
When the kitchen turns into a physical chemistry lab

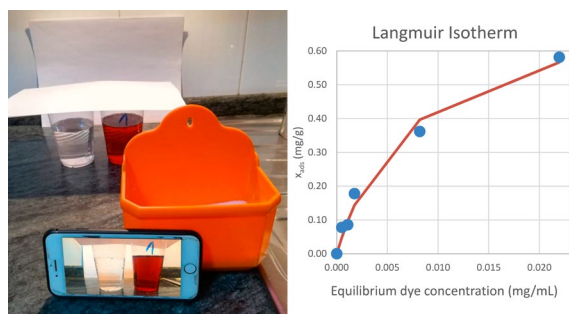
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5 ABSTRACT

We present our experience with transferring a four-day photometry and dye-adsorption laboratory experiment to the kitchens of students of Applied Thermodynamics from our degree in “Industrial Chemical Process Engineering”. The students designed and built a double-beam photometer using their smartphones and household materials, then prepared a series of dye-solutions with well-known relative concentrations and measured the absorbance-concentration calibration curve. After 24 hours of adsorption to kitchen absorbent paper the solutions were visibly decolorized. The students were able to determine the equilibrium dye concentrations from absorbance measurements and to calculate the adsorption isotherm. This home-lab experiment allowed them to keep on track with their lessons during the severe lock down in Spain due to COVID-19. They were highly motivated and achieved the learning objectives to a similar degree as in years before with conventional lab equipment.

GRAPHICAL ABSTRACT



KEYWORDS

First-Year Undergraduate / General, Second-Year Undergraduate, Chemical Engineering, Physical Chemistry, Surface Science, Thermodynamics, UV-Vis Spectroscopy, Hands-On Learning / Manipulatives, Laboratory Instruction

On Saturday, 14 March 2020 a severe lockdown was imposed in Spain as a reaction to the
25 alarming number of new COVID-19 infections. Our students were trapped wherever they happened to
be that weekend, at their parents' home, with friends they were visiting or at their student apartments.
Suddenly mobility was strongly restricted, and most personal interaction was reduced to those they
lived with at that moment. Our students had to continue their studies with the resources they
happened to have access to – some were lucky and had taken their computer and notes with them for
30 the weekend, but others not. Most found themselves unexpectedly far away from their place, where
they used to concentrate on their studies, and also away from their friends and fellow students. The
same happened to us as teachers, who were allowed to access our offices and labs only once, the
morning of the following Monday 16th of March, to fetch all we needed to continue our lessons from
home, with nearly no prevision on how long and under which conditions this new situation would last.

35 Being responsible for the four-day laboratory experiments accompanying the subject of “Applied
Thermodynamics” of the second year of the “Bachelor Degree in Industrial Chemical Process
Engineering” of the University of Santiago de Compostela in Spain, we assumed from the beginning
that the students would not be allowed to access the labs or able to perform the experiments the way
we did in previous years. Our first impulse was to substitute the laboratory practices with simulations
40 or calculus. However, we felt that our students had a right to get the best possible instructional
experience, and as teachers we had to do whatever it would take to help our students to succeed and
to stay on track. The syllabus includes two laboratory experiments, (1) the determination of a liquid-
vapor phase diagram of a binary system and (2) the construction of an adsorption isotherm of a dye at
the solution/solid interface. Due to the overall limitations during the COVID situation we decided to
45 select only one of these two experiments and, the second seemed much more easily transferable to a
home-lab, although this still presented quite a few challenges.

Several adsorption isotherm experiments for student labs have been published¹⁻⁷ We implement
that of Guirado et al., which comprises the following steps:³ (1) Prepare a stock solution of the dye and
10 diluted solutions with different dye concentrations. (2) Scan the absorption spectrum of the dye to
50 determine the wavelength of maximal light absorbance and then measure the absorbance at this
selected wavelength of each diluted solution. (3) Construct the absorbance-concentration calibration

curve. (4) Trim and weigh adsorbent (filter paper) and add it to a certain volume of each diluted solution. (5) Stir the solutions 24h in order to achieve equilibrium. (6) Measure again the absorbance of the solutions. (7) Process the data to obtain the adsorption isotherm, and discuss the results.

