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Lab at home: 3D printed and low-cost experiments for thermal engineering and separation processes in COVID-19 time

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A B S T R A C T

The SARS-CoV-2 virus pandemic has meant that face-to-face teaching activities have had to be replaced by distance learning. Experimental laboratories have been replaced, in most cases, by the utilization of experimental data or by simulations. In this work, we have designed four laboratory experiments to be conducted by students of thermal engineering and separation processes during confinement by COVID-19 at home, to maintain competence acquisition and learning outcomes. A mixed methodology of the educational models of autonomous learning and cooperative learning has been used in obtaining the experimental data and writing the laboratory report. Installations for thermal engineering have been 3D designed and printed and are aimed at studying the heat transmission by conduction and convection in heat exchangers. This work describes in detail the activities carried out and shares the files used in the 3D printing of the installations. The laboratory experiments of separation processes are focused on the removal of a dye (rhodamine B) from an aqueous solution by liquid-liquid extraction and adsorption. A survey made to the undergraduate students has confirmed that the methodology and installations designed have been satisfactory for their expectations on the acquisition of knowledge and skills in both subjects.

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1. Introduction

The SARS-CoV-2 virus has caused the first global pandemic of the 21st century. The rapid spread of COVID-19 throughout the world has caused governments in most countries to take extreme measures such as home confinement and the closure of schools and universities to control the pandemic. According to UNESCO, in April 2020, schools and universities were closed in 185 countries, affecting more than 1.5 million students (UNESCO, 2020). It is estimated that approximately 60% of the universities were closed and another 30% experienced major disruptions (Marinoni et al., 2020).

In this context, university teachers have adapted the face-to-face lessons to distance teaching and learning, affecting almost 90% of the activities taught in universities around the world (Marinoni et al., 2020). In the case of theoretical lectures and exercise seminars, most universities opted for the use of video conference software or recorded audio-visual material. Several papers have recently been published on the digital adaptation of theoretical and practical content of engineering degrees during the confinement period caused by COVID-19, ensuring the learning outcomes but limiting the physical contact (Glassey and Magalhães, 2020). Da Silva Junior et al. proposed the use of a game to review organic reactions by answering over 600 multi-choice questions (da Silva Júnior et al., 2020). Ripoll et al. adapted the face-to-face activities of Biochemical Engineering to online collaborative learning activities (Ripoll et al., 2021). Finally, Debaq et al. transposed engineering labs on four operations used in food industry to remote labs using virtual tours and analysis of real experimental data (Debaq et al., 2021).

According to the International Association of Universities report on the impact of COVID-19 on higher education, the greatest difficulties were found in disciplines depending on access to laboratories (Marinoni et al., 2020). In the degree in Chemical Engineering of the Complutense University of Madrid, it was decided to replace most of the laboratories by the treatment and discussion of experimental data obtained by students from previous courses or by

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Table 1
Characteristics of thermal engineering and separation processes subjects in the degree of chemical engineering at the Complutense University of Madrid.

| Subject                  | Thermal Engineering | Separation Processes |
|--------------------------|---------------------|----------------------|
| Course                   | Third               | Third                |
| Total Credits            | 9 ECTS              | 12 ECTS              |
| Laboratory Credits       | 2 ECTS              | 2 ECTS               |

simulation practices. In other Spanish universities, similar activities were chosen during the confinement, replacing the laboratories by virtual tools (Nogales-Delgado et al., 2020). However, with simple data processing, the students do not acquire the experimental skills in the same way as in face-to-face laboratories.

The teaching methodology used in the universities during the 2020/21 academic year is marked by the situation of the pandemic in each of the countries. In the specific case of Spain, the Ministries of Health and Universities have provided a series of guidelines to be followed by universities during the COVID-19 pandemic. In this regulation, it is indicated that face-to-face activities cannot be carried out in Spanish universities as long as distance of 1.5 m between all students is not guaranteed (Ministry of Universities of Spain, 2020). Moreover, the attendance at face-to-face activities by infected people, persons with compatible symptoms, or students who have had close contact with infected persons is prohibited. Considering this regulation, it is highly probable that there are students in a situation of confinement in their homes on the dates that the experimental laboratories of the chemical engineering degree are carried out. For this reason, several low-cost laboratory experiments have been designed to be sent from universities to students’ homes. A similar initiative has been developed by professors at Imperial College of London for the Physics degree. They have employed the ‘Lab in a Box’ methodology to achieve the same learning outcomes in a home laboratory as those achieved in the university laboratory (Mackay, 2020).

