A Sensitive Optical Method based on Open-source Hardware Technology Capable of Accurately Measuring the Refractive Indices of Aqueous Solutions

Konthee Boonmeeprakob[1,2], Panote Thavarungkul[1,2,3,4], Proespichaya Kanatharana[1,2,4], Chittanon Buranachai[*1,2,3]

[1]Center of Excellence for Trace Analysis and Biosensor, Prince of Songkla University, Hat Yai, Songkhla,Thailand
[2]Division of Physical Science, Faculty of Science, Prince of Songkla University, Hat Yai, Songkhla, Thailand
[3]Thailand Center of Excellence in Physics, Commission on Higher Education, 328 Si Ayutthaya Road, Bangkok, Thailand
[4]Center of Excellence for Innovation in Chemistry, Faculty of Science, Prince of Songkla University, Hat Yai, Songkhla, Thailand
* Corresponding author, E-mail: chittanon.b@psu.ac.th

Abstract

In this work, the authors presented a sensitive and accurate optical method for measuring the variation of the refractive index of sugar solution (sucrose) with various concentrations (10%-50%). The method is based on finding a critical angle of a laser beam impinging on a microchamber filled with a sugar solution. The chamber is attached to a semicircle glass block and mounted on a stage attached to a stepping motor (geared at 100:1 ratio) for finely adjusting the incident angles. The intensity of the reflected laser light is monitored by a linear sensor array. The stepping motor control and the signal read from the sensor array are done with an Arduino microcontroller. Once the critical angle is obtained, the refractive index is calculated from Snell's law at the critical angle. This method has many advantages such as being highly sensitive, having high stability and repeatability, and requiring only microliters of liquid sample, inexpensive optical, and electrical components. Our method showed that the refractive indices of sucrose solutions vary linearly with the sugar concentration of sugar in the range of 0 to 50% and the precision of the measurement is estimated to be better than 0.10% (the standard deviations are in the range of 0.04-0.10%), which is well below the limit set by the Association of Official Agricultural Chemists (AOAC). It suggests that, with a little further development to improve user-friendliness, our method could be used with confidence to quantify sugar concentrations in real liquid samples, such as soft drinks beverages, and juices where sugar contents are often listed as a whole percentage or even in whole blood.

Keywords: *laser light, refractive index measurement, critical angle, sucrose, open-source hardware*

1. Introduction

The refractive index (n) is a basic optical property of materials. It plays an important role in many disciplines and industries, such as thin-film technology (Xi et al., 2007), quality control in glasses and plastics manufacturing (Yunus & Rahman, 1988; Tan & Huang, 2015). Besides, it was reported that the refractive indices are related to the concentrations of liquid media (Tan, & Huang, 2015; Singh et al., 2013) and there were several attempts to determine the concentration of the solution by measuring the refractive indices (Yunus & Rahman, 1988; Tan & Huang, 2015; Singh et al., 2013).

The obvious benefits of measuring the solution's concentration optically via the refractive indices over other methods, such as using the interdigital capacitor sensor (Angkawisittpan & Manasri, 2012) include contactless measurement avoiding routine washing or sample contamination. However, popular optical techniques to determine solution's concentrations, such as refractometry (Ramasami et al., 2004), infrared spectroscopy (Cadet et al., 1991) and optical coherence tomography (Esenaliev, Larin, Larina, & Motamedi, 2001) are either expensive or prefer measuring transparent liquids. In this work, the authors propose a simple yet accurate alternative. The authors used inexpensive optical parts and open-source hardware and software technology to develop a method to measure refractive indices and concentrations of sucrose solutions. Our technique is based on finding the critical angle of light reflecting internally from the glass-solution interface and using Snell's law to calculate the solutions' refractive indices. When compared with commercially available portable refractometers, our system still cannot compete in terms of portability and user-friendliness. However, our system is lower in price with comparable precision and the authors are building our system on an open-hardware and open-software platforms, which are more flexible and welcome any further improvement from the scientific community. The authors believe that with further development on user-friendliness, it will be more suitable for applications in undeveloped or developing counties.