55 Due to the severe lock-down we found ourselves in an “Apollo 13” emergency - so we went to our kitchens at home and started to figure out what could be done with the means students have at hand. The main challenges for our home-lab experiment were

- (1) to construct a photometer
- (2) to find a house-hold dye and an adsorbent with suitable properties
- 60 (3) to define a procedure for the quantitative preparation of the concentration series without balances or volumetric flasks
- (4) to remotely guide the students through the experiment.

In the following we address each of these topics.

Please keep in mind that this is a contribution to the Special Issue on *Insights Gained While Teaching Chemistry in the Time of COVID-19*. It is not our aim to describe a full-fledged home lab experiment, but to show how we solved the COVID-19 emergency situation and made it possible that our students could perform one of their laboratory experiments with the means they had at home. We had to solve this ourselves being at home, under heavy restrictions, both in time, mobility and equipment. This is our first and, hopefully, last attempt to perform the experiment with students
70 under these circumstances. We have no information about experimental precision or reproducibility, nor comparative results over several years. Without doubt, many aspects could be improved and much better results would be obtained in a well-equipped student lab.

Most aspects of the experiment had been published before in this Journal with the exception of the mobile phone *double-beam* photometer, which is a new concept we will present in more detail in a
75 forthcoming publication.

THE HOME-LAB EXPERIMENT

An improvised double-beam mobile photometer

Several photometers based on mobile phones have been described, also in this Journal.⁸ However, 80 the adsorption experiment requires a relatively wide range of absorbance to be measured with sufficient precision and especially with good repeatability between days. We opted for an open construction without any special elements, which allows the students to improvise with what they have at hand.

A photometer requires at least four basic elements: a light source, a cuvette, a color filter and a 85 light detector. The cameras of mobile phones detect light intensity through red, green and blue color filters and cover thus two important design components. As cuvettes, any cylindrical plastic cup or glass tumbler can be used. As a light source we initially proposed indirect lighting by a table lamp, or a flashlight directed on or through a sheet of white paper. However, the students themselves later came up with the idea to use a PC monitor or a tablet as a much more homogenous light source.

90 The design of a simple photometer setup within clearly defined conditions and goals and within limited time is an excellent example of a *problem solving task* as described by cognitive science.^{9,10} The simple but open setup requires students to start to think about the relevant aspects of the problem and to be creative to solve it. The quality of the data they will obtain depends on their own understanding and initiative and not on their ability to reproduce a typical script using lab equipment 95 as black boxes. By acknowledging that the students' suggestions to improve the experimental design were valid and worth pursuing, we raised the chances that they will remember what they learned and be able to transfer it to novel problems in the future.

Our first tests with this simple setup, however, immediately revealed a problem of typical camera applications when used for quantitative light intensity measurements. Light intensity readings taken 100 from two consecutive photos of a clear water reference sample and of a much darker colored dye sample are not comparable because of the automatic exposure and white balance compensations of the camera. The photo of the darker and strongly colored sample will be exposed longer and with a different white balance than the brighter neutral reference photo. The observed changes in the RGB intensities are thus not only due to light absorption by the solution but also to changes in the camera

105 settings. To solve this problem, camera apps allowing for manual control of exposure and white balance would have to be installed in the private mobile phones of the students and often require payment of a fee or include advertisements. And, most importantly, they would add complexity to the experiment with no real educational advantage. Furthermore, separate measurements of the reference and the sample suffer also from typical short-term variations of the improvised light source.

110 After some tests we found a new and, we think, didactic way to solve this problem. The proposed setup represents a model⁹, which improves the student's understanding of commercial photometers and other similar instrumentation and the typical compensation and reference procedures they apply.

To obtain comparable intensity readings of reference and sample we take a single photo of both cuvettes together, mimicking the setup of *double-beam* photometers. This assures that all camera settings are always the same for reference and sample, and compensates for variations in the light intensity due to movements or fluctuations of the light source. As trivial as it may seem, this new setup solved the problems we describe above and allowed for sufficient precision in the absorption measurements.

