

Low-cost colorimetric setup for concentration measurement of manganese ions based on optical absorbance

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ABSTRACT

This study presents a cost-effective setup for measuring the concentration of Mn^{2+} ions using colorimetry. The current method involves a calibration curve created with expensive and large commercial laboratory-based instruments, limiting its use in financially constrained situations. To address this issue, the study proposes a low-cost setup consisting of a light-emitting diode and photodiode that utilizes colorimetric and absorbance effects for Mn^{2+} concentration measurement. Mn^{2+} colorimetric samples were prepared using the 1-(2-pyridylazo)-2-naphthol (PAN) method with concentrations ranging from 0.2 to 1.0 mg/L. The samples were tested using the proposed setup, followed by a spectrophotometry test to determine the optimal configuration for the setup. The validity of the setup was confirmed by measuring the voltage and calculating the optical absorbance, which exhibited a good correlation with the concentration, consistent with the initial expectation. The correlation coefficient for voltage and absorbance against Mn^{2+} concentration was found to be 0.9976 and 0.9987, respectively, indicating good linearity and suitability as a calibration curve for Mn^{2+} detection and measurement. Consequently, the study's objectives were successfully achieved, and the proposed setup is considered a viable platform for more complex applications, such as real-time monitoring activities.

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1. INTRODUCTION

In analytical chemistry, concentration measurement of heavy metal ions is usually performed using a standard laboratory instrument such as ultraviolet-visible spectroscopy (UV VIS), inductively coupled plasma atomic emission spectroscopy (ICP OES) and atomic absorption spectroscopy (AAS) [1], [2]. The instrument works based on the changing properties of light (i.e., absorbance) at different concentration of heavy metal ions (HMI) solution. Under colorimetric effect, the solution containing HMI reacts with specific reagents and produces a change of color that reflects different concentration of analyte. Based on the principles that lights with different wavelength have different absorption towards different color of solution, the amount of analyte being measured can be quantified [3]. The use of the above-mentioned instruments are popular in various field of water quality assessment to detect a variety of water quality parameter [4], [5], which includes heavy metal ions (HMI) detection in water industries [6]–[9]. Apparently, one of the crucial heavy metal ions is manganese,

normally exists as Mn^{2+} ion and is typically monitored closely during municipal water treatment processes. Concentrations of manganese above the limit can cause adverse negative effects to human health, environment and ecosystem [10], [11]. Essentially, it is believed that excessive level of Mn is harmful for nervous system and could lead to Parkinson's disease [12]. The source of manganese toxicity could be traced back from different sources such as drinking water, contaminated food and environmental setting [13]. The use of standard laboratory instrument to detect manganese in water is sometimes restricted by several limitations [4]. Apparently, the instrument is bulky in size and needs to be recalibrated after movement of the tool which prevents its use for outdoor analytical work such as for in situ measurement. On one hand, the need for in situ measurement is critical in some applications to minimize inaccuracy and instability of the measurement due to transportation issue which will jeopardize the quality of the samples [14], [15]. On the other hand, the expensive cost of the instrument also limits its use in any work that has financial constraint. The use of manual sampling method also yields inaccurate measurement due to high spectral and temporal variability [16].

As mentioned above, in order to overcome this, there are recent interests and efforts to set up a low-cost colorimeter that mimics the conventional spectrophotometer [17], [18]. One of the main features are portability and flexibility for future retrofitting which can be targeted for real time, in situ and remote implementation. Generally, light emitting diode (LED) and photodiode, acting as light source and photodetector respectively are two main components suitable for this type of implementation. Recent development of such system which is targeted for nitrate and glucose detection has been successfully developed as analytical device [19], [20]. Similar works include a setup for monitoring fluoride in drinking water supply as well as for educational purpose [21], [22]. Apart from using LED and photodiode, colorimetric sensing is also implementable through the use of mobile camera and scanner [23], [24]. This approach however, requires an additional digital and image processing work which could complicate the processing part and increases its power consumption. Even though it is simple, the use of mobile phone is more costly compared to the use of LED and photodiode. To the best of our knowledge, the implementation of low-cost spectrophotometer based on colorimetry effect for HMI detection is still yet to be explored. In this work, we aim to develop a colorimetric-based setup for HMI detection and measurement, by selecting Mn^{2+} as the target ions or model studies. The setup is based on the use of commercial-of-the-shelf (COTS) components such as LED and photodiode. This work is considered as a preliminary work towards developing a more complex system in the future targeting for real time and in situ monitoring of Mn^{2+} (in specific) for water quality assessment.