2. Objectives

1) To develop a simple method and set up for measuring the refractive index of an aqueous solution

2) To measure the concentration of sucrose solutions by measuring the refractive index with the developed setup.

3. Materials and Methods

3.1 Setup

The microchannel for the aqueous solution is made by sandwiching two pieces of double-stick tape with a block of half-cylindrical glass (n=1.5) and a block of Poly(methyl methacrylate) (a.k.a. acrylic), before sealing both ends with epoxy glue, as shown in Figure 1. Two holes serving as an inlet and outlet were made in the acrylic. After the microchannel was filled with the desired aqueous solution, a monochromatic beam of light from a Helium-Neon laser (632.8 nm) was directed into the glass and incident at the center of the half-cylindrical glass situating above the microchannel (Figure 1A). The authors mount the microchannel on top of a stepping motor with a gear ratio of 100:1 (step angle = 0.018 degrees) with the axis of rotation passing through the light incident point. Therefore, the incident angle is changed simply by rotating the stepping motor. Then, a linear sensor array (TSL1402R, AMS AG, Austria) was used to measure the intensity of reflected light at each incident angle. Two Arduino microcontrollers (Uno R3 and Mega 2560 R3, Kuongshun Electronic Limited, China) were used to control the rotation of the stepping motor and reading the signal from the linear sensor array.

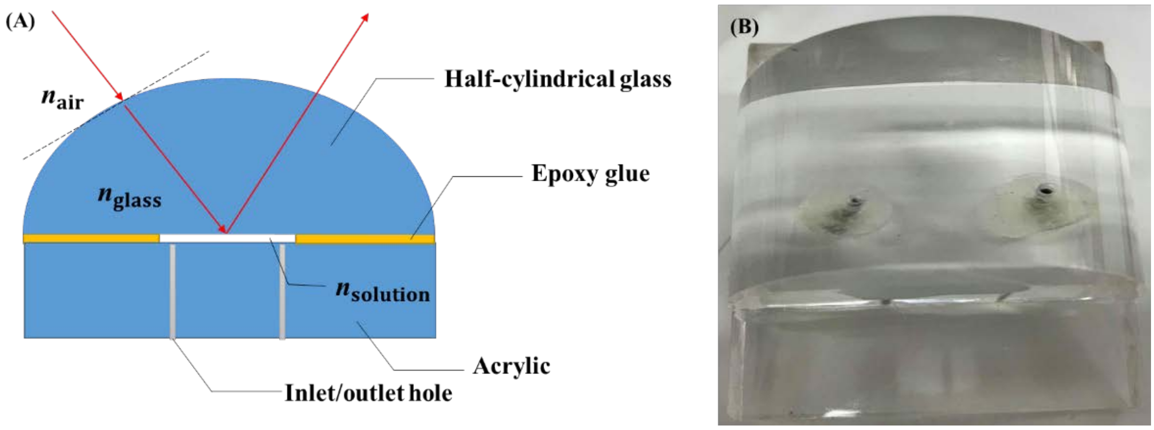

Figure 1 A sample chamber is made by cylindrical glass and a transparent acrylic together with a double-sided tape and by sealing with epoxy and the holes on the slide are used for the inlet and outlet of solution exchange
(A) Schematic of designed chamber, (B) Picture of chamber