120 We proposed the setup of Figure 1 as a basis for student's own improvised photometers. It uses the lamps of a kitchen hood as light source and plastic cups as cuvettes.

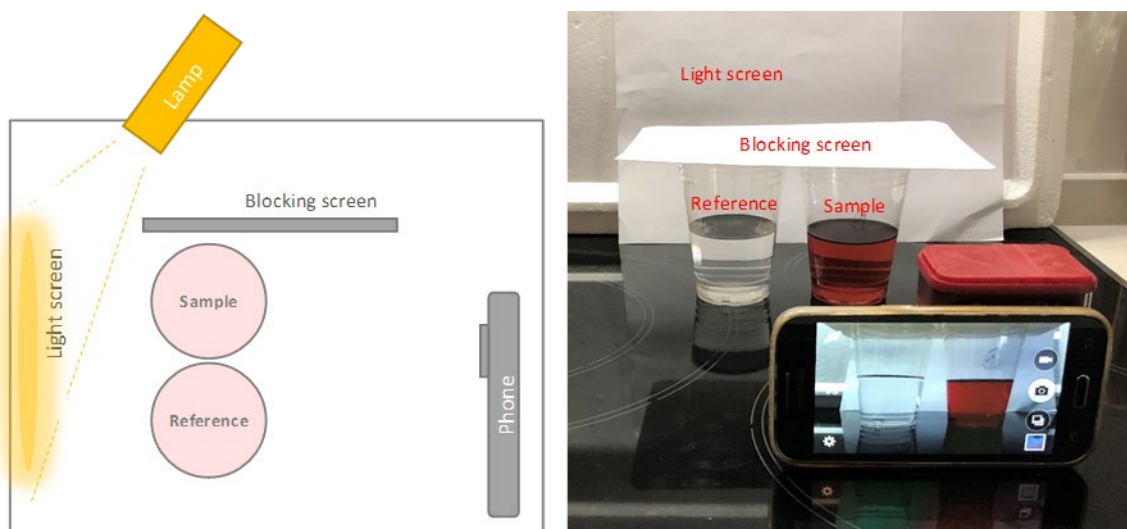
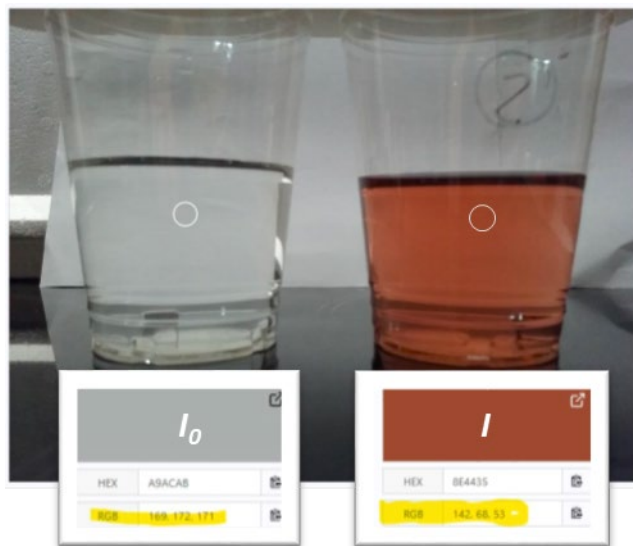


Figure 1: Setup proposed to the students for their improvised double-beam photometers. The mobile phone takes a photo of sample and reference cuvettes against a white background homogeneously illuminated by a table lamp or other conveniently placed light source. The blocking screen prevents direct light from reaching the samples and the camera.

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Once the photo is taken it can be uploaded to a PC and analyzed by free color picker web applications, such as <https://image-color.com/>. Sampling the mean value of many pixels within a region in each cuvette reduces the noise and small variations in the background luminosity (See Figure 2).



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Figure 2: A color picker application returns the RGB intensity values in selected regions (white circles) of the reference and sample cuvettes.

