units]	A [arb. units]
0			3.19	1	1	0
0.08			3.21	1.0068	0.9933	0.0029
0.16	3.19	0.24	3.23	1.0136	0.9866	0.0058
0.24			3.24	1.0169	0.9833	0.0073
0.32			3.25	1.0203	0.9801	0.0087

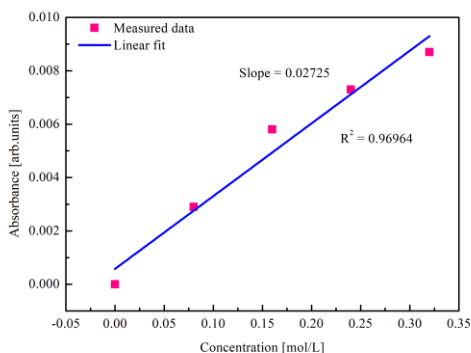


Figure 4. 1: Concentration versus absorbance of a stock solution of 0.32 M CuSO₄ using voltage output with a red filter (at about 650 nm).

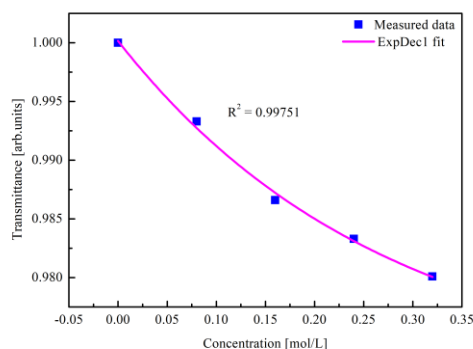


Figure 4. 2: Concentration versus Transmittance graph for 0.32 M CuSO₄ solution using voltage output with a red filter (at 650 nm).

According to Beer's law, the absorbance equation has the same form as the straight line equation, i.e.

$$y = m x + b \quad \text{has the same form with} \quad A = k c$$

Where, 'm' is the slope which is equal to the absorbance constant k, 'b' is the y-intercept and 'x' represents the concentration of the sample solution [33,37]. So, the above 4.1 graph has the slope of 0.02725 ± 0.00278 with y-intercept of $5.8 \text{ E-}4 \pm 5.4552$. Its R^2 value is 0.96964 which indicates the precision level of the measured values. When x is equal to zero, we will get the y intercept. From the above straight line equation and absorbance equation relationship, zero concentration means that making the value of x zero. From this, we can understand that the y intercept tells the absorbance level of the solvent. If the graph passes through (0, 0), the absorbance level of the solvent is zero. This means that the solvent transmits all the light passing through it. As a result, the y intercept shows the absorbance level of the solvent (blank) we used.

Table 4. 2: Transmittance and Absorbance data for a stock solution of 0.32 M CuSO₄ using a red filter (at about 650 nm).

C [mol/L]	V _{solvent} [V]	V _{sample} [V]	V _{zero} [V]	T [arb. units]	1/T [arb. units]	A [arb. units]
0.00		3.19		1	1	0
0.04		3.20		1.0034	0.9966	0.0015
0.08		3.21		1.0068	0.9932	0.0029
0.12		3.22		1.0102	0.9899	0.0044
0.16	3.19	3.24	0.24	1.0169	0.9834	0.0073
0.20		3.25		1.0203	0.9801	0.0087
0.24		3.26		1.0237	0.9768	0.0102
0.28		3.27		1.0271	0.9736	0.0116
0.32		3.27		1.0271	0.9736	0.0116

From the relationship, $y = m x + b$ has the same form with $A = k c$, the slope of figure 4.3 is 0.03983 with uncertainty value of ± 0.00227 and the y-intercept is $9.33333 \text{ E-5} \pm 4.33204 \text{ E-4}$. Its R^2 value is 0.97768 which means that the measurement is precise with an amount of 97.768%.

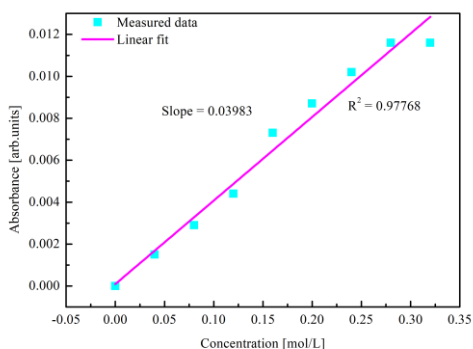


Figure 4. 3: Concentration versus absorbance of a stock solution of 0.32 M CuSO₄ using voltage output with a red filter (at about 650 nm).

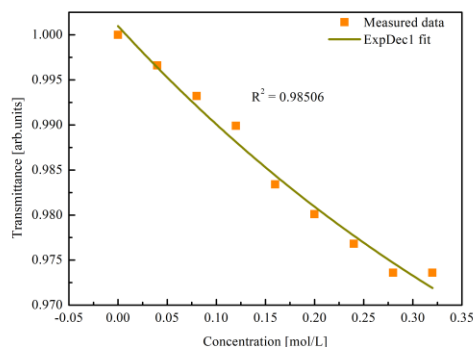


Figure 4. 4: Concentration versus Transmittance graph of 0.32 M CuSO₄ using voltage output with a red filter (at about 650 nm).

Table 4. 3: Transmittance and Absorbance data for a stock solution of 0.16 M CuSO₄ using a blue filter (at about 450 nm).

C [mol/L]	V _{sample} [V]	V _{solvent} [V]	V _{zero} [V]	T [arb.units]	1/T [arb.units]	A [arb.units]
0	3.34			1	1	0
0.05	3.38			1.0129	0.9873	0.0056
0.07	3.41	3.34	0.24	1.0226	0.9779	0.0097
0.09	3.42			1.0258	0.9748	0.0111
0.10	3.42			1.0258	0.9748	0.0111

From the relationship, $y = m x + b$ has the same form with $A = k c$, the slope of figure 4.5 is 0.11831 with uncertainty value of ± 0.012 and the y-intercept is $1.6465 \text{ E-}4 \pm 8.57153 \text{ E-}4$. Its R^2 value is 0.97005 which means that the measurement is precise with an amount of 97.005%.

