

Development of an optical pH measurement system based on colorimetric effect

Puteri Nur Syahirah Mohamed Mustafa¹, Aiman Shahmi Azam², Mohd Suhaimi Sulaiman³,
Ahmad Fairuz Omar⁴, Mohamad Faizal Abd Rahman⁵

^{1,2,3,5}School of Electrical Engineering, College of Engineering, Universiti Teknologi MARA, Shah Alam, Malaysia

⁴School of Physics, Universiti Sains Malaysia, Shah Alam, Malaysia

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ABSTRACT

This paper proposed an optical pH measurement system developed to measure the changes of pH levels based on colorimetric reactions with phenol red reagent. The optical sensing was achieved through the implementation of a pair of light emitting diode (LED) and photodiode. The detection mechanism was based on different absorbance of light intensity at pH values of 2.36, 8.32, 9.08 and 12.83. The data processing method was carried out using LabVIEW software and interfaced with NI USB DAQ 6008. The measured voltage showed a good correlation in relation to the pH level with R2 equals to 0.9624. This relationship was used as a calibration curve in the final system testing. The final measurement of pH showed a good agreement between the actual and measured values with an error of less than 5%, thus indicating the reliability of the proposed system.

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Corresponding Author:

Mohamad Faizal Abd Rahman
School of Electrical Engineering, College of Engineering
Universiti Teknologi MARA
Kampus Permatang Pauh, Shah Alam, Malaysia
Email: faizal635@uitm.edu.my

1. INTRODUCTION

In numerous scientific and industrial research areas, pH level is an important parameter that requires continuous measurement and monitoring. pH measurement is necessary in accessing a liquid/solution's chemical properties and behavior, hence it is useful for controlling and preventing undesired chemical reactions as well as optimising desired beneficial reactions [1], [2]. Several physical, biological, and chemical parameter factors that have a substantial impact on water quality must be recognised in order to classify it [3], [4]. Many applications in medicine, biology, chemistry, environmental science, oceanography, agriculture, forestry, civil engineering, chemical engineering, water treatment and purification, food science, and nutrition require the measurement of pH [5], [6].

Several sensing mechanisms have been adapted for pH measurement such as potentiometric, electrochemical and colorimetric [7]-[9]. The implementation of pH measurement based on the combination of optical and colorimetric effect is possible and preferred in solution-based application due to several advantages such as sensitivity, selectivity and more responsive [10]-[12]. The variation of pH level in colorimetric solution reflects its colour, which consequently affects the optical properties such as absorption and reflection. For sensing purpose, this behaviour can be manipulated to produce a measurable signal [13], [14]. In order to produce such colorimetric effect, different reagents and chemoresponsive colourants are available and used in the construction of such optical chemical probes [15]-[17]. Colorimetric analysis for pH

measurement has been widely used in both small scale and industrial size application due to its profound advantages [18]-[20]. In fact, the sensing concept has been adapted in the design of commercial colorimeter with good reputations and remarkable performance and widely used in industrial application and research purposes remarkable performance [21], [22]. As based on traditional technique, they take the water sample to a lab to determine the values of the water quality parameters and then take the necessary controls. Due to the numerous processes involved, this procedure is exhausting, tedious, and costly [23]. Nevertheless, such instrument is commonly coming with expensive cost which limits its usage. The need to have a low cost system is thus needed for general purpose measurement activities to verify and monitor the surrounding setting, particularly for pH related application [24].

Based on this scenario, this work is aimed to develop a low-cost colorimetric pH measurement system. The ability to develop such system can benefit small industries as well as for educational purposes. The system uses an optical sensor that consists of a photodiode coupled with a blue light emitting diode (LED) light source to detect the changes of colour and pH in aqueous medium based on beers law and colorimetric reactions. It transforms electrical input signals into light signals and sends them across the optical fibre. They are made of several semiconductor materials, allowing the designer to control the emission wavelength [25]. LEDs are commonly used as light sources in many low cost systems due its advantages in terms of life cycle, exceptional colour range, low radiated heat as well as low power consumption [26]. In terms of colorimetric reagent, phenol red is used to produce colorimetric effect that associated with pH variation. This reagent is widely used as pH indicator in many colorimetric based pH measurements such as in cell culture, medical and water quality tests [27]-[29]. Its goal is to learn how well an optical sensor performs in terms of light absorption as a medium for photon absorption, as well as its ability to provide higher electrical parameter outputs, including current and voltage [30].