The intensity values of reference and sample can be processed applying different compensations. In the simpler case, the transmittance T and the absorbance A of the sample is calculated directly from the ratio of sample intensity I to reference (blank) intensity I_0 :

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$$T = \frac{I}{I_0} \quad A = -\log_{10} T \quad (1)$$

The students observe rapidly that a measurement with water as blank in *both* cuvettes does not yield the expected transmittance of 1.0 nor an absorbance of 0.0 – the intensity I of water as a sample does not usually equal the intensity I_0 of the reference water, due to (small) variations in the background illumination. The solution adopted in commercial double-beam photometers is the *baseline correction*. The ratio between sample and reference intensities from a *baseline* measurement with water in *both* cuvettes is used to correct for the variation in the background illumination and assures a transmittance of $T = 1$ for a water sample.

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$$T = \left(\frac{I}{I_0} \right) / \left(\frac{I}{I_0} \right)_{blank} \quad (2)$$

145 This correction may not have a significant influence on the overall result of this experiment, as other factors introduce much bigger errors, but it may be very educational to introduce this correction in the analysis.

The mobile photometer is further limited by the 8-bit resolution of the camera, with intensity values between 0 and 255 units. Assuming an optimal reference intensity of $I_0 = 250$ and a lowest
150 detectable sample intensity of $I = 5$, the minimum detectable transmittance value is $5/255 = 0.02$ and thus the maximum absorbance $-\log_{10} 0.02 = 1.7$. This range will be further limited by the *stray light* reaching the camera directly without passing through the solutions due to reflections on the cuvettes or from ambient light. Students used many different camera models (iPhone, Samsung Galaxy, Redmi Note or Huawei P30) with similar success.

155 The adsorbate - adsorbent pair

The dye as adsorbate must comply with several conditions; to absorb light in the visible range, to be well soluble in water, to show sufficient adsorption onto the adsorbent, to be safe to use and, of course, to be available to the students during the COVID-19 lock-down. We tried several dyes we had at hand, from food-dyes, dyes extracted from spices or food, and even dyes covering colored chocolate
160 candies. The dyes we tried worked mostly great for the calibration curve, but they adsorbed poorly on kitchen paper. Accidentally spilled red wine during a dinner led us to the solution. At least in Spain, red wine is present in most house-holds or at least easily available from neighbors or a nearby store. It is intensely colored and stains fabric easily. With an adequate safety instruction to use the wine exclusively for the preparation of the solutions no further problems were expected. Alternatives to red
165 wine may have been other filtered transparent fruit juices, such as grape, cranberry or elderberry juices. Red wine and fruit juices are very variable in their chemical composition, which is one of the possible reasons for the great variety of results the students obtained. Under normal conditions we would recommend to use well defined pure dyes such as food-coloring dyes or other synthetic dyes as proposed by several authors.¹⁻⁷

170 In the lab we use filter paper as adsorbent, as recommended by other authors,³ but this was not available to our students. Our experiments showed that red wine adsorbed well on kitchen absorbent paper. We found the wine to adsorb less to tissue paper. Toilet paper is not recommended as it disintegrates in water leading to turbidity which interferes with the absorbance measurement - one of the sources of error which can be discussed.

175 [Preparing a precise concentration series with kitchenware](#)

Both the absorbance calibration curve and the adsorption experiment require a series of solutions with different known concentrations of the adsorbate. Most students had no balance or calibrated volumetric material with sufficient precision at hand. Being a student laboratory exercise, we didn't need to obtain accurate results, but we needed good relative concentration values in order to assure 180 that both the calibration curve (a straight Lambert-Beer line) and the adsorption isotherm had the correct form. We solved this with a stepwise extraction-dilution procedure¹¹ with a dilution factor of two which required only a comparison of the liquid heights.

As explained in Figure 3 the students start preparing solution 1 with the highest dye concentration. They fill cup 1 with water at least double of the height they later need for the 185 absorbance measurement (3-4 cm). Then they add some dye and measure the RGB light intensities with the improvised photometer. Red wine is a mixture of dyes, mainly of the anthocyanin family¹², and shows a broad absorbance from green to blue. The students readily observe that the red channel is much less affected by the absorbance of the solution than the green or blue channels. We treated the fundamentals of color absorption briefly during the tutorial sessions and in the accompanying 190 learning sheets. Adding more dye, the students adjust the intensity in the blue channel to 5 to 10 units, assuring that the sample with highest absorbance is just within the measurement range of the photometer.