The lab at home methodology used in this teaching innovation project was approved and financially supported by the Complutense University of Madrid. Also, the teaching guides of the Chemical Engineering degree were modified to allow for three different scenarios depending on the evolution of the pandemic during the 2020/21 academic year. In these scenarios, face-to-face, semi-face-to-face, and online activities were designed. The practices designed in this work can be used by students in a situation of confinement at home during the 2020/21 academic year, in future confinement situations, or for health problems that prevent them from going to the face-to-face laboratories.

2. Competencies and learning outcomes

In this work, two laboratory experiments have been designed to be done at the homes of the confined students of the thermal engineering subject and two practices for the separation process subject. In Table 1, the characteristics of both subjects are shown. As observed, in both subjects the experimental laboratories suppose a significant percentage of the total credits.

According to the teaching guide of both subjects, students must acquire experimental skills related to the measurement of parameters in equipment and installations of heat transfer and separation processes. The thermal engineering teaching guide details two specific competencies that must be acquired by the students of the subject: to measure the technical parameters in heat transmission equipment and installations and their technical interpretation and to recognize the principles on which the different mechanisms of heat transmission are based. The two practices designed for this subject have sought to ensure that students acquire both skills, using installations that allow them to measure and calculate technical parameters. In these experiments, heat transmission through the mechanisms of conduction and convection will be studied.

In the teaching guide of separation processes, it is indicated that students must acquire two specific competencies during the laboratories of that subject. The first competency is to measure technical parameters in equipment and installations of separation processes based on mass transfer. The second competency is to identify the principles on which the different separation processes are based. For students to acquire both competencies, two practices have been designed based on two separation operations: liquid-liquid extraction and adsorption. In both experiments, students will determine experimental data and interpret the technical results. They will also compare the results obtained with installations for the solution of the same problem: the treatment of wastewater contaminated by a dye.

In addition to the specific competencies indicated above, the teaching guides for both subjects describe four transversal competencies that must be acquired by the students in the laboratories: Consult, use and analyse bibliographic sources; working in groups; autonomous learning; and demonstrate initiative and creativity to solve new situations. The experiments designed in this work have sought to ensure that students acquire these four transversal competencies in the same way they did in previous courses in face-to-face laboratories.

In the case of thermal engineering practices, two experimental installations have been designed using a 3D CAD software and 3D printing. In the experiments, students will use hot and cold water at home to study the processes of convection and conduction that occur in heat exchange equipment. These experiments are intended to replace two facilities used in the face-to-face laboratories of thermal engineering subject at the Complutense University of Madrid, maintaining the same learning outcomes. These installations commonly used in on-site laboratories are a shell and tube heat exchanger where heat is exchanged between cold and hot water, and a non-stationary heat conduction practice in a polymer part immersed in a thermostatic bath.

On the other hand, for the subject of separation processes, we have chosen to study the two separation operations that can be carried out at room temperature: liquid-liquid extraction and adsorption. It has been selected to replace the distillation practice usually performed in the face-to-face laboratories to separate the cyclohexane and toluene mixture with a liquid-liquid extraction practice to minimize the risks of the experiments performed in the students’ houses. In the case of the adsorption practice, the separation of benzoic acid is usually performed and has been replaced by the adsorption of a dye (rhodamine B) to increase safety and avoid the use of analytical equipment. The design of these experiments has sought to maintain the acquisition of skills and learning outcomes that those obtained courses prior to the COVID-19 pandemic in the subject of separation processes.

The following two sections describe the objectives pursued with the design of the four experimental installations and the education models used in the design of learning activities by the lab at home methodology.