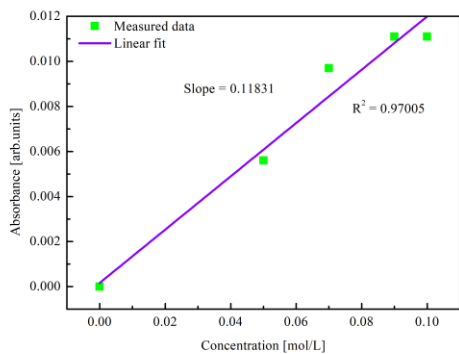


Figure 4. 5: Concentration versus absorbance of a stock solution of 0.16 M CuSO₄ using voltage output with a blue filter (at about 450 nm).

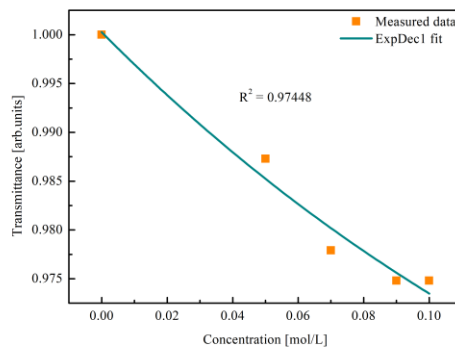


Figure 4. 6: Concentration versus Transmittance graph of 0.16 M CuSO₄ using voltage output with a blue filter (at about 450 nm).

Table 4. 4: Transmittance and Absorbance data for a stock solution of 0.16 M CuSO₄ using red filter (at about 650 nm).

C [mol/L]	V _{sample} [V]	V _{solvent} [V]	V _{zero} [V]	T [arb.units]	1/T [arb.units]	A [arb.units]
0	3.23			1	1	0
0.05	3.29			1.0201	0.9803	0.0086
0.07	3.30	3.23	0.24	1.0234	0.9771	0.0101
0.09	3.31			1.0268	0.9739	0.0115
0.10	3.32			1.0301	0.9708	0.0129

From the relationship, $y = m x + b$ has the same form with $A = k c$, the slope of figure 4.7 is 0.12576 with uncertainty value of ± 0.01416 and the y-intercept is $8.22611 \text{ E-}4 \pm 0.00101$. Its R^2 value is 0.96335 which means that the measurement is precise with an amount of 96.335%.

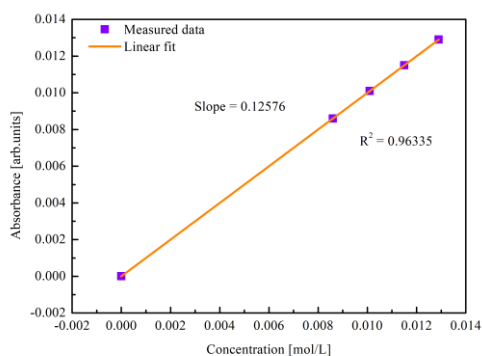


Figure 4. 7: Concentration versus absorbance of a stock solution of 0.16 M CuSO₄ using voltage output with a red filter (at about 650 nm).

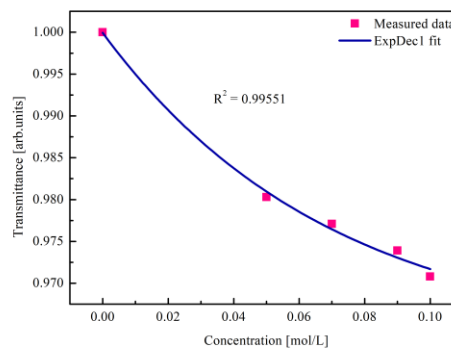


Figure 4. 8: Concentration versus Transmittance graph of 0.16 M CuSO₄ using voltage output with a red filter (at about 650 nm).

From the relationship, $y = m x + b$ has the same form with $A = k c$, the slope of figure 4.9 is 1.325 with uncertainty value of ± 0.11902 and the y-intercept is 0.006 ± 0.02332 . Its R^2 value is 0.97636 which means that the measurement is precise with an amount of 97.636%.

II. Plotted graphs of absorbance as a function of concentrations using current output

Table 4. 5: Transmittance and Absorbance data for a stock solution of 0.32 M CuSO₄ using red filter (at 650 nm).

C [mol/L]	I _{solvent} [μA]	I _{sample} [μA]	T [arb. Units]	A [arb. Units]
0		360	1	0
0.08		290	0.81	0.09
0.16	360	200	0.56	0.26
0.24		170	0.47	0.33
0.32		140	0.39	0.41

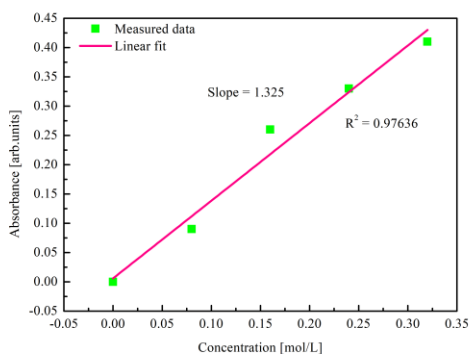


Figure 4. 9: Concentration versus absorbance of 0.32 M CuSO₄ using current output with a red filter (at about 650 nm).