For the first dilution step, half of the solution is poured into cup 2, carefully adjusting the heights of both liquids (Figure 3, panels a and b). Then a third cup is filled with water to the same height as 195 the other two, which is then added to cup 2 (panel c), diluting the solution to half the concentration. These extraction-dilution steps are repeated until all 5 samples are obtained, with an overall dilution

of $2^4 = 16$, sufficient to cover the necessary concentrations for the adsorption isotherm and still well within the resolution of the photometer.



200 Figure 3: Extraction-Dilution steps for the concentration series.

Once the relative concentration series is obtained, the absolute concentration values have to be determined. For this aim we use a nominal value of $\epsilon = 20 \text{ mL}/(\text{mg cm})$ for the specific absorption coefficient of anthocyanines for blue light. This very approximate value was estimated with a commercial photometer from a volumetric solution of red wine and a reported typical concentration of 0.15 mg/mL of anthocyanines in red wine.^{13,14} The students determined the absorbance in the blue channel of sample 1 and calculated its anthocyanine concentration from the given nominal values of ϵ . (Those students who could measure the volume or the weight of the added wine with sufficient precision at home calculated the dye concentration of sample 1 directly.) Then they calculated the dye concentration of the other samples applying the known dilution factors. These values were then used for the absorbance-concentration calibration curve and the determination of the experimental value of ϵ . A typical mistake of the students was to calculate the anthocyanine concentration of all 5 samples from their absorbance values and the nominal value of ϵ . This results of course in a perfect calibration curve with a correlation coefficient of 1.00, which quite a few students presented proudly.

215 GUIDANCE OF THE STUDENTS THROUGH THE EXPERIMENT

The COVID-19 lock-down forced the students into a new unknown situation. Most of them never made experiments on their own, much less built their own instruments. The program had to be very detailed but also had to offer enough flexibility to be executed by each student on his or her own pace and under the conditions they encountered. At the same time, the students expressed the wish to keep

220 the experimental tasks within the schedule defined before lock-down, during four afternoons, April
27th to 30th. The sudden change to online teaching, improvised by teachers and students, disrupted
their habits and introduced new stress and anxiety about how to be able to succeed with their duties.
Following the previously defined teaching schedule helped both students and teachers to keep track
and to feel satisfied to have complied with their daily tasks.

225 The guidance was therefore based on both synchronous and asynchronous instruction. Each
afternoon session started with an online group meeting of about 30 min where the teacher introduced
the concepts relevant for the tasks and experiments of the day. After this, a 15 min online quiz gave a
fast feedback about the student's attention and understanding of these concepts. It also helped to
relax after the theoretical introduction. Then followed a 30 min meeting where more practical aspects
230 were discussed. After this, each student worked on his own, solving the defined tasks, continuously
supported by the teachers, who were available for chat and videoconferencing to offer help and
guidance.

Additionally, we prepared several detailed documents to allow for the asynchronous instruction:

(1) A *teaching guide* defining detailed daily schedules, the syllabus and the assessment process.

235 (2) Detailed *learning sheets* explaining the theoretical concepts, the experimental setups and the tasks
and assignments. They also included comprehension questions, short exercises and problems.

Questions and exercises were checked by the online quizzes, whereas the problems and some specific
tasks were part of the daily assignments. (3) Spreadsheets with numerical data of the laboratory
experiments. Students who did not obtain valid experimental results could at least analyze and

240 discuss this “fallback” data and respond to the problems and tasks.

TEACHING PROGRAM

Under normal circumstances, the four lab sessions would have been preceded by two 45 min
sessions to introduce the theoretical concepts and data analysis. Due to the exceptional conditions
and the huge burden it charged on the students, we selected one of the original two experiments,
245 moved the introductory sessions to the beginning of each afternoon, and simplified all assignments,
including the final lab report, which could be turned in by the end of the fourth day. This way we

delimited the necessary dedication to this subject and gave the students the possibility to concentrate immediately afterwards on other duties.