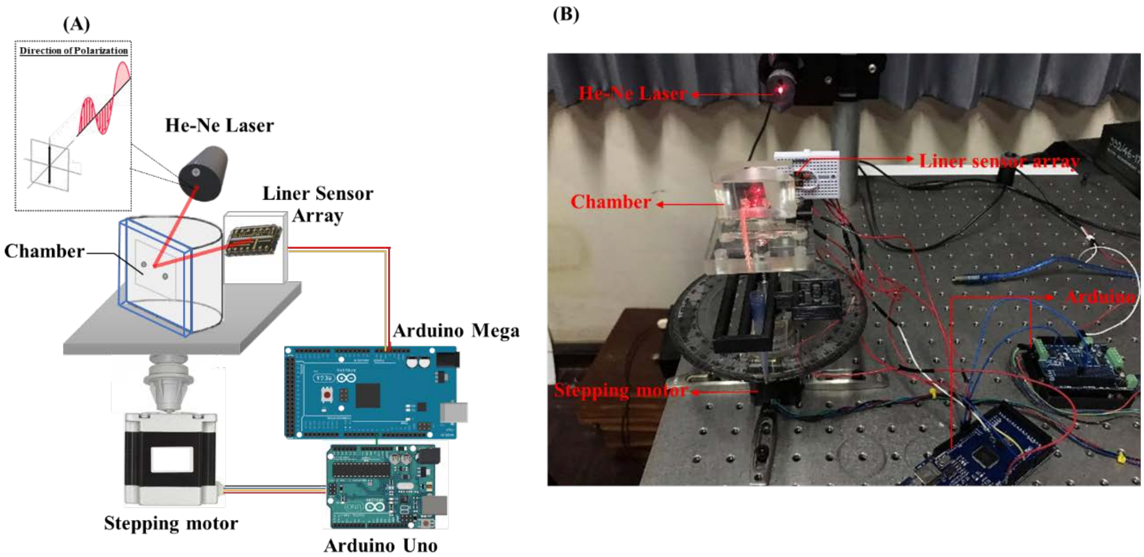

Figure 2 The developed method and set up for measuring refractive indices of aqueous solution (A) a schematic diagram and (B) an actual picture of the setup

3.2 Preparation of standard sucrose solutions

Standard sucrose solutions were prepared by dissolving sucrose in deionized water at various concentrations (10%, 20%, 30%, 40%, and 50% w/w or degree Brix).

3.3 Theory and Methods

One of the most straightforward methods to measure the refractive index of a solution is to measure the incident angle and the transmission angle and then calculate the refractive index using Snell's law. However, the method requires measurements of two angles with high accuracy and does not work very well with turbid solutions (Yunus & Rahman, 1988; Tan & Huang, 2015; Singh et al., 2013). In this work, the authors proposed a slightly different method of measuring the index of refraction optically by first measuring the critical angle θ_c and then using Snell's law to calculate the index of refraction.

For an internal reflection with the incident angle equal to or greater than θ_c, all the incoming energy is reflected into the incident medium; a phenomenon known as total internal reflection. It is evident from Fresnel's Equation (Hecht, 2017):

$$r_{\perp} = \frac{n_i \cos\theta_i - n_t \cos\theta_t}{n_i \cos\theta_i + n_t \cos\theta_t} \quad (1)$$

$$r_{\parallel} = \frac{n_t \cos\theta_i - n_i \cos\theta_t}{n_i \cos\theta_t + n_t \cos\theta_i} \quad (2)$$

Where $r_{\perp}$ denotes the amplitude reflection coefficient for light that has a polarization perpendicular to the plane-of-incidence and $r_{\parallel}$ is the amplitude reflection coefficient for light that has polarization parallel to the plane-of-incidence

n_i and n_t denote the refractive indices of the incident and the transmitting medium.

Graphically, it can be seen that both amplitude reflection coefficients reach their maximum value of 1.0 at the critical angle.