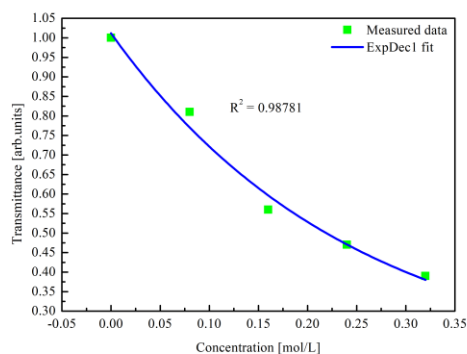


Figure 4. 10: Concentration versus Transmittance graph for 0.32 M CuSO₄ solution using current output with a red filter (at 650 nm).

Table 4. 6: Transmittance and Absorbance data for a stock solution of 0.32 M CuSO₄ using a red filter (at about 650 nm).

C [mol/L]	I _{solvent} [μA]	I _{sample} [μA]	T [arb. Units]	A [arb. Units]
0.00		360	1	0
0.04		275	0.76	0.12
0.08		225	0.63	0.20
0.12		215	0.60	0.22
0.16	360	195	0.54	0.27
0.20		175	0.49	0.31
0.24		170	0.47	0.33
0.28		150	0.42	0.38
0.32		130	0.36	0.44

From the relationship, $y = m x + b$ has the same form with $A = k c$, the slope of figure 4.11 is 1.20417 with uncertainty value of ± 0.10173 and the y-intercept is 0.05956 ± 0.01937 . Its R^2 value is 0.95242 which means that the measurement is precise with an amount of 95.242%.

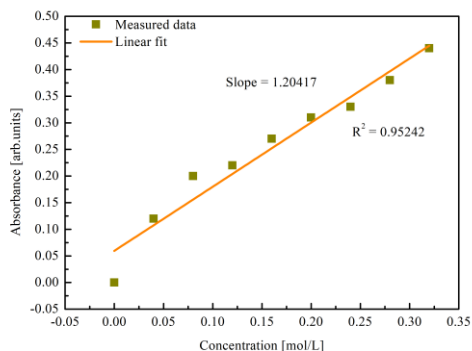


Figure 4. 11: Concentration versus absorbance of a stock solution of 0.32 M CuSO₄ using current output with a red filter (at about 650 nm).

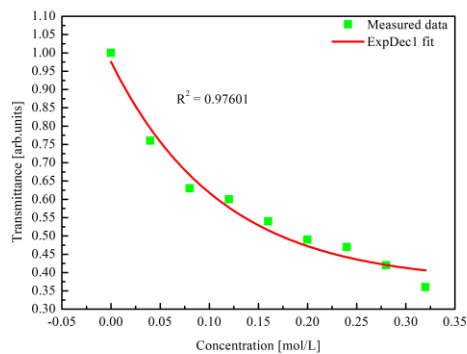


Figure 4. 12: Concentration versus Transmittance graph of 0.32 M CuSO₄ using current output with a red filter (at about 650 nm).

III. Comparing absorbance using current output with different color filters

A. For blue filter

Table 4. 7: Transmittance and Absorbance data for a stock solution of 0.16 M CuSO₄ using red filter (at about 450 nm).

C [mol/L]	I _{sample} [μA]	I _{solvent} [μA]	T [arb. Units]	A [arb. Units]
0	60		1	0
0.05	30		0.5	0.30
0.07	25	60	0.42	0.38
0.09	20		0.33	0.48
0.10	15		0.25	0.60

From the relationship, $y = m x + b$ has the same form with $A = k c$, the slope of figure 4.13 is 5.68153 with uncertainty value of ± 0.36884 and the y-intercept is $-2.54777 \text{ E-4} \pm 0.02634$. Its R^2 value is 0.98751 which means that the measurement is precise with an amount of 98.751%.

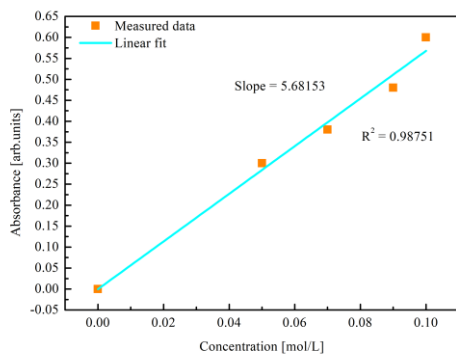


Figure 4. 13: Transmittance and Absorbance data for a stock solution of 0.16 M CuSO₄ using current with a blue filter (at about 450 nm).

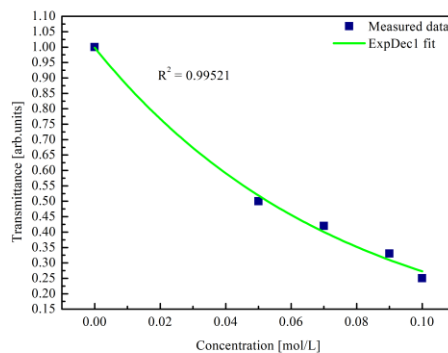


Figure 4. 14: Concentration versus Transmittance graph of 0.16 M CuSO₄ using current output with a blue filter (at about 450 nm).

B. Using red filter

Table 4. 8: Transmittance and Absorbance data for a stock solution of 0.16 M CuSO₄ using a red filter (at about 650 nm).

C [mol/L]	I _{sample} [μA]	I _{solvent} [μA]	T [arb. Units]	A [arb. Units]
0	240		1	0
0.05	115		0.48	0.32
0.07	95	240	0.40	0.40
0.09	90		0.38	0.42
0.10	80		0.33	0.48

From the relationship, $y = m x + b$ has the same form with $A = k c$, the slope of figure 4.15 is 4.67516 with uncertainty value of ± 0.61204 and the y-intercept is 0.03414 ± 0.04371 . Its R^2 value is 0.9511 which means that the measurement is precise with an amount of 95.11%.