2. RESEARCH METHOD

In order to achieve the objectives, this work was divided into two main experiments. Initially, spectrophotometry technique was used to identify a suitable LED color/wavelength to be used for the final setup. Then, the system was developed prior to several preliminary tests to determine the suitable configuration for the system such as the optimum resistance value and supplied current for working LED.

2.1. Spectrophotometry test

To evaluate the proposed setup's performance, a spectrophotometry test was conducted using a Hach DR2800 spectrophotometer. The visible wavelength range of 400 to 700 nm was selected for absorbance measurement. This range was chosen because it corresponds to the wavelength of visible light that can be absorbed by the Mn^{2+} ions, allowing for accurate detection and measurement.

2.1.1. Samples and reagents

To prepare the samples for testing, the standard procedure outlined in Hach's preparation manual was followed [25]. The samples were prepared using a titration and dilution procedure, which involved using chemicals, listed in Table 1 a blank sample and Mn^{2+} solution ranging from 0.2 to 1.0 mg/L were prepared to cover a range of concentrations for testing. This allowed for a comprehensive evaluation of the proposed setup's ability to detect and measure Mn^{2+} ion concentrations.

Table 1. List of materials and reagent used to prepare Mn^{2+} solution sample

Materials/Chemicals	Function
Manganese (II) sulphate	To investigate the manganese ions contain in the salt.
Ascorbic acid powder pillow	To reduce the high oxidation state within the sample to the lowest oxidation state of $+2$.
Alkaline cyanide reagent	To masks all the interferences by other metals except manganese ions.
0.1% PAN indicator solution	To detect the presence of manganese ions by changing the color of prepared sample solution from yellow to orange color.
Distilled water	To act as solvent to dilute the salt.

10 mL of distilled water is used as blank sample. For sample solutions, manganese (II) sulphate which is in powder form is weighted by using analytical balance at weight of 0.001, 0.002, 0.003, 0.004, and 0.005 g respectively. Then, 500 mL of distilled water is added to get concentration of 0.2, 0.4, 0.6, 0.8 and 1.0 mg/L. Then 10 mL of sample solutions with different concentration are transferred into test cells. One ascorbic acid powder pillow is then added into each cell, including blank sample and all cells are inverted to dissolve the powder. 12 drops of alkaline cyanide reagent are added into all cells and are swirled gently. After that, 12 drop of 0.1% PAN indicator solution is finally added and the cells are swirled again to obtain a set of colorimetric test solution with gradual change of color from yellow to orange color.

2.1.2. Peak wavelength scanning and identification

Wavelength scanning was performed to identify the peak wavelength that gives maximum absorbance for the intended range of heavy metal ions under test. For wavelength scanning, 1 mg/L analyte solution was used as a reference sample to identify the peak wavelength by assuming that at this concentration, the absorbance effect has the most responsive reading. The finding was significant in determining the color of LED to be used in the proposed colorimetric setup.

2.2. LED-based colorimetric setup

In designing the colorimetric setup, the primary objective was to use inexpensive components that could be easily obtained from a local electronic store to ensure low-cost implementation. The goal was to create a setup that could effectively mimic the operating concept of a commercial spectrometer, which typically uses expensive and bulky equipment. By using low-cost components and simplifying the design, the proposed setup aimed to provide an affordable alternative for measuring Mn^{2+} ion concentrations, particularly in settings where budget constraints are a limiting factor. Figure 1 shows the circuit used for the colorimetric setup.