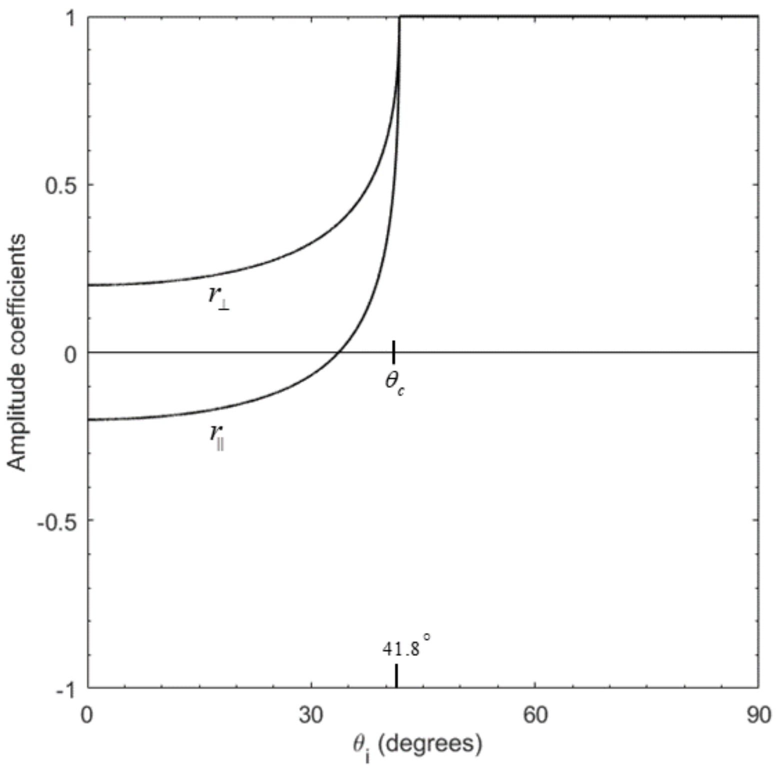

Figure 3 The amplitude coefficients of reflection as a function of incident angle. These correspond to internal reflection $n_t < n_i$ at an air-glass interface ($n_{ti} = 1/1.5$)

Once the critical angle is obtained, it can be used to calculate the transmitting medium's refractive index by Snell's laws.

$$n_t = n_i \times \sin\theta_c \tag{3}$$

In this work, the authors find the critical angle by setting the light's polarization perpendicular to the plane of incidence (shown in Figure 2(A)). The authors chose to work with the polarization in the perpendicular direction because the reflected light intensity using this polarization direction is more than that using the polarization parallel to the plane of incidence at almost all of the incident angles. The brighter the reflected light intensity, the more efficient the intensity measurement is. The reflection coefficient in the perpendicular direction $\boldsymbol{R}_\perp = \boldsymbol{r}_\perp{}^{\boldsymbol{2}}$ (Equation 1 and Figure 3) continue to increase with increasing incident angles, while the reflection coefficient in the parallel direction $\boldsymbol{R}_\parallel = \boldsymbol{r}_\parallel{}^{\boldsymbol{2}}$ decreases to zero initially before making a sharp increase with increasing incident angles. The light ray was incident on the microchamber containing the aqueous solution while the intensities of the reflected beam at various incident angles were measured by the linear sensor array. The critical angle was the smallest incident angle that produced the maximum measured intensity of the reflected light as shown in Figure 3. Then, the refractive index was calculated by using Equation 3.

4. Results and Discussion

Figure 4(A) shows the apparent light intensity measured by the linear sensor array. The light is reflected from the interface between the bottom of the half-cylinder glass block (n = 1.50) and sucrose solutions with various concentrations in the microchannel. For comparison, the authors simulated the reflected light intensity by finding the square of the $r_{\perp}$ (Equation 1) in Matlab (Mathworks Inc., USA) reported refractive indices in the range of 1.3330 to 1.4201 at various concentrations for sucrose solution (Misto, Mulyono & Cahyono, 2019). Qualitatively, our experimental data show a similar pattern of increasing critical angles (at which the apparent light intensity first reached the maximum value) with increasing sugar concentration. Then, the measured critical angles were used to calculate the indices of refraction from Equation (3). The values are shown in Table 1 along with another set of values reported in a prior publication (Misto, Mulyono & Cahyono, 2019).