2. METHODOLOGY

In this work, four colourless solutions with different pH were selected as samples, namely vinegar, tap water, baking soda and bleach. 10ml of each sample solution was placed inside a test cuvette for pH measurement using a standard spectrometer (HACH) as in Figure 1(a). This reading was used as a baseline pH with no reagent presence. The process was repeated by adding several drops (1 to 4 drops) of colorimetric reagent; phenol red as in Figure 1(b). Phenol red water-soluble dye was used as a colorimetric reagent or pH indicator, changing from yellow to red all over. The pH level of solutions under the presence of different amounts of phenol red were measured and recorded to observe the effect of reagent presence. This is crucial to observe any influence in pH due to the addition of different drops of reagent, which could give inaccurate results. The samples were then labelled and to be used for testing with the proposed system.

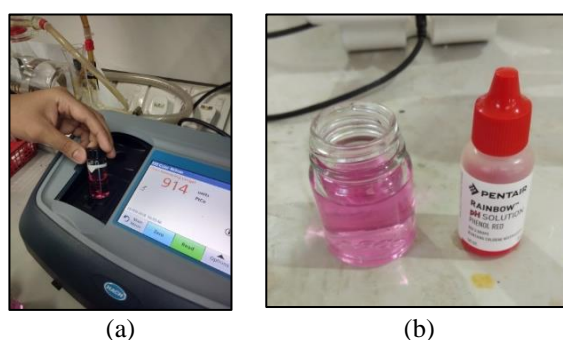


Figure 1. Instrument and colorimetric reagents (a) pH measurement using a spectrometer
(b) Phenol red as colorimetric reagent

System development. This section explains the related works in developing the proposed system:

a) Optical system configuration. Figure 2 shows the optical sensor setup for pH measurement

In this setup, a blue LED ($\lambda=560$ nm) was selected as the light source while a photodiode was used as a photodetector. A voltage divider circuit was implemented as the readout circuitry, which could be configured by a variable resistor of 100k Ω . In this work, it was targeted to have a maximum of 5V across X2 (Figure 1(b)) for the maximum value of pH. The value was also compatible with the data acquisition system (DAQ) which worked at a maximum of 5V. The distance between the LED and photodiode was 12mm to allow sufficient space for a cuvette with a dimension of 50 \times 10 \times 10mm. The setup was placed inside a black box container to reduce the stray light effect that could interfere with the measurement.

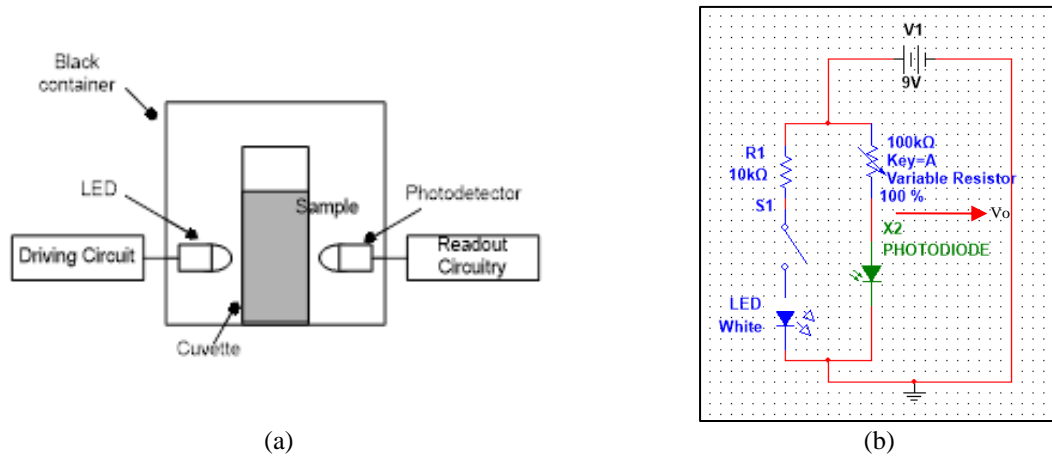


Figure 2. Optical sensor setup of the proposed setup; (a) optical system and (b) schematic circuit of optical sensing system

b) Data acquisition (DAQ) and graphical user interface (GUI)

