Good practice report

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Introducing spectrophotometry in the school lab employing LEGO bricks and LEDs

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<https://orcid.org/0000-0002-4627-3034>.**Abstract:**

In this paper we present the simple process of manufacturing a spectrophotometer based on an assembly of LEGO bricks and LED lights. The procedure is simple and the cost is extremely low. Spectrophotometer's capabilities were tested employing two analytical chemistry problems as a reference. In the first one we investigated the concentration of a blue food dye in an isotonic beverage. The results obtained are compared with those of a commercially available instrument. In the second one we searched for the concentration of two dyes present in a shampoo. The results suggest that simple DIY spectrophotometers can replace the need of purchasing expensive analytical instruments, at least for the secondary school level.

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Introduction

Spectrophotometry is one of the most basic and simple analytical techniques aimed at measuring compounds' concentration based on their color. It is an integral part of both university curricula and many upper secondary education programs (age group 15–18) (CollegeBoard 2014). The instruments used for this purpose have evolved considerably over the last two decades in terms of their accuracy and simplicity of use. In fact, portable instruments are also available. Nevertheless, their purchase costs remain particularly high, which makes the application of the corresponding laboratory techniques to the average school class essentially prohibitive. To this end, in recent years, the construction of simple DIY-type devices has been proposed, which significantly lowers the required cost (Albert, Todt, & Davis, 2012; Vanderveen, Martin, & Ooms, 2013). Moreover, some researchers have proposed the use of "smart" mobile phones as optical detectors (Kehoe & Penn, 2013; Knutson et al., 2015; Kuntzleman & Jacobson, 2016; Montangero, 2015; Morales, Confessor, & Gasparotto, 2015; Scheeline, 2010).

In the present study we present the construction of a DIY spectrophotometer in which the cell chamber is made of LEGO bricks, while the role of both the light source and the detector is played by LEDs (Wagner II, 2016). LEDs in principle can convert incident light to a potential difference (voltage), thus acting as a photo-voltaic cell (Lindsay & Paton, 1976; Mims III, 1992). However, there are only few references in the literature employing LEDs in this sense (Asheim, Kvittingen, Kvittingen, & Verley, 2014; Kvittingen, Kvittingen, Melø, Sjursnes, & Verley, 2017; Pokrzywnicka, Koncki, & Tymecki 2009; 2010). Furthermore, we employed the spectrophotometer we constructed in order to find the concentration of the blue dye E133 in a well-known isotonic beverage ("energy drink") as well as the concentration of both E133 and E102 (yellow) dyes, which create the green color of a commercial shampoo.

It is our strong belief that the introduction of simple, low cost devices, as the one we describe, in the upper secondary school would further engage students' interest in STEM lessons.

Theoretical background

In absorption spectrophotometry, light passes through a sample and then its intensity is measured and compared to the original. Let a light beam of a certain wavelength λ and of specified intensity I_0 pass through a

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solution of length b of a colored sample of concentration C (Figure 1). The light loses intensity, which becomes I . Thus, the following quantities are defined (Skoog, West, Holler, & Crouch, 2014; Strobel & Heineman, 1989):

a. Transmittance T :

$$T = \frac{I}{I_0} \quad (1)$$

b. Absorbance A :

$$A = -\log T = -\log \left(\frac{I}{I_0} \right) \quad (2)$$

Figure 1: The principle behind absorption spectrophotometry. Green light of I_0 passes through a dilute red dye solution. Part of the incident radiation is absorbed by the sample, and $I < I_0$.

For relatively dilute solutions the concentration of the colored substance in the sample is proportional to its absorbance. The mathematical relationship that describes the phenomenon is known as the Beer-Lambert law:

$$A = \varepsilon \cdot b \cdot C \quad (3)$$

where ε is the molar absorptivity of the sample depending on the wavelength λ .

The food dyes

In the present work we employed two widely used synthetic food dyes: brilliant blue FCF and tartrazine. Their basic properties are summarized in Table 1, while their formulas are depicted in Figure 2.

Table 1: The food dyes that we used.

Food dye	E number ^a	C.I. ^b	M_r	$\lambda_{\max}$
Brilliant blue FCF ^c or blue 1	E133	C.I. 42090	792.85	630 nm
Tartrazine or FD & C ^d yellow 5	E102	C.I. 19140	534.36	427 nm

^aE, The colorant has been approved to be used as food additive within the European Union and the European Free Trade Association.

^bC.I., Color Index International.