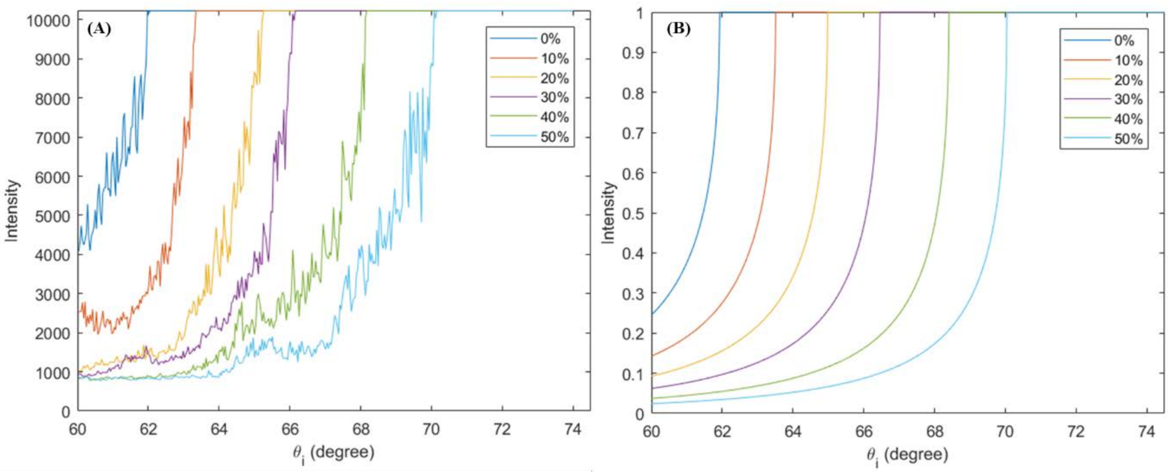

Figure 4 intensity as a function of incident angle of sucrose with various concentration (A) experimental data (B) simulated data

Table 1 The measured critical angles, the measured and the reported refractive indices of sucrose

% by weight/Degree Brix (w/w)	Critical angle (degrees)	Measured Refractive indices	Reported Refractive indices
0	61.92±0.04	1.3234±0.0005	1.3235
10	63.44±0.13	1.3417±0.0015	1.3425
20	65.04±0.09	1.3599±0.0010	1.3592
30	66.16±0.12	1.3720±0.0013	1.3751
40	68.18±0.11	1.3925±0.0010	1.3947
50	70.06±0.07	1.4100±0.0006	1.4099*

*Remark: The actual refractive index of 1.4099 at 50 %w/w (degree Brix) is not available in the publication being cited. Therefore, the authors extrapolated the value from the actual measured data presented in that citation.

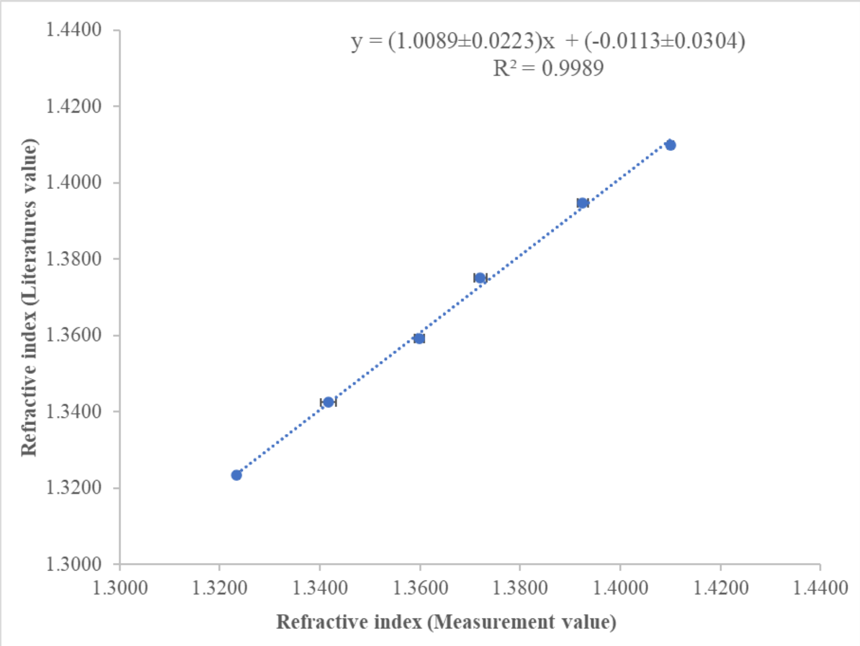

Figure 5 The comparison of refractive indices measure by our method and the one in a prior publication (Misto, Mulyono & Cahyono, 2019)