The function of each component used is described in Table 2. For this setup, measuring instrument such as voltmeter and ammeter were used in order to verify the electrical signal during circuit operation. This is important as the components used came with certain tolerance levels.

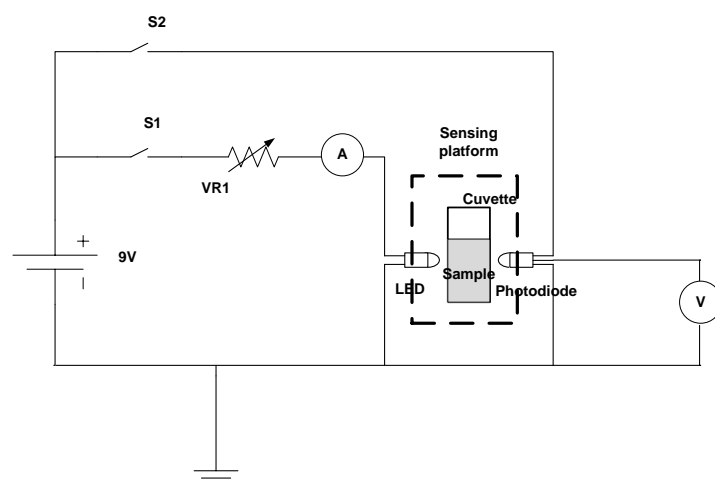


Figure 1. Schematic circuit of experimental setup

Table 2. Description of the colorimetric setup and its configuration

Component/Instrument	Functionality
LED	To provide light source
Photodiode	To detect the light intensity
Potentiometer, (VR1)	To avoid excessive current through LED (Current limiting resistor)
Cuvette	To place solution under test
Switches (S1 and S2)	To control the supply to LED and photodetector
Ammeter (A)	To measure the current through LED
Voltmeter (V)	To measure the voltage output (represent the detected intensity)

2.2.1. Components' configuration

LED and the value of current limiting resistor were two components that needed to be predetermined before the final test was executed. The selection of suitable color (wavelength) of LED is crucial due to

absorbance-wavelength dependency. A commercial LED with the closest wavelength obtained in spectrophotometry test will be used in the final test circuit. In the meantime, the selection of resistor's value was also crucial as the wrong value of resistor could damage the LED due to excessive current. The maximum setting of resistor, which was acting as current limiter was calculated based on measured voltage across LED, voltage LED (VLED) and the maximum current, current LED (ILED) max through it. In the setup, VLED was measured as 3.4 V. If the maximum current was set to maximum value according to datasheet, the resistance was calculated to be 0.28 kΩ. That means, if the limiting resistor was selected to be less than the calculated value, LED will draw more current than the maximum rating and might blow due to excessive current. The use of variable resistance is useful if the intensity of LED is to be varied. In this setup however, preliminary test has found that 1 mA current is sufficient to observe the absorbance effect.

2.2.2. Experimental procedures

Based on the selected components outlined in previous section, the final setup was realized to obtain the calibration curve of the Mn^{2+} sample at the intended range of interest. Firstly, by following the procedures outlined by Hach manual [25], a set of colorimetric sample solutions, blank solution (contains only colorimetric agent) and analyte solutions (containing Mn^{2+} ions) were prepared. For analyte solution, the samples were prepared at concentration of 0.2, 0.4, 0.6, 0.8, and 1.0 mg/L. For each sample, voltage measurement was taken and plotted for the range of concentration of interest to visualize the pattern of the detected signal. The voltage is a representative of the detected light with the assumption that the absorbance effect (due to different tone of color) will reduce the amount of light received by the photodiode, thus reducing the measured voltage. The voltage measurement was then used to calculate the absorbance based on (1) [22].

$$A = -\frac{V_{sample} - V_{zero}}{V_{solvent} - V_{zero}} \quad (1)$$

where A is absorbance, V_{sample} is the voltage of the sample at specific concentration, $V_{solvent}$ is the voltage of the blank solution and V_{zero} is the offset voltage.