The adapted teaching program was as follows:

250 Day 1. Introduction to data analysis with spreadsheets

- Linear fit with parameter error analysis
- Nonlinear fit with solver

Day 2. Photometry

- 255
- Introduction to photometry
 - Building a photometer at home
 - Preparation of the concentration series, measurement of the calibration curve
 - Preparation of the samples for adsorption measurements

Day 3. Adsorption Isotherm

- 260
- Introduction to adsorption at liquid/solid interfaces
 - Measurement of the adsorption isotherm

Day 4. Adsorption Isotherm, wrap up

- Completion of all measurements
- Data analysis
- Drafting of the report with the results and discussion.

265 **ASSESSMENT**

The assessment was based mainly on the active and constructive participation during the laboratory sessions and on the completeness and quality of the assignments. In addition, the originality, improvisation and ability to solve the problems encountered during the experimentation at home were rewarded.

270 The subject could be passed with a grade of 5 in 10 only solving the exercises and problems with the data given in the spreadsheets. The home-lab experiments were optional, but they were necessary to raise the overall grade above the minimum of 5. All students opted for the experiments.

EXPERIENCE

275 Students were very open and keen to build the home-lab in their kitchens. After six weeks of lock-down and online-teaching they enjoyed the change in their routine. Figure 4 shows six examples of the setups the students built on day 2. One of them used her notebook screen as the light source, an excellent, although somewhat risky, idea which we now apply in an improved setup using a tablet.