Data acquisition (signal interfacing) was established using NI USB DAQ 6008. In this process, the analog voltage signal from the readout circuitry was converted into binary code through data acquisition system (DAQ) using its internal analog-to-digital converter (ADC). Once the interfacing was established, the digital data was then passed through a graphical code established using LabVIEW 2015 for the next processing. Using LabVIEW, the graphical user interface was developed as a medium for data collection and interaction between the user and the optical sensing system. The data can be monitored directly through a PC or laptop for data collection and monitoring purposes. Figure 3 shows the graphical block diagram and the front panel of the system using NI LabVIEW.

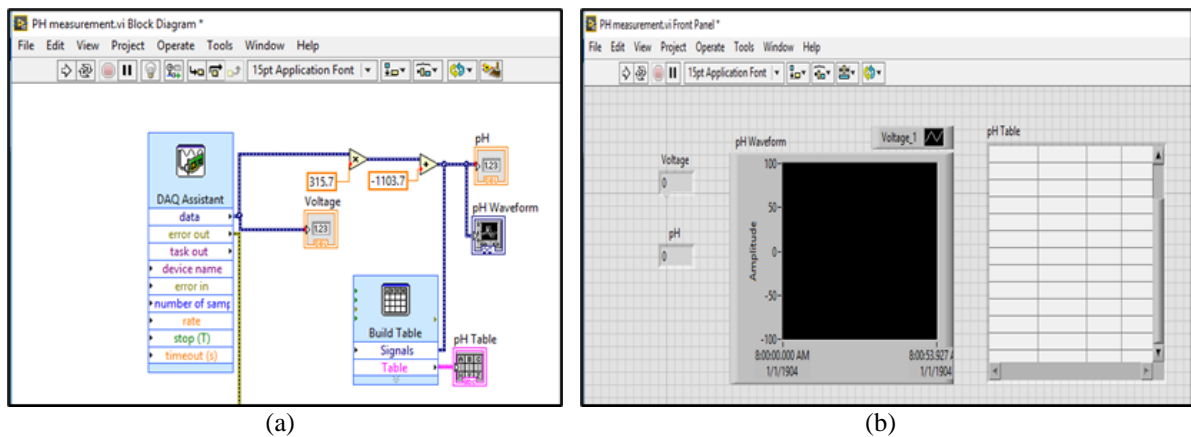


Figure 3. LabVIEW programming; (a) graphical block diagram and (b) front panel (GUI)

c) Experimental setup and testing procedures

In the experimental work, two types of measurement were conducted. Initially, voltage measurement was performed to obtain the relationship between the voltage and pH of the prepared samples. This step is important to study how the signal voltage is changing with respect to the colorimetric samples. This relationship is to be used as a calibration curve for the final conversion process. Meanwhile, the final testing was aimed to verify the performance of the proposed system in measuring the pH level. Samples were initially prepared according to the procedure described earlier and the actual pH was measured using a conventional spectrometer.

- Voltage measurement (pH vs voltage)

Initially, the test was performed by measuring the voltage through each sample in the cuvette using the optical setup explained earlier. The samples were placed in a cuvette and the voltage obtained were

measured accordingly. The voltage represented the amount of light received by the photodiode after passing through a colorimetric solution with different pH values. Several readings were taken to reduce the systematic error that could happen during the measurement process. The readings were then recorded and plotted to observe the behaviour of pH and voltage relationship. The relationship was analysed in terms of its linearity and sensitivity. The equation extracted from this relationship will be used as a calibration curve for pH conversion.

- Final testing (actual pH vs measured pH measured)

The second experiment was executed using the final setup as illustrated by Figure 4. The prepared samples were measured accordingly and repeated several times. The data of pH measured by the spectrometer and the proposed system were collected and plotted to observe their difference and correlation.