3. Objectives in the design of the experimental installations

The following objectives have been taken into consideration in the design of the four experimental installations presented in this paper:

a) To carry out an experimental research. In the design of the experiments, it has been sought that the students carry out an investigation by obtaining experimental data, in order to guarantee the acquisition of the competencies indicated in the teaching guides
of the subject, as described previously. Also, based on this experimental data obtained at home, students will discuss in groups the results and write a laboratory report with the usual sections of the research articles: introduction, experimental procedure, results and discussion, conclusions, and literature references. In this way, despite the students are in a lockdown situation or that the laboratory practices cannot be carried out in person at the university, the students of the degree in chemical engineering will acquire the same competencies and learning outcomes as those acquired in previous courses by the students in face-to-face laboratories.

b) **Low-cost installations.** The installations used in the experimental laboratories of the degree in chemical engineering often have high costs in materials and analytical methods and large sizes that prevent them from being sent to the home of students who are in a situation of lockdown. For this reason, we have sought to design small, lightweight, and low-cost facilities that do not require the use of analytical equipment to obtain experimental data.

In the case of thermal engineering experiments, we have chosen to design small installations using 3D printing, to use water as hot and cold fluids, and to determine the temperatures of the fluids with low-cost alcohol thermometers. Besides, all these materials can be sent and reused by other students, further minimizing the costs associated with conducting these laboratories.

In the experiments of separation processes, it has been chosen to carry out the elimination of a dye (rhodamine B) from an aqueous solution. The concentration of the contaminant before and after the experiment can be easily determined by the students using a visual scale of concentrations of the dye in water, avoiding the use of analytical methods. In the extraction experiment, a low-cost solvent of natural origin, limonene, has been selected, whereas the adsorption will be carried out in Eppendorf plastic tubes with small amounts of activated carbon. In this way, two different experiments of separation processes are performed using low-cost and low-weight materials that can be easily sent to students’ homes.

c) **Safety and waste management.** In the design of installations to be used at the homes of chemical engineering degree students, the use of highly safe experiments and materials with very low or no toxicity has been a fundamental requirement. In the practices of the thermal engineering course, we have chosen to use hot and cold water as heat exchange fluids, as this ensures the safety of students working with a substance of zero toxicity and not generate waste. Students will be instructed how to heat the water to be used as a hot fluid using a microwave or cooktop. In the results shown in this work, water heated to 80 °C has been used as a hot fluid in the two thermal engineering experiments, but hot water at a lower temperature could also be used, reducing risks, and obtaining similar results. It is important to emphasize that the initial temperature of the hot fluid should not exceed 65–80 °C, as prolonged heating of PLA may cause softening of the material due to its glass transition temperature.

In the case of separation process experiments, we have chosen to work with an aqueous solution to ensure the safety of the students. A very low concentration of rhodamine B (20 mg/L) has been used in the solutions fed to the extraction and adsorption experiments. Students will receive this aqueous solution at home to avoid contact with pure rhodamine B and the use of analytical balances.

For the liquid–liquid extraction experiments, a natural solvent has been selected, limonene, which forms two liquid phases with water. The limonene will be provided to students in the glass vials in which they will perform the liquid–liquid extraction processes after adding the aqueous solution of rhodamine B. This will prevent students from handling the limonene. In addition, students will be provided with latex gloves and will work at room temperature in the extraction experiments. At the beginning of the experiments, safety instructions were given to the students to minimize the risks during the activity and days before they were given the safety data sheets of the chemicals to be used.

The waste generated in the separation process experiments: aqueous solutions of rhodamine B, organic phases with limonene, and spent activated carbon, will be properly managed. For this purpose, students will be provided with small bottles where they can safely store these wastes, which will be returned to the university where they will be managed in the usual way for the waste generated in the laboratory experiments.

d) **Incorporation of Master and PhD students into teaching tasks.** In the design of the laboratory experiments presented in this work, one of the objectives has been to incorporate PhD students and master thesis students so that they can begin to receive training in teaching activities.