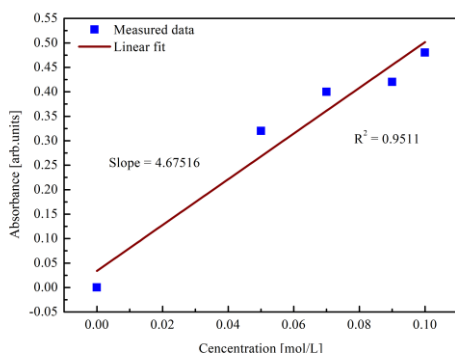


Figure 4. 15: Transmittance and Absorbance data for a stock solution of 0.16 M CuSO₄ using current with a red filter (at about 650 nm).

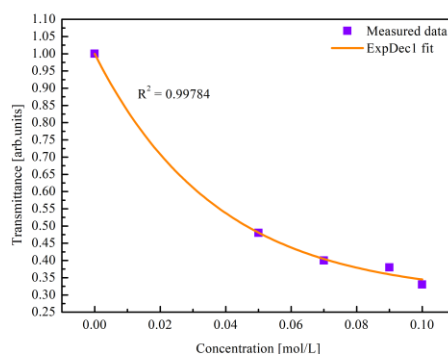


Figure 4. 16: Concentration versus Transmittance graph of 0.16 M CuSO₄ using current output with a red filter (at about 650 nm).

IV. Comparison of absorbance measurement of homemade visible spectrophotometer with commercial UV-Vis spectrophotometer

The following graph shows the commercial UV-Vis spectrophotometer (DR 6000) absorbance measurement of CuSO₄ solutions with a concentration of 0.08mol/L, 0.16mol/L, 0.24mol/L and

0.32 mol/L. As it is shown in the figure, the absorbance is increased with the increase of the concentration of sample solutions around red spectrum of light (at about 650 nm). This means that at the region of red spectrum of light, there is a relative absorbance peak. When this UV-Vis absorbance graph is compared with the plotted graphs of the measured data using home-made spectrophotometer, the concentration versus absorbance relationships are almost similar. In both the home-made visible spectrophotometer and UV-Vis spectrophotometer, the absorbance is increased as the concentration of the sample solutions is increased.

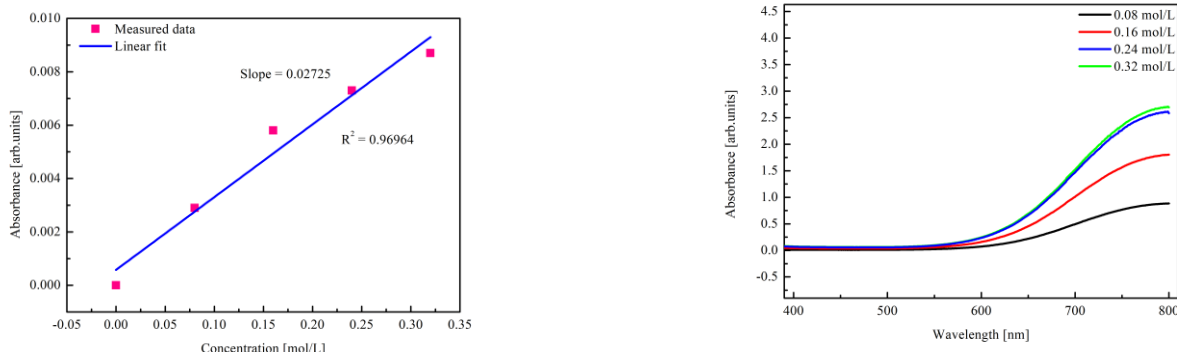


Figure 4. 17: Measurement of 0.32 M CuSO₄ solutions of absorbance versus wavelength using commercial UV-Vis Spectrophotometer.

The above graphs of absorbance versus concentration values in figure 4.1, 4.3, 4.5, 4.7, 4.9, 4.11, 4.13 and 4.15 show linear relationship giving slopes of 0.02725 ± 0.00278 , 0.03983 ± 0.00227 , 0.11831 ± 0.012 , 0.12576 ± 0.01416 , 1.325 ± 0.11902 , 1.20417 ± 0.10173 , 5.68153 ± 0.36884 and 4.67516 ± 0.61204 along with their corresponding intercepts of $5.8 \text{ E-}4 \pm 5.4552$, $9.33333 \text{ E-}5 \pm 4.33204 \text{ E-}4$, $1.6465\text{E-}4 \pm 8.57153 \text{ E-}4$, $8.22611\text{E-}4 \pm 0.00101$, 0.006 ± 0.02332 , 0.05956 ± 0.01937 and 0.03414 ± 0.04371 respectively. The slope represents the value of ϵl product or 'k' value [33,37], from where ϵ (molar absorptivity) is calculated considering the value of path length, $l = 1$ cm. Since the path length of the light is 1 cm, the calculated molar absorptivity (ϵ) values for each concentrations of copper sulphate (CuSO₄) solution will be each corresponding slopes respectively.

For the ideal case, the R-squared (R^2) value is 1 for the measured data which fits exactly. The curve fitting for the calibration of figure 4.1, 4.3, 4.5, 4.7, 4.9, 4.11, 4.13 and 4.15 give approximate linear model with their R-squared (R^2) values of 0.96964, 0.97768, 0.97005, 0.96335, 0.97636, 0.9524, 0.98751 and 0.9511 respectively.

Figure 4.1, 4.3, 4.5 and 4.7 in the above are plotted by using output voltage signals whereas figure 4.9, 4.11, 4.13 and 4.15 is plotted using current outputs in the same sample concentration CuSO_4 . When both the voltage and current output graphs are compared, the linear curve fitting for the calibration of figures with voltage outputs is almost similar to the linear fitting of figures with current outputs. They have approximate linear model with small difference. This small difference might be due to the measuring scale difference of the Voltmeter (with 0 - 20 volt scale) and Ammeter (with 0 – 500 micro ampere scale) or any other experimental factors. Plus to this the voltmeter is digital whereas the Ammeter is analogue. This can also be the factor which brings small reading differences between Ammeter and Voltmeter. Having the small measurement variations, it is possible to say that both Ammeter and voltmeter can be used as a displayer unit for a home-made visible spectrophotometer.