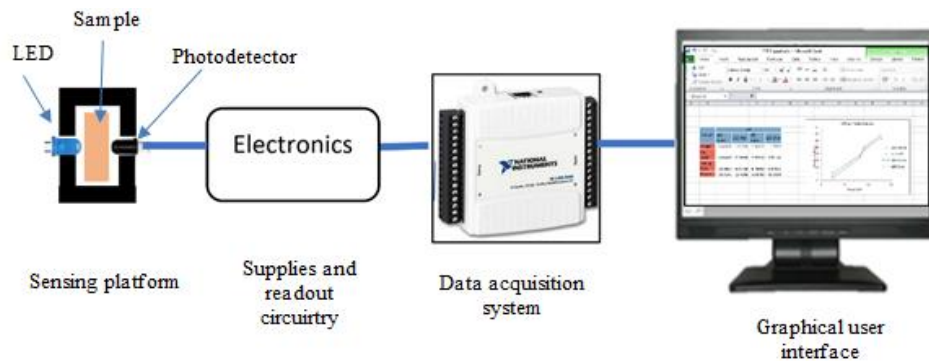


Figure 4. Block diagram of overall setup

d) Data analysis and system verification

Based on the collected data, two types of analysis were carried out to measure the performance of the proposed work. The first analysis was performed to evaluate the voltage measured for different pH samples during the establishment of the calibration curve. For the relationship between the pH and measured voltage, parameters such as correlation coefficient, linearity as well as sensitivity were obtained.

The second analysis was performed to verify the performance of the developed system. The performance was evaluated based on the difference between the actual pH measured using a standard spectrometer and the pH measured using the proposed system. The two parameters used for this purpose were absolute error and percentage of error which were calculated based on (1) and (2).

$$Absolute\ Error = |V_A - V_E| \tag{1}$$

$$Percentage\ Error = \left| \frac{V_A - V_E}{V_E} \right| \times 100\% \tag{2}$$

Where,

$V_A =$ approximate (measured) value

$V_E =$ exact value

3. RESULTS AND DISCUSSION

Sample preparation. Table 1 shows the pH level obtained for each sample under different effects of colorimetric reagent. The measurement shows the pH level with no reagent presence as well as when several drops were added. It can be observed that there is only a slight variation of pH when the reagent was added at a higher pH level. By comparing the average value of pH (presence of reagent) with the pH (no reagent presence), the data gives convincing results to show the insignificant pH variation. The data shows that for all samples, the percentage of deviation is all less than 5%. Due to such a small value of relative error, it is concluded that under the effect of less than four drops of colorimetric reagent, the samples do not show a significant variation of pH while maintaining its colour. This indicates that the use of phenol red to produce a colorimetric effect will not influence much on pH and is suitable for this testing.

Table 1. Table of sample preparation

No. of Reagent Drop	SAMPLE 1 10ml Vinegar (pH)	SAMPLE 2 10ml Tap Water (pH)	SAMPLE 3 10ml Baking Soda (pH)	SAMPLE 4 10ml Bleach (pH)
0	2.35	8.28	8.86	12.69
1	2.35	8.32	9.14	12.80
2	2.36	8.31	9.14	12.83
3	2.37	8.32	9.13	12.86
4	2.34	8.36	9.12	12.88
	2.37		9.07	12.90
	Average 2.36 pH	Average 8.32pH	Average 9.08 pH	Average 12.83 pH

Figure 5 shows the prepared colorimetric samples as outlined in the sample preparation method. As expected, the colour of the solution goes yellowish for pH less than 6 and reddish for pH more than 6 and higher. The samples indicate the colorimetric effect upon the addition of the colorimetric reagent, phenol red. The pH measured using a standard spectrometer was then labelled as in the Figure 5. Voltage measurement. Table 2 shows the voltage measurement of pH level of the prepared samples. These values were used in generating the calibration curve meant for the voltage-pH conversion in the final system setup.