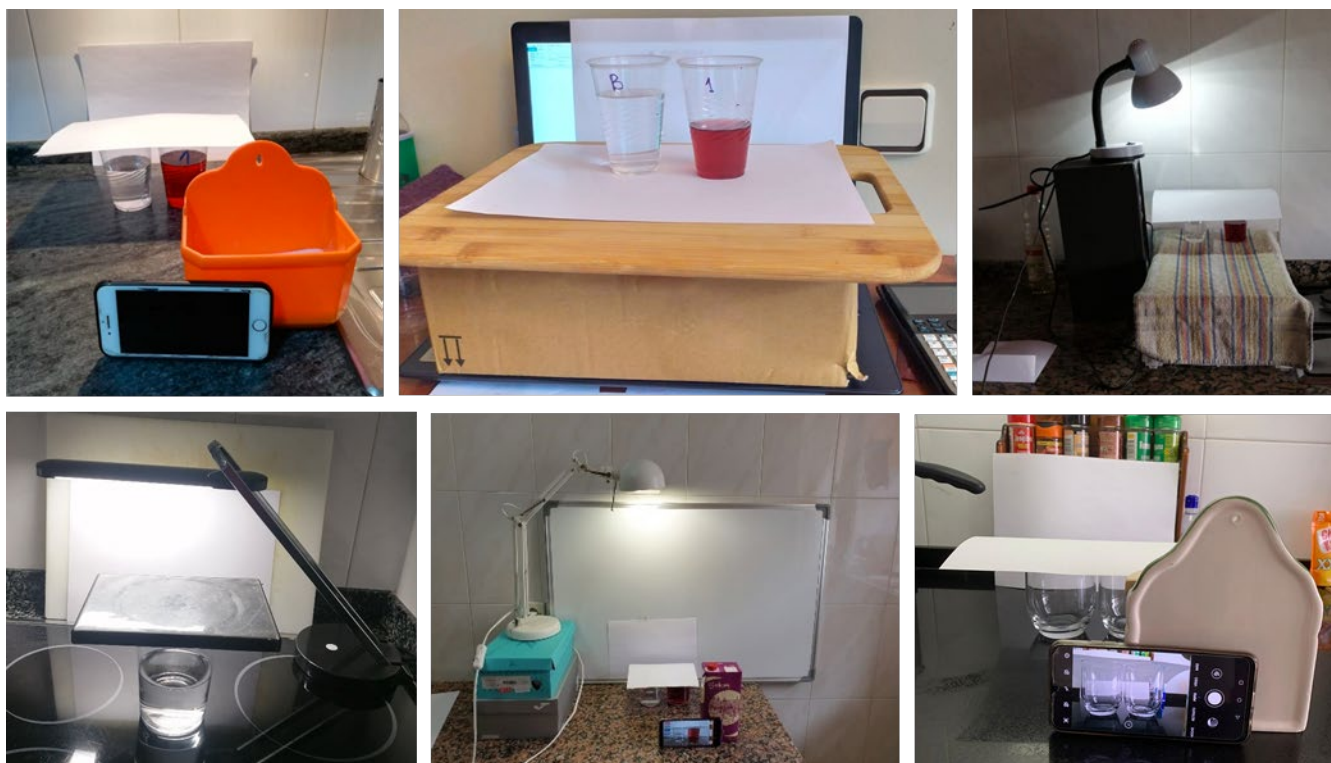


Figure 4: Examples of the setups of the photometer improvised by the students

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In Figure 5 the first photos taken with these setups can be seen. Setups and photos were of differing quality with some typical mistakes, which we discussed with the whole group at the start of day 3. Most students decided to improve their setups and repeated their experiments. Some gave up and used the fallback data given in the spreadsheets.

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Two trivial, but important, errors were recurrent: several students had started with a much too high dye concentration, which blocked nearly all light. And, surprisingly, many took their photos from such an elevated viewpoint that they had nearly no free sight through their solutions onto the illuminated background. Both mistakes, once explained, were very instructive for the students. They had just not understood the very basic concept of a light intensity measurement and the importance to get comparable readouts for both reference and sample.

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The students who opted to repeat the experiment were rewarded with much better results and good calibration curves like that of Figure 6.



Figure 5: Examples of smartphone photos taken by the students for the absorbance measurements

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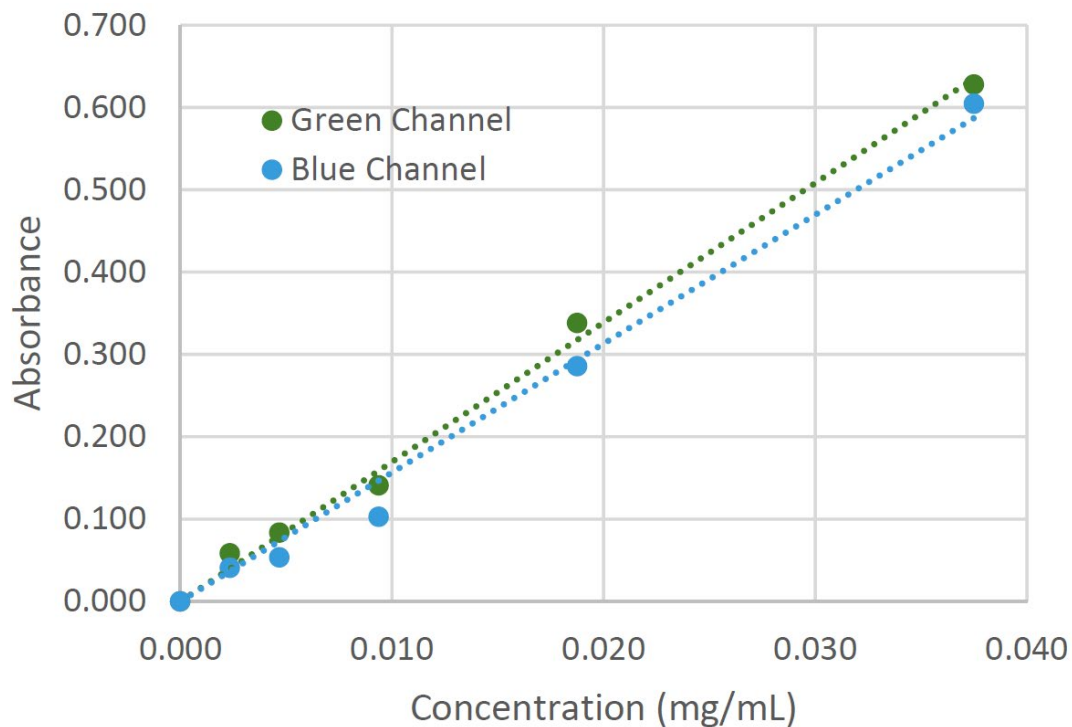