^cFCF, For Coloring Food, a term used to differentiate a food-grade *versus* an industrial grade batch of dye.

^dFD & C, The colorant has been approved by the American Food and Drug Administration for use in food, drugs and cosmetics.

Figure 2: The dyes employed. (a) Brilliant blue (E133). (b) Tartrazine (E102).

Experimental

Safety rules

There are no particular security issues that should be taken into consideration when performing the following procedure.

Materials

Most LEGO bricks used are common pieces of 4×1 and 2×1 dimensions. The setup was placed on a LEGO base of 16×6 . The two LEDs necessary for each experiment were fitted inside the hole of 2×1 LEGO bricks. Bricks with code numbers 370021, 370023, 370026 and 4211440 are suitable for that purpose (Figure 3). Caution should be provided to the fact that after their insertion, the LEDs (Figure 3b) are practically impossible to be pulled out without destruction.

Figure 3: The 2×1 sized bricks used to adjust the LEDs. (a) Before adjustment. (b) After adjustment.

The LEDs used were common 5 mm diodes, with a transparent housing. They were powered by two 1.5 V batteries connected in series, while potential differences were recorded by a common voltmeter. The red LED in order to operate optimally as a light source was connected in series with a 100 Ω resistor. This was not necessary for the blue LED.

The cuvettes employed were common tubular ones. The commercial single beam spectrophotometer used for reference purposes was purchased by Motic. Finally, for the preparation of the reference solutions of the two dyes, concentrated food dyes were used. These dyes were purchased from a local store and they are prepared by Vahiné. The product, which is depicted in goo.gl/HSWJnY, contains three 6 mL vials of concentrated blue (E133), red (E122) and yellow (E102) food colorants, respectively. Both blue and yellow dyes own a mass fraction of 0.595 % w/w in the concentrated solutions, as we were informed by the manufacturer.

The “energy drink” employed as well as the shampoo specimens were also purchased from a local store.

Instrument construction

In Figure 4 two intermediate stages of the construction are depicted, while in Figure 5 the final analytical device is shown.

Figure 4: (a) Constructing the cell chamber. The foam ring depicted was used in order to stabilize the cuvette in place. (b) The chamber in its final form. The illuminated red LED acts as a detector.

Figure 5: The final instrument. Each LEGO level apart from the first (white frame) and the last (light blue lid) is 1 cm high.

From these two images it becomes apparent that the two LEDs were placed one across the other within the cell chamber. Their position is indicated by the two orange bricks on the base. One LED acts as a light source while the opposite one acts as a detector. Thus, when the detector LED is illuminated, a voltage V is developed, which is measured by the connected voltmeter.

For each sample measurement, V_i , the corresponding measurement for the solvent was also recorded, V_s . Subsequently, the permeability of the samples were calculated based on the relationship (4).

$$T_i = \frac{V_i}{V_s} \quad (4)$$

The brilliant blue dye has a maximum absorption wavelength at 630 nm. To minimize errors, the light source must emit monochromatic light of this certain wavelength in all measurements. LEDs do not emit monochromatic light. However, the light they emit usually extends over a small wavelength range around a characteristic value. For this series of experiments, we chose red LEDs with $\lambda_{em} = 625$ nm as a light source. Based on the LED operation principle (Wagner II, 2016), the detector lamp should have a λ_{em} value greater than or equal to the source light ($\lambda_{detector} \geq \lambda_{source}$). In other words, a red LED can be used as a detector of all other LEDs emitting to the visible spectrum. So, in this case, as a detector, we chose a red LED.

The yellow dye E102 has a maximum absorption wavelength at 427 nm. In order to detect it, we chose as the light source a blue LED with $\lambda_{em} = 463$ nm. Unfortunately, we did not have a LED with a λ_{em} closer to the desired one. However, at a wavelength of 463 nm, the molecular absorbance of the dye is still quite high (about 60 % of ϵ_{max}) (Sha, Zhu, Feng, & Ma, 2014). On the other hand, we found some difficulty to achieve high repeatability to our measurements when we employed a blue LED as the detector. For that reason we used a red LED as a detector also in this case.

Particular care was taken to ensure that the cylindrical 10 mm cell remained steady throughout the measurements. To achieve this, a sponge ring (Figure 4a) was used. In this way it was possible to maintain the optical path at the same cell position constantly. Instead of a cylindrical cuvette, a square one could also be used. A square type cell would not have the aforementioned stabilisation problems. Unfortunately, we did not have such a cell in our disposal.