The plotted graph of pH against the measured voltage is shown in Figure 6. It can be seen from the plot that the relationship between pH and voltage is generally linear with the coefficient of linear correlation, R^2 is equal to 0.9624. This value indicates the reliability of the linear relationship between voltage and pH for the given range of pH level. The equation for this relationship was computed to be equal to $y = 0.0069x + 3.39$. The gradient of this equation gives the sensitivity of the pH-voltage relationship to indicate that for a unit change of pH, it will produce a change of 6.9mV, while the intercept gives the value of an offset voltage. It is also concluded that the colour changes due to the colorimetric effect (associates with pH variation) which produces different absorbance behaviour that is reflected by the voltage detected by the photodiode. Thus, the ability of the photodiode to detect small changes in light absorption becomes one of the most crucial factors to produce a sensitive measurement pH measurement (actual vs measured). Table 3 shows the measured pH obtained using both the spectrometer and the proposed LED system.

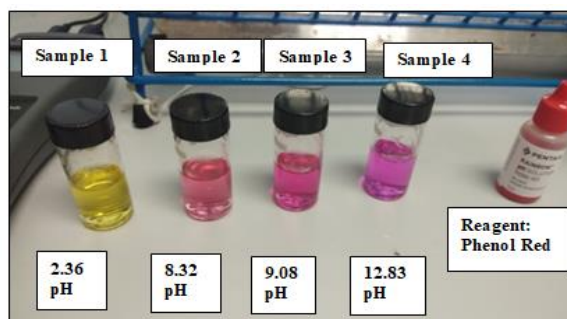


Figure 5. Sample preparation with true value measurement

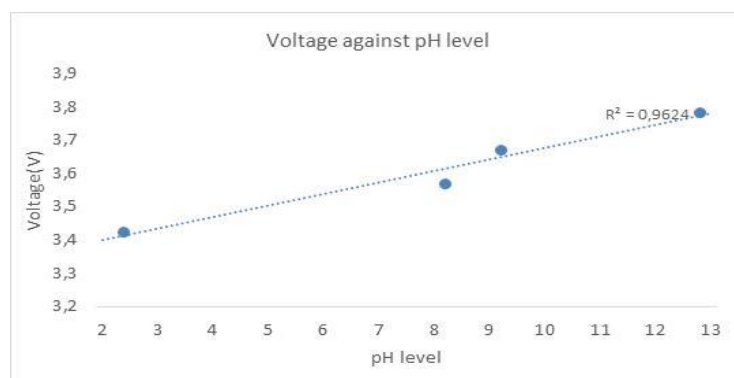


Figure 6. Graph of voltage against pH level

Table 2. Voltage reading at photodiode for blue LED

Sample	Voltage (V)
Vinegar	3.42
Tap Water	3.53
Baking Soda	3.67
Bleach	3.78

Table 3. pH reading for each sample

Sample	pH		Error	
	Actual pH	Measured pH	Absolute Error	Error (%)
Vinegar	2.36	2.25	0.11	4.66
Tap Water	8.32	8.02	0.30	3.61
Baking Soda	9.08	8.87	0.21	2.28
Bleach	12.82	13.10	0.28	2.18

It can be observed that the measured pH values give a very small deviation in magnitude as compared to the expected values. In terms of its percentage of error, in general, the calculated percentage error produces less than 5% of errors. It is also noticed that the percentage of errors becomes less as the pH level increases. In order to see it clearly, the percentage error is included in the final graph. Figure 7 shows the actual plot and measured pH with the error bar of 5%. In this graph, the ideal and targeted line (measured values equal to actual values) is also included. It is observed that in comparison with the targeted values, the measured pH for all samples lies within the range of 5% errors, which indicate a good performance of the measurement process. In other words, the measured value lies within 5% of the given tolerance.

It can be observed that for higher level of pH, the error bar (percentage of error) becomes larger, thus allowing a larger deviation to be within 5% tolerance. That means for a lower pH, a small changes of error magnitude contributes to significant percentage error. There are several sources that could possibly caused the errors. Simplification error caused by the used of calibration curve that is not exactly equal to unity might introduce errors during the conversion from voltage into pH.