The test and measurement were performed in a black interior and closed container platform, similar to the commercial instrument setup to minimize the interference that could be originated from the background lighting. This setup is adapted to ensure that no light can escape or penetrate into the sensing platform. Apart from that, the offset voltage was also predetermined in the absence of colorimetric agent. The voltage was to be subtracted from the actual measurement as given in (1). This will automatically remove any offset voltage that could interfere the measurement. The use of LED and photodiode for measurement were repeated for 10 times to get the average data of absorbance for different concentration of Mn^{2+} solution. In this experiment, it was predicted that the higher concentration of Mn^{2+} will cause higher absorbance. So lesser light will be detected at photodiode, thus reducing the voltage. Besides, the variance and standard deviation of the voltage measurement were also calculated to indicate the repeatability of the measurement.

2.2.3. Performance evaluation

In order to evaluate the effectiveness of the proposed setup, the linearity of the plots for both voltage and absorbance measurements were examined using the correlation of coefficient factor, R^2 . The linearity of the voltage plot was used to observe the behavior of voltage in response to increasing levels of Mn^{2+} concentration, which resulted in changes in color and subsequent light absorption. Similarly, the absorbance-concentration calibration curve was examined to verify the proposed setup's ability to accurately predict the relationship between absorbance and Mn^{2+} concentration. By analyzing the linearity of both measurements, the researchers were able to determine the effectiveness of the proposed setup in detecting and measuring Mn^{2+} ion concentrations.

3. RESULTS AND ANALYSIS

The results section presents the findings of the study, which are based on the methodology described earlier. The experimental work conducted to verify the proposed setup is discussed, and the observations are presented. The results are presented in a structured manner to enable the reader to understand the findings in a logical sequence. The discussion is based on the experimental observations, which are interpreted to provide insights into the effectiveness of the proposed setup in measuring Mn^{2+} concentration.

3.1. Spectrophotometry test

Figure 2 shows the color of the sample containing various concentrations of manganese. The figure indicates that the sample's color changes according to the concentration of Mn^{2+} ions present, which is further

enhanced by the addition of a colorimetric agent (0.1% PAN indicator solution). The colorimetric agent provides a specific color to the solution with and without Mn^{2+} ions, allowing for easy visual detection and measurement of the Mn^{2+} concentration.

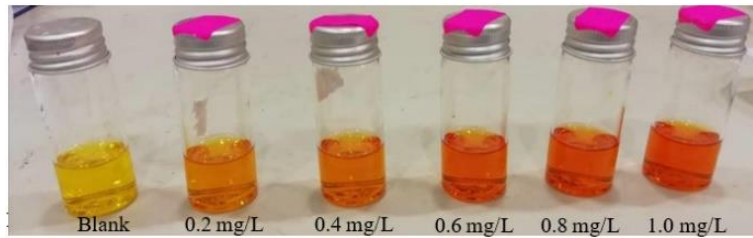


Figure 2. Colorimetric assay of manganese

The transition of yellow color (blank solution) into orange color was observed for all five samples containing Mn^{2+} ions. The light orange color was observed for low concentration of manganese while the dark orange color was observed for high concentration of manganese i.e., the higher the concentration of manganese in the sample would cause the orange color to become darker. Figure 3 shows the spectrophotometry wavelength scan to identify the peak absorbance for a wide spectrum of the light.