From Table 1 and Figure 5, it can be seen that the calculated refractive indices of sucrose solutions at various concentrations agree well with the values reported in prior work (Misto, Mulyono & Cahyono, 2019). Therefore, it is confirmed that our method, despite being simple, is powerful and highly accurate. Besides, it can be seen from Table 1 that the standard deviations of the measured refractive indices are in the range of 0.04-0.10 %, which are well below the limit set by the Association of Official Agricultural Chemists (AOAC), ranging from 1.3 to 2.7% depending on the concentrations (AOAC, 1993). Since the sugar contents listed on the containers of soft drinks and juices are in whole percentage values, this suggests that our method could be used with confidence to quantify sugar concentrations in real liquid samples. Finally, the authors plot a calibration graph between the concentration of sucrose and the measured refractive indices. The plot in Figure 6 shows a linear relationship between the two sets of values suggesting that the sucrose concentration can be quantified optically by measuring the solution's index of refraction using our simple yet powerful method. The authors later verified the accuracy of our setup by measuring another sucrose solution with 25% sugar content and obtain the measured value of 24.65% (data not shown), which accounts for only a 1.4% deviation, which still is acceptable according to AOAC (~1.6% at 25% sugar content). There is only one caveat that this method can measure the concentration of sucrose up to 50% only because the setup cannot accommodate the incident angle larger than 72 degrees.

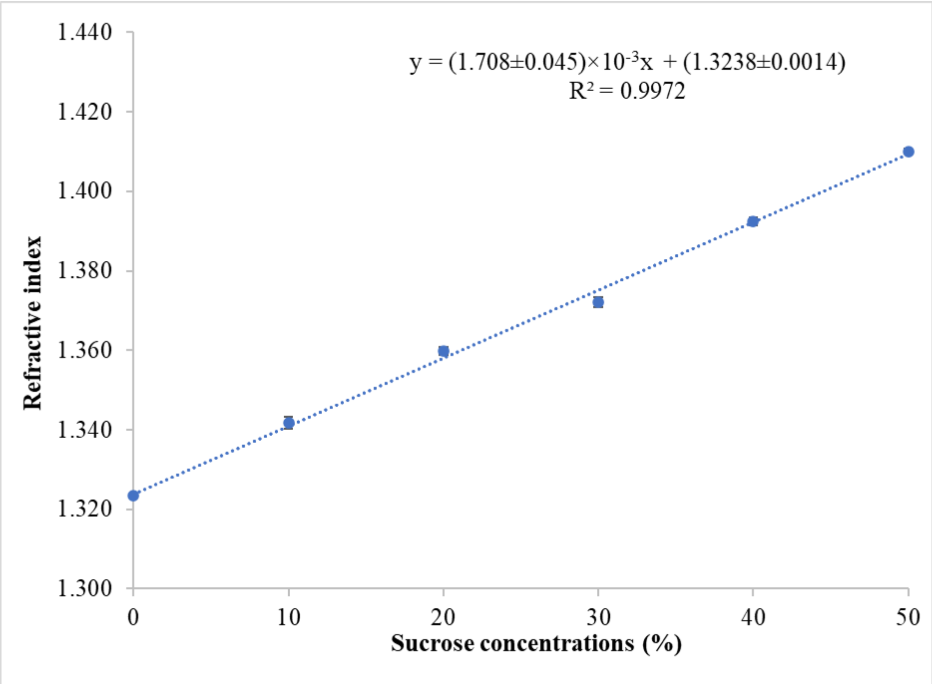

Figure 6 Calibration plot for the variation of refractive index with the concentration of sucrose solution up to 50%

5. Conclusion

In the present paper, the authors have successfully developed a novel and simple yet powerful method and set up the capability of measuring refractive indices of liquid solutions with high accuracy using just microliters of solutions. Using sucrose solution as a model, the authors have successfully determined the concentration of the sucrose solutions up to 50% by measuring the refractive indices. This finding will be beneficial for quantifying the concentration of sucrose solution optically. Furthermore, our method has the potential to determine the refractive indices of other solutions in the range of 1.3234 to 1.4100 with good accuracy when compared with values reported in prior work. Besides, the precision of our method is acceptable according to a regulation set by AOAC and, with some improvement on user-friendliness, could be used with confidence in measuring sucrose contents in liquid samples even when the liquid is extremely turbid.

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