On the other hand, the measuring performance of the newly designed visible spectrophotometer is also compared with the commercial UV-Vis spectrophotometer measurement using the same sample concentration of CuSO_4 . Their comparative relationship is observed in the above current or voltage output graphs and the commercial UV-Vis spectrophotometer (DR6000) graph of figure 4.17. In all cases, the absorbance versus concentration relationship is precise with minor measurement errors since their R^2 value is closely approaches the theoretical R^2 value.

4.1.2. Chromium Chloride (CrCl₃) sample

I. Absorbance as a function of concentration using voltage output

Table 4. 9: Transmittance and Absorbance data for a stock solution of 0.075 M CrCl₃ using yellow filter (at about 580 nm).

C [mol/L]	V _{sample} [V]	V _{solvent} [V]	V _{zero} [V]	T [arb. Units]	1/T [arb. Units]	A [arb. Units]
0	3.18			1	1	0
0.021	3.21			1.0102	0.9899	0.0044
0.033	3.23	3.18	0.24	1.0170	0.9833	0.0073
0.041	3.24			1.0204	0.9800	0.0088
0.046	3.25			1.0238	0.9768	0.0102
0.050	3.25			1.0238	0.9768	0.0102

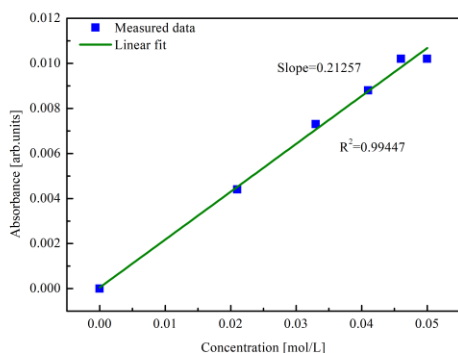


Figure 4. 18: Transmittance and Absorbance data for a stock solution of 0.075 M CrCl₃ using voltage output with yellow filter (at about 580 nm).

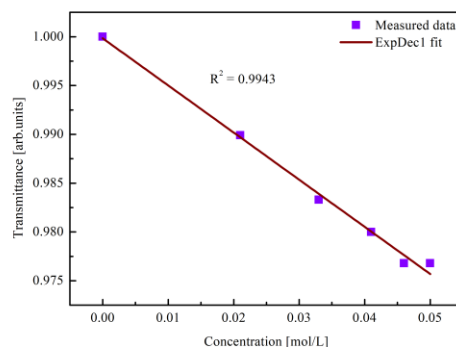


Figure 4. 19: Concentration versus Transmittance graph of 0.075 M CrCl₃ using voltage output with yellow filter (at about 580 nm).

From the relationship, $y = m x + b$ has the same form with $A = k c$, the slope of figure 4.18 is 0.21257 with uncertainty value of ± 0.00793 and the y-intercept is $4.99952 \text{ E-5} \pm 2.86345$. Its R^2 value is 0.99447 which means that the measurement is precise with an amount of 99.447%.

Table 4. 10: Transmittance and Absorbance data for a stock solution of 0.075 M CrCl₃ using yellow-green filter (at about 510 nm).

C [mol/L]	V _{sample} [V]	V _{solvent} [V]	V _{zero} [V]	T [arb.units]	1/T [arb.units]	A [arb.units]
0	3.22			1	1	0
0.021	3.28			1.0201	0.9803	0.0086
0.033	3.30	3.22	0.24	1.0235	0.9770	0.0101
0.041	3.31			1.0302	0.9707	0.0129
0.046	3.32			1.0336	0.9675	0.0143
0.050	3.32			1.0336	0.9675	0.0143

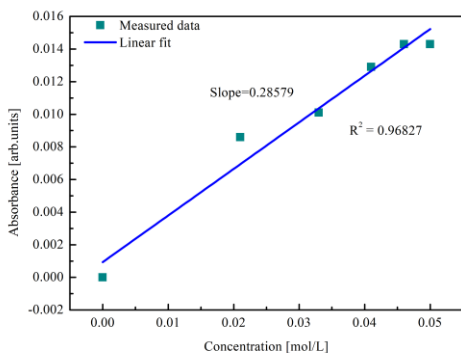


Figure 4. 20: Transmittance and Absorbance data for 0.075 M CrCl₃ using voltage output with yellow-green filter (at about 510 nm).

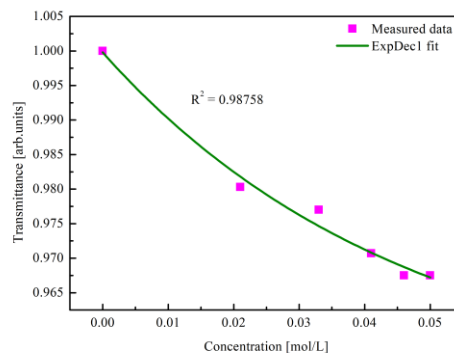


Figure 4. 21: Concentration versus Transmittance graph of 0.075 M CrCl₃ using voltage output with yellow-green filter (at about 510 nm).

From the relationship, $y = m x + b$ has the same form with $A = k c$, the slope of figure 4.20 is 0.28579 with uncertainty value of ± 0.02587 and the y-intercept is $9.35579 \text{ E-4} \pm 9.34314 \text{ E-4}$. Its R^2 value is 0.96827 which means that the measurement is precise with an amount of 96.827%.