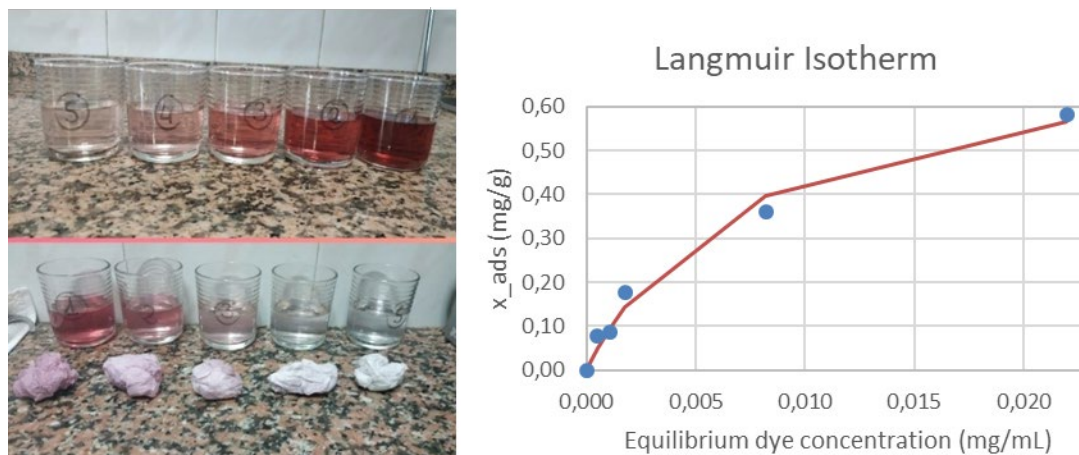
Figure 6: Example of a calibration curve, absorbance vs. anthocyanine concentration obtained by a student with his home-lab photometer.

The day after the addition of the adsorbent (two sheets of kitchen paper), the students measured again the absorbance of the samples, which allowed them to calculate the equilibrium dye concentrations and the adsorbed dye mass per adsorbent mass. They then applied both linear and nonlinear fits of Langmuir and Freundlich isotherms to their data and discussed whether the

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adsorption was of the single or of the multi-layer type. Figure 7 shows the results of a successful experiment.

305 Of course, not all students obtained data of sufficient quality to finish all steps. All 23 students built the setup and obtained experimental data. 17 students represented the calibration curve, 13 the adsorption isotherm, and 5 applied the full (linear and nonlinear) analysis to their data (See SI). So most students were successful in designing and mounting their photometers, in preparing their samples and in performing measurements. Under the conditions we lived at that moment, this was a
310 great success. Those students with insufficient data discussed the limitations and problems they encountered and then used sample data for the missing steps. All students fulfilled all assignments and submitted their lab-report. All learned their lesson, some by succeeding, others by failing.



315 Figure 7: Examples of concentration series before and after addition of the adsorbent. Adsorption isotherm obtained by a student: Adsorbed mass per adsorbent mass, x_{ads} , versus dye concentration in equilibrium conditions. The red line is the Langmuir isotherm fitted to the experimental data (blue points).

CONCLUSIONS

The students were highly motivated and tried hard to get best possible results. The quality of the assignments and of the final reports, and of course also the direct contact with the students,
320 confirmed that most of them achieved the learning objectives, at a comparable level as in years before with conventional lab equipment. The distribution of the grades was similar to that of previous years (see SI). The feedback from the students was very positive and many expressed how much they enjoyed designing their own instrument. This activity helped them very much to understand the fundamentals of photometry, much better than with the closed-box photometers used in the faculty. In
325 summary, this emergency home-lab solution was a great success and an excellent instructional

experience. It allowed our students to improve their competence in the laboratory, stimulated their improvisation skills and boosted their self-esteem. We all desire that this emergency will not repeat in any near future, but we will definitely introduce some aspects of this experience in our teaching practice, once the “new” normality is recovered.

330 ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at ...

Data analysis procedure, summary of experimental results, comparison of grades in the last four years (PDF)

335 Spreadsheet with example data obtained by students and full data analysis procedure (XLSX)

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