Two of the students who have participated in this work have as one of the objectives of their doctoral theses the use of 3D design and printing in the optimization of separation processes. Their experience in these activities has been fundamental for the adequate design of the facilities that are going to be used in the thermal engineering subject. On the other hand, three of the master and doctoral students use adsorption and liquid–liquid extraction in the treatment of wastewater, so they have contributed their experience in the development of small installations that will allow them to tackle the elimination of a dye from an aqueous solution using both separation processes. Master’s and PhD students participated in the conceptual design of the facilities, testing, and preparation of explanatory materials. Doctoral students, who have permission to carry out teaching activities, participated in the teaching activities described in section 4.

With the incorporation of Master’s and PhD students in this teaching innovation project, synergies have been generated by providing knowledge from young researchers to chemical engineering degree students. Also, the participation of undergraduate students in the subjects of thermal engineering and separation processes in the final design of the facilities has been encouraged. Vocations will be generated among students towards research by learning more about the activities carried out by the research group that has developed this project.

4. **Educational models and teaching methodology**

4.1. **Educational models**

Because the collection of experimental data must be done individually by the student who is in a situation of confinement at home, it has been chosen to combine characteristics of two educational models: autonomous learning and cooperative learning. Thus, the experimental work will be done individually at home, but results interpretation and the elaboration of the laboratory report will be done in groups of two or three students through cooperative learning. The following four characteristics of the autonomous learning model have been applied in the design of the activities (Betts, 2003):

1. **Learners are involved in guided open-ended learning experiences.**
2. **Teachers are facilitators of the learning process, as well as dispensers of knowledge.**
3. **Learners develop appropriate questioning skills.**
4. **Curriculum is based on the interests and passions of learners.**

These characteristics have been considered in the design of the activities described in section 4.2, with the students taking an active role and the professor a facilitator role. The fourth characteristic was used in the design of the survey to students described in section
5. This survey had the aim of checking that the designed activities are in agreement with the interests of the students. Cooperative learning in small groups will be the model used for the development of the laboratory reports. This educational model has been successfully used in chemical engineering degree activities, with students participating in the achievement of the competencies, the attainment of the objectives, and acquiring strategies to learn in a group (Arteaga et al., 2013; Maceiras et al., 2011). The following five characteristics of cooperative learning were followed in the development of group activities (Felder and Brent, 2007):

1. Positive interdependence. All members will work together in the development of the common objective: the writing of the laboratory report.
2. Individual accountability. All students in the group are responsible for the collection of experimental data that will be aggregated with the data obtained by the other group members.
3. Face-to-face promotonal interaction. Students must interact in the discussion of the results obtained and the conclusions.
4. Appropriate use of collaborative skills. Students will be required to work on leadership, decision making, and conflict management skills.
5. Group processing. Group members should set goals, assess what they are doing well, and propose changes to work more effectively.

Considering the characteristics of the autonomous learning and cooperative learning models just named, the teaching activities described below have been designed.

4.2. Description of teaching activities

The activities described have been developed between September and December 2020. Each teaching activity begins with a brief description of the experiment to be carried out on the students who are at home in a lockdown situation. This communication is done using video conferencing tools provided by the university: Google Meet or Collaborate. In this explanation, the professor explains the materials provided, the safety measures required of the students, and the methodology to be followed to obtain experimental data.

Students will then autonomously use the materials sent to obtain experimental data. In this period of approximately two hours, the professor will be at the students' disposal through the videoconferencing software. In addition, the professor may ask students individual or group questions to assess the acquisition of skills and learning outcomes, evaluating each student based on the answers. The collection of experimental data will be done individually since each student is confined to his own home. However, the students will be grouped for the writing of the laboratory report, so that they can obtain the transversal competence of working in groups. Also, the search for bibliographical results and the reasoned discussion of the results obtained will be encouraged, to guarantee the acquisition of transversal competencies. The previously described characteristics of the cooperative learning model will be applied throughout this period.