II. Absorbance as a function of concentration using current output

Table 4. 11: Transmittance and Absorbance data for a stock solution of 0.075 M CrCl₃ using yellow filter (at about 580 nm).

C [mol/L]	I _{sample} [μA]	I _{solvent} [μA]	T [arb. Units]	A [arb. Units]
0	380		1	0
0.021	295		0.78	0.11
0.033	225	380	0.59	0.23
0.041	195		0.51	0.29
0.046	190		0.5	0.30
0.050	185		0.49	0.31

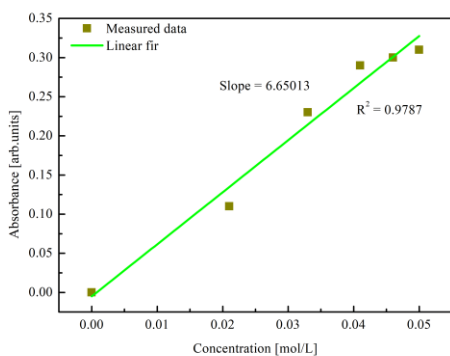


Figure 4. 22: Transmittance and Absorbance data for a stock solution of 0.075 M CrCl₃ using current output with yellow filter (at about 580 nm).

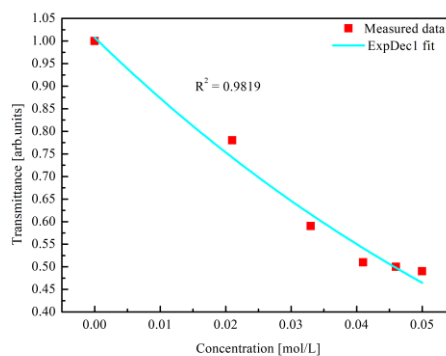


Figure 4. 23: Concentration versus Transmittance graph of 0.075 M CrCl₃ using current output with yellow filter (at about 580 nm).

From the relationship, $y = m x + b$ has the same form with $A = k c$, the slope of figure 4.22 is 6.65013 with uncertainty value of ± 0.49049 and the y-intercept is -0.00503 ± 0.01772 . Its R^2 value is 0.9787 which means that the measurement is precise with an amount of 97.87%.

Table 4. 12: Transmittance and Absorbance data for a stock solution of 0.075 M CrCl₃ using yellow-green filter (at about 510 nm).

C [mol/L]	I _{sample} [μA]	I _{solvent} [μA]	T [arb. Units]	A [arb. Units]
0	260		1	0
0.021	155		0.60	0.22
0.033	140	260	0.54	0.27
0.041	125		0.48	0.32
0.046	120		0.46	0.34
0.050	120		0.46	0.34

From the relationship, $y = m x + b$ has the same form with $A = k c$, the slope of figure 4.24 is 6.78847 with uncertainty value of ± 0.79204 and the y-intercept is 0.03223 ± 0.02861 . Its R^2 value is 0.94836 which means that the measurement is precise with an amount of 94.836%.

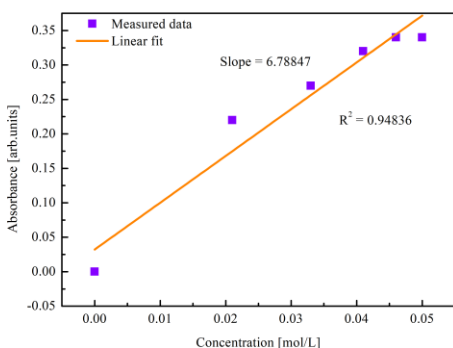


Figure 4. 24: Transmittance and Absorbance data for a stock solution of 0.075 M CrCl₃ using current output with yellow-green filter (at about 510 nm).

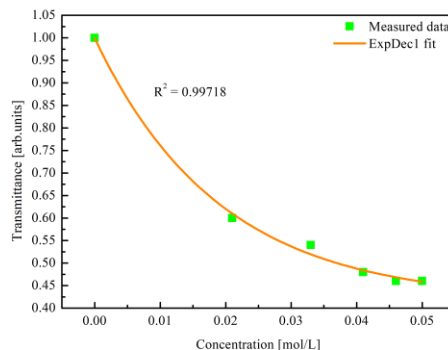


Figure 4. 25: Concentration versus Transmittance graph of 0.075 M CrCl₃ using current output with yellow-green filter (at about 510 nm).

According to Beer's Law, the slope of the straight line graph in which absorbance is plotted against the concentration of solution samples will give a constant value k [33,37]. The equations of Beer's law, $A = k c$ has the same form as the equation for a straight line, $y = m x + b$. A comparison of these equations indicates that $y = A$, $x = c$. Where, m is the slope k and b is an intercept on y-axis which is 0 for this case. Consequently, a straight line graph is obtained

when the absorbance graph is plotted against those concentrations. Moreover, the slope of that line will be given by k , and the line must pass through the origin ($A = 0, c = 0$), because the intercept is zero.

The slope is obtained by a method called linear regression. A straight line passing through the origin was drawn in an attempt to provide the best fit for all the data. Experimental error is the reason why some of the points deviate from that line. Any arbitrary point on the line will provide enough information for us to calculate the slope k . Before measuring absorbance, however, obtain the correct wavelength at which maximum absorbance occurs. This will allow maximum sensitivity for each measurement.

4.2. Transmittance as a function of concentration

4.2.1. Copper Sulphate (CuSO₄) samples

The transmitted light of single wavelength is directly proportional with the current output and inversely proportional with the voltage output [40]. In this thesis, when we use the current output to calculate the transmitted data, we can use directly equation (3.3) but if we use the voltage output we should inverse each transmitted data that are calculated using equation (3.3). This is due to the LDR detector which detects the transmitted light as a voltage reading inversely. According to Beer's law, the transmittance versus concentrations graphs of a sample has an exponential decay relationship. By keeping these rule, this newly designed home-made spectrophotometer can also be tested for transmittance versus concentration variations of a given copper sulphate (CuSO₄) sample solutions. So, based on the transmittance and concentrations data of table 4.1, 4.2, 4.3, 4.5, 4.6, 4.7 and 4.8 in the above, the transmittance versus concentration graphs are plotted respectively.