Application 1: determination of the E133 dye concentration in a blue colored isotonic drink

Several of the isotonic beverages that are widely marketed are blue. Color comes from the addition of the synthetic dye brilliant blue (*see above*). In order to determine this dye's concentration in a widely available "energy drink", five reference solutions of this dye in water were initially prepared. Subsequently, their absorbance, along with the absorbance of the isotonic drink in question was measured in both our spectrophotometers, e.g. the one constructed with LEGOs and LEDs and the commercial one. Our results are summarized in Table 2 and Figure 6.

Table 2: Voltages (V_i) for the reference solutions and the sample and V_{water} for the solvent used), transmittance (%T) and absorbance (A) values measured for the solutions under study in the two spectrophotometers.

Sample	Concentration, μM	Motic		LEGOs/LEDs			
		%T	A	V_{water}	V_i	%T	A
1	8.38	8.0	1.097	171	14	8.2	1.09
2	6.70	13.6	0.866	172	23	13.4	0.874
3	5.03	21.4	0.670	173	37	21.4	0.670
4	3.35	35.5	0.450	172	61	35.5	0.450
5	1.68	60.4	0.219	175	100	57.1	0.243
Isotonic beverage		16.0	0.796	175	28	16.0	0.796

Figure 6: Absorbance of light passed through the dye samples as function of their concentration for (a) the commercially available single beam spectrophotometer and (b) our LEGOs/LEDs spectrophotometer.

As it is apparent both from the experimental values of Table 2 and the relative diagrams of Figure 6, the two spectrophotometers provided almost identical results. In both instruments the isotonic beverage sample gave a transmittance value of 16.0 %. Therefore, the concentration of E133 dye in the isotonic drink bought was 6.07 μM or 2.41 mg/500 mL. Moreover, in both cases, the molar absorptivity value ϵ calculated (given by the slope of the $A = f(C)$ plots) is very close to the one reported in the literature ($\epsilon = 130.000 \text{ M}^{-1} \text{ cm}^{-1}$) (Sigmann & Wheeler, 2004).

Application 2: determination of the content of a green colored commercially available shampoo in blue (E133) and yellow (E102) dyes

The usual green color of shampoos, as well as the one of several commercially available liquid detergents, comes from mixing blue and yellow dyes. The blue dye is usually brilliant blue, while the yellow one is often tartrazine. We chose a widely available green shampoo containing these two dyes (referred to as C.I. 42090 and C.I. 19140 in the package) in order to further test our spectrophotometer.

When two chromophore groups are present in a sample, the sample's absorbance at a particular wavelength is due to both. Let λ_1 be the maximum absorption wavelength of the first dye (brilliant blue, bb), λ_2 the maximum absorption wavelength of the second one (tartrazine, tt), $\epsilon_{bb}^{(1)}$ and $\epsilon_{bb}^{(2)}$ the molar absorptivities of bb in λ_1 and λ_2 , respectively, $\epsilon_{tt}^{(1)}$ and $\epsilon_{tt}^{(2)}$ the molar absorptivities of tt in λ_1 and λ_2 , respectively, A_1 the sample's absorbance in λ_1 , A_2 the relative absorbance in λ_2 , C_{bb} the concentration of bb in the sample and C_{tt} the concentration of tt in the sample. The following equations hold:

$$C_{bb} = \frac{\left(A_1 - \frac{\epsilon_{tt}^{(1)}}{\epsilon_{tt}^{(2)}} \cdot A_2 \right)}{b \cdot \epsilon_{bb}^{(1)} \cdot \left(1 - \frac{\epsilon_{tt}^{(1)}}{\epsilon_{tt}^{(2)}} \cdot \frac{\epsilon_{bb}^{(2)}}{\epsilon_{bb}^{(1)}} \right)} \quad (5)$$

$$C_{tt} = \frac{\left(A_2 - \frac{\epsilon_{bb}^{(2)}}{\epsilon_{bb}^{(1)}} \cdot A_1 \right)}{b \cdot \epsilon_{tt}^{(2)} \cdot \left(1 - \frac{\epsilon_{tt}^{(1)}}{\epsilon_{tt}^{(2)}} \cdot \frac{\epsilon_{bb}^{(2)}}{\epsilon_{bb}^{(1)}} \right)} \quad (6)$$