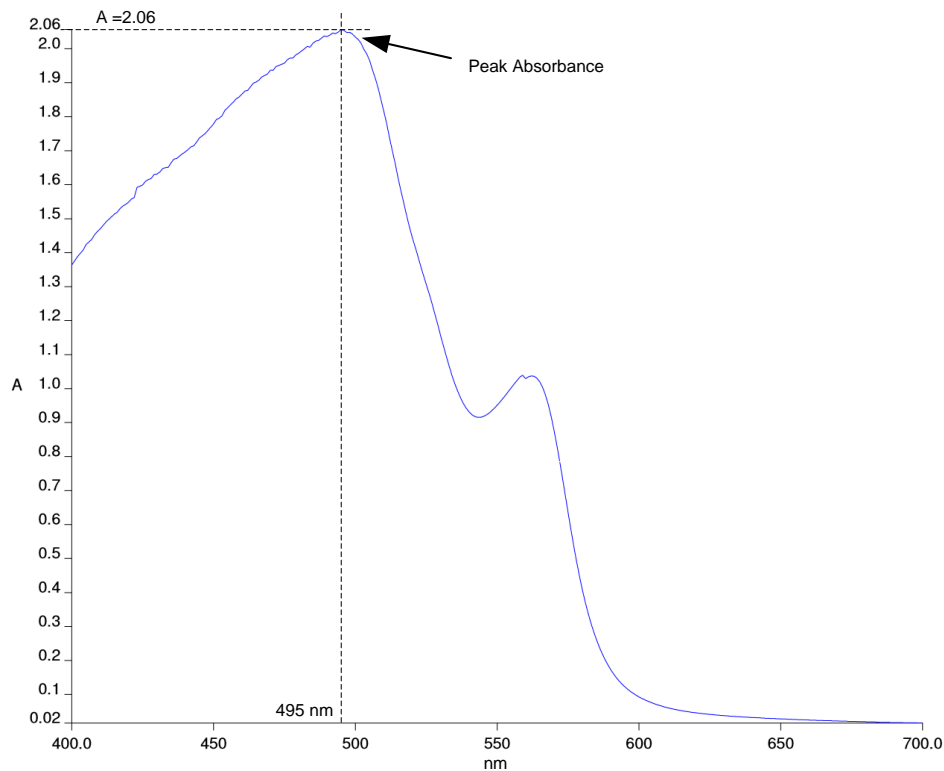


Figure 3. Absorbance spectra of optical wavelength between 400 to 700 nm

It was observed the absorbance behavior for different wavelength had varied. Different wavelength had different absorbance characteristics when passing through a colorimetric sample. This indicates that for monochromatic optical light source design, the selection of LED's color is crucial to ensure optimum sensitivity of detection is achieved. Peak absorbance occurred at the wavelength equals to 495 nm with absorbance value of 2.06. According to Beers law, this value means that at 1 mg/L, the light will absorb nearly 99% of the light source. Since the peak wavelength lies within the blue light spectra (450 to 495 nm), the blue LED was selected to be used as a light source for our colorimetric experimental setup.

3.2. LED photodiode colorimetric setup

In Figure 4, the voltage detected at the photodiode is plotted against different concentrations of the prepared Mn^{2+} samples. The graph shows a clear increasing trend in voltage as the concentration of Mn^{2+} in the samples increases. This is expected since the colorimetric agent PAN reacts with Mn^{2+} ions in the sample, resulting in a color change, which in turn affects the amount of light absorbed by the sample and the voltage detected by the photodiode. The plot shows a good linearity, which indicates that the proposed setup is reliable and suitable for use as a calibration curve for Mn^{2+} detection and measurement.