The above transmittance versus concentration graphs in figures 4.2, 4.4, 4.6, 4.8, 4.10, 4.12, 4.14 and 4.16 illustrate an exponential decay relationship which supports the proposal of Beer's law [41]. All R-square (R^2) value of the graphs shows that the transmittance measurements made in this homemade visible spectrophotometer are in close agreement with the theoretical R-square values which confirms the precise measurement capacity of the home-made visible spectrophotometer.

4.2.2. Chromium Chloride (CrCl₃) sample

Here, the shape of the graph deviates from exponential decay relationship of transmittance as a function of concentration. It shows some errors. This might be due to the experimental errors.

Figure 4.19, figure 4.21, figure 4.23 and figure 4.25 show the exponential decay relationship of transmittance as a function of concentration of chromium chloride (CrCl₃) solution sample. Figure 4.19 and 4.21 are based on voltage outputs whereas figure 4.23 and figure 4.25 are based on the current outputs. As shown in the figure above, both current and voltage output graphs are similar with small variations.

4.3. Absorbance as a function of Wavelength

4.3.1. Copper Sulphate (CuSO₄) sample

Table 4. 13: Transmittance and Absorbance versus wavelength for a 0.18 M of CuSO₄ solution.

Wavelength [nm]	V _{solvent} [V]	V _{sample} [V]	V ₀ [V]	T [arb.units]	A [arb.units]
Violet[400]	3.30	3.35		1.0168	0.0073
Blue[450]	3.33	3.39		1.0200	0.0086
Green[550]	3.27	3.28		1.0034	0.0015
Yellow[580]	3.19	3.22	0.33	1.0105	0.0045
Orange[600]	3.21	3.24		1.0104	0.0045
Red[650]	3.22	3.29		1.0242	0.0104

Figures of 4.26 and 4.27 show the newly designed visible spectrophotometer absorbance versus wavelength (in the visible range of light) relationship of a 0.18 mol/L copper sulfate (CuSO₄) solution. In both graphs, some peaks were detected using both outputs (voltage output or current output) result graph. A sharp peak observed between violet and blue color indicates a strong absorption of the solution and a weak transmittance of a solution at the indicated color. The low peak (valley) around the green color indicates a weak absorbance or a strong transmittance around the indicated color (wavelength).

Table 4. 14: Transmittance and Absorbance versus wavelength for a 0.18 M of CuSO₄ solution.

Wavelength [nm]	I _{solvent} [μA]	I _{sample} [μA]	T [arb.units]	A [arb.units]
Violet[400]	60	25	0.42	0.38
Blue[450]	40	15	0.38	0.43
Green[550]	85	60	0.71	0.15
Yellow[580]	240	150	0.63	0.20
Orange[600]	200	100	0.50	0.30
Red[650]	150	60	0.40	0.40

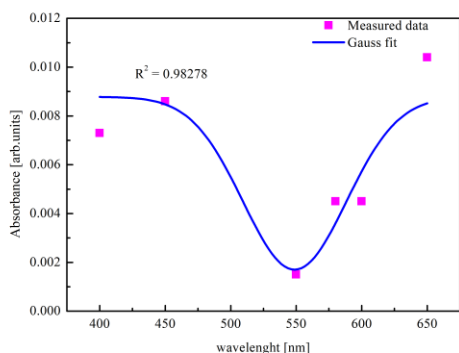


Figure 4. 26: Absorbance versus wavelength graph using current output for 0.18 mol/L of CuSO₄ solution with measured data of homemade visible spectrophotometer.

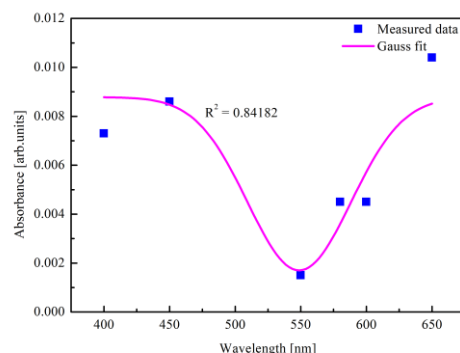


Figure 4. 27: Absorbance versus wavelength graph using voltage output for 0.18 mol/L CuSO₄ solution with measured data of homemade visible spectrophotometer.

Both of the above graphs, plotted based on voltage and current output, are almost similar. From this, it is possible to say Ammeter and Voltmeter can be used interchangeably as a displayer unit for the home-made spectrophotometer.

Generally, the absorbance measuring capacity of the newly designed visible spectrophotometer has a close agreement with the theoretical and the commercial UV-Vis spectrophotometers [40].

This low cost newly designed visible light spectrophotometric system has a capability of measuring transmittance and absorbance of samples at a given wavelengths. Hence, it is validated without having access to any sophisticated laboratory set-up.

4.3.2. Chromium Chloride ($\text{CrCl}_2 \cdot 216$) sample

Table 4. 15: Transmittance and Absorbance versus wavelength for a $\text{CrCl}_2 \cdot 216$ solution.