In order to find the molar absorptivities ratios depicted in relations (5) and (6) two series of dye solutions (brilliant blue and tartrazine) of known concentration have been prepared. The absorbance of these solutions were then measured in our LEGOs/LEDs spectrophotometer in both wavelengths λ_1 and λ_2 . For the measurements in λ_1 a red LED was used as both the light source and the detector ($\lambda_{em} = 625 \text{ nm}$), while for λ_2 measurements a blue LED ($\lambda_{em} = 463 \text{ nm}$) was employed as the light source and a red one as the detector (see above). In addition to that, since shampoo samples are not entirely optically transparent, we considered it not appropriate for our reference solutions to be prepared with water as a solvent. For this reason we purchased a colourless shampoo of the same brand with the same composition according to its ingredients label and we prepared a solution consisted of 3 parts of colourless shampoo and two parts of distilled water. This dilution was necessary since the reference solutions were more easily prepared if they are not too viscous. In order the aforementioned mixture to become a homogeneous solution it was stirred for sufficient time. Finally, we employed the latter as

the main solvent throughout our experiments. Similarly, when calculating tartarazine, our sample, before being measured, was diluted with water at the same ratio.

The values obtained are summarized in Table 3 and Table 4, while the corresponding reference plots are shown in Figure 7. Moreover, using the more concentrated among our reference solutions we measured $\varepsilon_{bb}^{(2)}$ and $\varepsilon_{tt}^{(1)}$. Thus, we derived $\varepsilon_{bb}^{(2)} / \varepsilon_{bb}^{(1)} = 0.028$ and $\varepsilon_{tt}^{(1)} / \varepsilon_{tt}^{(2)} = 0.005$.

Table 3: Voltages ($V_{3,2}$ for the solvent mixture and V_i for the samples), transmittance (%T) and absorbance (A) values measured for the solutions under study in the case of the brilliant blue dye.

Sample	Concentration, μM	LEGO/LEDs ($\lambda_{em} = 625 \text{ nm}$)			
		$V_{3,2}$	V_i	%T	A
1	4.11		54	30.3	0.518
2	3.28	178	70	39.3	0.405
3	2.46		84	47.2	0.326
4	1.64		103	57.9	0.238
5	0.82		124	69.7	0.157
Green shampoo		178	94	52.8	0.277

All dilutions have been prepared with the 3:2 mixture. Shampoo sample was not diluted.

Table 4: Voltages ($V_{3,2}$ for the solvent mixture and V_i for the samples), transmittance (%T) and absorbance (A) values measured for the solutions under study in the case of the tartrazine dye.

Sample	Concentration, μM	LEGO/LEDs ($\lambda_{em} = 463 \text{ nm}$)			
		$V_{3,2}$	V_i	%T	A
1	24.6		88	54.0	0.268
2	19.6	163	98	60.1	0.221
3	14.7		115	70.6	0.151
4	9.8		127	77.9	0.108
5	4.9		146	89.6	0.048
Green shampoo (3:2)		163	125	76.7	0.115

All dilutions have been prepared with the 3:2 mixture. Shampoo sample was also diluted with water at the same ratio.

Figure 7: Absorbance of light passed through the reference solutions of (a) the brilliant blue dye and (b) the tartrazine dye. All measurements have been performed with our LEGOs/LEDs spectrophotometer.

From the derived plots, the molar absorptivities ratios calculated above and the relations (5) and (6), the concentration of the two dyes can be calculated. Caution should be given to the fact that the sample's absorbance values in the two wavelengths should be corrected for the systematic errors before employing equations (5) and (6). The errors are provided by the equations given as insets in Figure 7.

The concentration of the two dyes in the commercially available product (green shampoo) was calculated to be $1.98 \mu\text{M}$ for the brilliant blue and $17.5 \mu\text{M}$ for the tartrazine, respectively. In other words a bottle of green colored shampoo (500 mL) contains 0.78 mg of E133 dye and 4.68 mg of E102 dye.

Conclusion and perspectives

One of the most useful analytical instruments in a chemistry laboratory is the visible spectrophotometer. In this work we described the construction of a simple and particularly low cost spectrophotometer, the main parts of which are LEDs, LEGO bricks, batteries and a voltmeter. Moreover, we showed that our spectrophotometer can be used to design and perform simple laboratory experiments, providing two possible educational scenarios. These scenarios were safe for the students and all the materials purchased were readily available in local stores. Therefore, DIY spectrophotometers can replace expensive analytical instruments at a school experiment level. As a result analytical techniques can be introduced to the higher secondary education and accordingly the students' engagement to laboratory work should be raised.

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