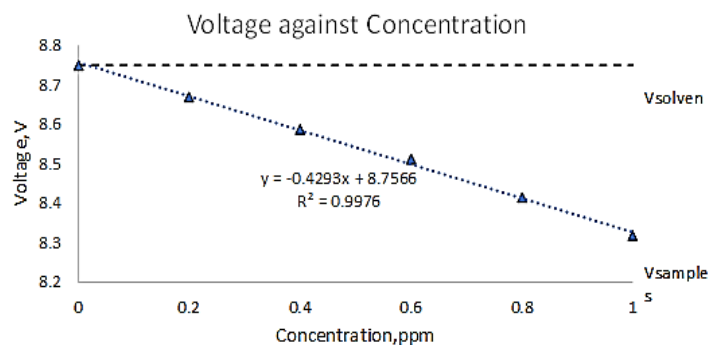


Figure 4. Voltage against concentration

$V_{solvent}$ represents the voltage for the blank sample consisting only the solvent without any Mn^{2+} ions in the solution. Meanwhile, the plot of V_{sample} shows a decreasing pattern of voltage if the concentration of Mn^{2+} ion was increased, which is in line with our initial hypothesis. At higher concentration, it is expected that more light will be absorbed, thus reducing the light that is passing through the sample (absorbance effect). This in turn reduces the voltage detected by photodiode (as photodiode voltage is proportional to the light received). The gap between $V_{solvent}$ and V_{sample} proves the capability of the setup to give the signal that correlates between the absorbance and concentration of the tested samples. For each concentration, the variance and standard deviation of voltage measurement was calculated to be less than 0.025 and 0.159, respectively, indicating high repeatability for the proposed setup. In terms of linearity, the plot shows a correlation coefficient, R^2 of 0.9976, suggesting that the voltage behavior for the setup is suitable to represent the amount of light absorbed by the samples at different concentration. However, it was observed that the range between the minimum and maximum voltage is quite small, at around 1 V gap. It is due to several factors such as the range of sample concentration used in this experiment, the sensitivity of the photodiode itself, disturbance from stray light and others. The use of suitable amplifier and signal processing techniques might be needed in future implementation to improve its sensitivity. Figure 5 shows the plot of absorbance against concentration of the Mn^{2+} sample. The absorbance value was extracted from the voltage measurement according to the (1).

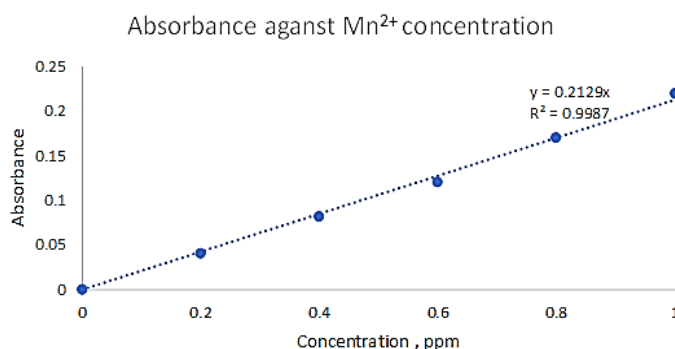


Figure 5. Absorbance against concentration

It was observed that relationship between calculated absorbance and concentration behaved as expected. The absorbance was increasing as the concentration of Mn^{2+} ions was increased. In terms of linearity, the plot gives a good linear relationship between the absorbance and concentration, with R^2 value equal to

0.9987, indicating the suitability of the setup to be used for measuring the absorbance of different concentration of Mn^{2+} ions at the proposed concentration range. This behavior is significant in estimating the concentration within this range.

However, the value of the calculated absorbance value seems to be degraded by several factor in comparison with the peak value obtained from spectrophotometry test. From equation, this is due to the small difference of voltage between V_{sample} and $V_{solvent}$ which leads to a very low calculated absorbance. Meanwhile, several reasons related to the use of discrete LED could also be the factors of this low absorbance, which require more specific testing to be performed in the future. In this work, the LED was chosen based on the best wavelength region (blue region), which might not have the same wavelength to match the peak wavelength obtained in spectrophotometry test. The need for spectrum analysis is thus required in the future to confirm the exact wavelength of the LED. Apart from that, there is a potential deviation due to different intensity of light between our colorimetric setup and commercial spectrometer used in qualitative work, which require another instrumentation setup to study its effect. In real implementation, the issue can be solved by having a proper amplification or signal processing technique to increase the representation of the absorbance data.

4. CONCLUSION

As a conclusion, the objective to develop a colorimetric setup for Mn^{2+} ions have been successfully achieved. The setup shows a good linearity with R2 approaching unity for both voltage and absorbance plot against Mn^{2+} concentration. Such setup can be a basic instrumentation platform for a more complex application such as for in situ and real time monitoring, subjected to further improvement with another test and performance assessment. The sensitivity can be further increased either by means of hardware (amplifier) or software (signal processing) to ensure a higher absorbance can be produced. In general, all steps, procedures and approaches used in this work are also significant and useful for low-cost development of colorimetric setup for other heavy metal ions depending on the selection of suitable colorimetric reagents and optical components.

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