Wavelengths [nm]	$I_{\text{sample}}[\mu\text{A}]$	$I_{\text{solvent}}[\mu\text{A}]$	T [arb.units]	A [arb.units]
Violet [400]	10	90	0.11	0.96
Blue [450]	5	60	0.08	1.10
Cyan [500]	15	100	0.15	0.82
Green [550]	30	140	0.21	0.68
Yellow [580]	95	380	0.25	0.60
Orange [600]	70	310	0.23	0.64
Red [650]	50	240	0.21	0.68

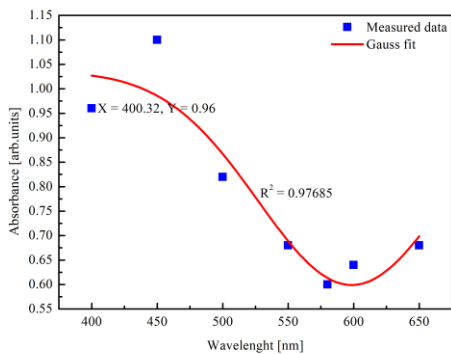


Figure 4. 28: Absorbance versus wavelength graph for $\text{CrCl}_2 \cdot 216$ solution using current output with the measured data of homemade visible spectrophotometer.

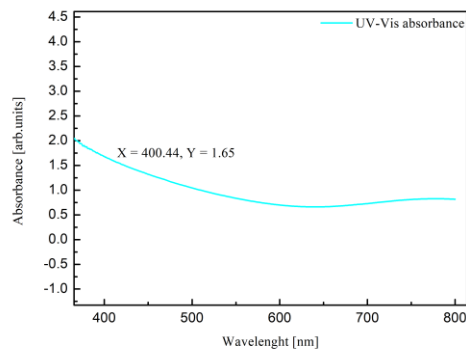


Figure 4. 29: Absorbance versus wavelength graph for $\text{CrCl}_2 \cdot 216$ solution using commercial UV-Vis spectrophotometer.

The above figures of 4.28 and 4.29 are the absorbance as a function of wavelength graphs of $\text{CrCl}_2 \cdot 216$ solution sample using home-made visible spectrophotometer and commercial UV-Vis spectrophotometer (DR600) respectively [40]. These two graphs have similar shape with each other although each absorbance values at a specific wavelength measured by a commercial UV-Vis spectrophotometer is greater than each corresponding absorbance values at a similar wavelength measured by the home-made visible spectrophotometer as it is observed from the

graphs above. This is due to the lack of amplifier in the output part of the home-made visible spectrophotometer. With such small absorbance value differences, the shape of the graph of absorbance of home-made visible spectrophotometer is similar with the commercial one.

4.3.3. Nickel Chloride (NiCl₂) sample

Table 4. 16: Transmittance and Absorbance versus wavelength for a NiCl₂ solution.

Wavelengths [nm]	I _{sample} [μ A]	I _{solvent} [μ A]	T [arb.units]	A [arb.units]
Violet [400]	50	90	0.56	0.26
Blue [450]	40	60	0.67	0.18
Cyan [500]	70	100	0.70	0.15
Green [550]	105	140	0.75	0.12
Yellow [580]	245	380	0.64	0.19
Orange [600]	175	310	0.56	0.25
Red [650]	120	240	0.50	0.30

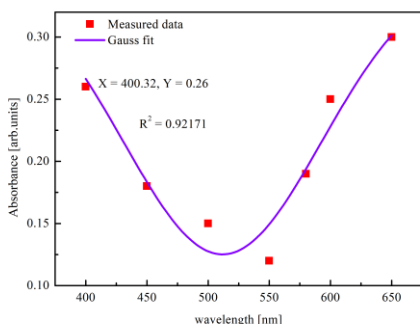


Figure 4. 31: Absorbance versus wavelength graph for NiCl₂ solution using current output with the measured data of home-made visible spectrophotometer.

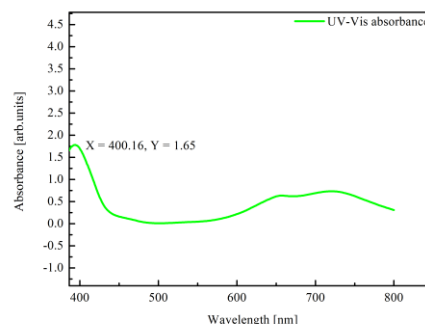


Figure 4. 30: Absorbance versus wavelength graph for NiCl₂ solution using commercial UV-VIS spectrophotometer.

A similar result is observed in the figures of 4.30 and 4.31 of absorbance versus wavelength graphs of a NiCl₂ solution samples. Figure 4.30 and 4.31 are graphs using home-made visible spectrophotometer and commercial UV-Vis spectrophotometer (DR6000) respectively [40]. They show a similar absorbance graph shape with small absorbance value differences.

Chapter Five: Conclusion and Outlook

5.1. Conclusion

UV-VIS Spectrophotometer is very powerful to determine materials absorption. It is an important tool to support research and teaching learning activities. However, commercial UV-Vis spectrophotometers are very expensive and limited in most developing countries science labs. Therefore, the homemade spectrophotometer was designed to alleviate such laboratory problems. The designed instrument has a tungsten white light bulb source, light dependent resistor (LDR) as a sensor, a diffraction grating for light dispersion, a converging lens to collimate the light, filters of different wavelengths, digital voltmeter and ammeter as a detection element. Then, the absorption at different filters was measured on the Copper Sulphate (CuSO_4), Chromium Chloride ($\text{CrCl}_2 \cdot 2\text{H}_2\text{O}$), and Nickel Chloride (NiCl_2) colorful samples. The result found had an R-squared value at the order of 98%. Finally, the comparison of the measured value of the absorption of these samples of the home made and commercial DR6000 UV-Vis were made. Spectrometer was designed from the available resources in the Department. The instruments had consistent absorbance values. Therefore, scaling up the homemade spectrophotometer highly helps in using in our Department and some other similar labs.

5.2. Outlooks

The range and the capacity of the home made spectrophotometer will be improved using proper diffraction grating/prism and CCD. Besides, the software should be developing for proper utilization of UV-VIS spectrophotometer. We believe that in the extension of this thesis project, these will be addressed to commercialize the home made spectrophotometer.

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