Apart from that, the experimental setup and measurement activity could also introduce some systematic error that affect the measurement data. Gross and random error caused by the environmental such as the lighting could introduce stray light during measurement. Finally, based on the analysed data, in terms of its good linearity, small deviation and errors and within the specified tolerances, it is suggested that the developed optical based system is suitable and efficient to measure a very wide range of pH in solutions with acceptable accuracy and tolerances.

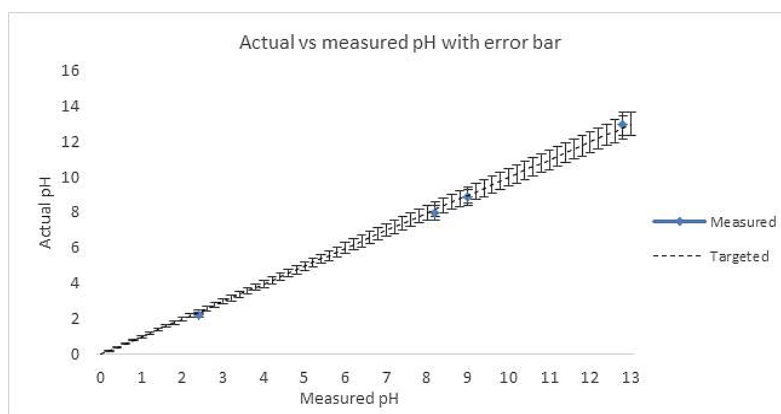


Figure 7. pH measurement using the propose system

4. CONCLUSION

It is concluded that the main objectives of this work are successfully achieved. The system shows a good performance and performs within an acceptable tolerance. The use of organic dye such as phenol red as colorimetric reagent is found to be suitable to produce a change of color according to pH variation. Despite the successful and functionality of the proposed system, future work will also consider several improvements to be taken such as by increasing the sample population as well as to minimise the possible errors that might affect the results. This system can also be expanded in future by considering integrating it with internet of things (IoT) capabilities, making it fully automated as well to implement the sensing activity on microfluidic platform.

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BIOGRAPHIES OF AUTHORS



Puteri Nur Syahirah Mohamed Mustafa was born in Sungai Petani, on October 23, 1997. She is a research assistant at Universiti Teknologi MARA Pulau Pinang. She obtained her Diploma in Electrical (Power) Engineering and Bachelor Degree in Electrical and Electronic Engineering from Universiti Teknologi MARA (UiTM) Pulau Pinang. Her interests are in optical sensing, infrared thermography and artificial neural network.



Aiman Shahmi Azam is currently a product engineer at Sony EMCS (Prai) Sdn. Bhd. He obtained his degree in 2018 and graduated from Universiti Teknologi MARA (UiTM) Pulau Pinang in Electrical and Electronic Engineering. His final year project was in optical colorimetric sensing with industrial experience at water distribution company as a trainee. During his industrial training period, he involved in testing the water quality parameter such as pH and chlorine for water quality inspection.



Ts. Dr. Mohd Suhaimi Sulaiman is a senior lecturer at the Faculty of Electrical Engineering, Universiti Teknologi MARA Cawangan Pulau Pinang. Ts. Dr. Mohd Suhaimi completed his PhD in Electrical Measurement and Instrumentation at the Faculty of Electrical Engineering, UiTM in 2019. His research interests are Electrical Measurement, Water Quality, Agriculture Technology and Artificial Neural Network ranging from theory, design up to implementation.



Dr. Ahmad Fairuz Omar is a senior lecturer and researcher at the School of Physics, Universiti Sains Malaysia. He obtained his bachelor degree in electrical and electronics engineering from Universiti Sains Malaysia in 2002, master degree in electronics engineering from Open University Malaysia in 2008 and doctorate in optical spectroscopy from Universiti Sains Malaysia in 2012. His research mostly focuses on the application of optical system in environmental monitoring, food analysis and medical research.



Dr. Mohamad Faizal Abd Rahman is a senior lecturer and researcher at the Faculty of Electrical Engineering, Universiti Teknologi MARA Cawangan Pulau Pinang. He obtained his PhD in Sensor and Instrumentation from Universiti Sains Malaysia. His research interests are in optical sensor and instrumentation, microfluidic based application and colorimetric sensing in various application.