About the methodology used for the evaluation of the acquisition of competencies and learning outcomes, the attitude and capacity of autonomy shown by the students during the obtaining of the experimental data will count 15% on the final mark. The quality of the answers given by the students to the teacher during the realization of the experiment will be 25% of the mark. Finally, the laboratory report delivered in group 10 days after the realization of the practices will account for 60% of the overall mark.

5. Survey to students about the ‘Lab at home’ methodology in COVID-19 time

To check that the designed activities follow the fourth characteristic previously described of the autonomous learning model: the curriculum is based on the students’ interests and passions, a survey has been carried out to know the students’ opinion about the designed activities. The first question asked about the methodology of carrying out substitute activities for laboratory experiments in the case of suffering confinement. Then, a brief description of each of the experiments designed in this work was presented to the students using images of the installations designed in 3D for the thermal engineering subject and of the materials to be used in the separation process laboratories. The last four questions were focused on these installations to check that the students considered these experiments suitable to be done at home and to understand the contents explained in the theoretical classes of both subjects. The questions with the four possible answers are shown in Fig. 1, along with the results obtained from the answers of the 84 students participating in the survey.

As it can be observed in the answers to question 1, the great majority of students selected as possible substitute activities the “Lab at home” methodology (43%) and the use of simulators (36%). Therefore, the methodology seems to be in accordance with the interests of the students. Only 13% selected the option of carrying out the treatment of experimental data obtained by students in previous years. This last data treatment option was the methodology used in most of the subjects of the chemical engineering degree during the 2019/20 academic year in the Complutense University of Madrid during the lockdown period. Given the results obtained in the survey, it seems that students prefer other methodologies to simple data treatment.

In relation to the two installations designed and printed in 3D for the subject of thermal engineering, the great majority (more than 90%) have evaluated positively in the survey both installations for the study of heat conduction and heat exchangers. During the survey, students have provided valuable comments on the final design of the installations used in the thermal engineering course. For example, the way the heat exchanger is connected to the tap was modified, using a funnel and a rubber band that could be adapted to the different configurations of the students’ homes. Similar results have been obtained for the two experiments designed for the subject of separation processes, which will be focused on the liquid-liquid extraction and the adsorption of a dye from an aqueous solution. The extraction practice has been found to be adequate or very adequate by 91% of the students, while the adsorption practice has received the approval of 98%. Therefore, considering the results obtained in the survey, the “Lab at home” methodology has the majority approval of the enrolled students and was successfully used in the cases where there are students confined to their homes on the same dates in which the laboratories of the subjects thermal engineering and separation processes would be carried out.

6. Lab at home: 3D printed experimental installations for thermal engineering

To carry out experimental practices of the thermal engineering subject in students’ homes, two practices have been designed using 3D design and printing. The first installation will be formed by two concentric cavities that will contain water at different temperatures to study the process of heat transmission by conduction. The second installation represents a heat exchanger of concentric
tubes in which the cold fluid will enter and leave the exchanger continuously, whereas the hot fluid will remain in the inner cavity, as it happens in jacketed reactors. Due to the moderate incidence of COVID-19 in Madrid during the months of November and December 2020, when the thermal engineering practices took place, installations were printed for 20% of the students, since the number of students in confinement was low. However, this percentage could have increased rapidly, as two installations a day can be produced using a 3D printer.

6.1. Thermal conduction in a 3D-printed installation

The software Rhinoceros 7 has been used to carry out the 3D design of the installation. Fig. 2 shows the 3D design carried out in Rhinoceros, together with the installation plans, indicating the main measurements of the pieces.

Once the 3D design has been completed, it has been saved as a STL extension file and sent to be printed on an Ultimaker 3 Extended printer, which works with Fused Filament Fabrication (FFF) technology, using a filament of polylactic acid (PLA). In Fig. 3, the printed installation is shown together with the two alcohol thermometers required to measure temperatures as a function of time. The STL files with the 3D design are available as supplementary material.

The materials shown in Table 2 will be sent to the homes of thermal engineering students who are in a lockdown situation. The table shows the approximate costs of these materials and the total cost per installation provided to each student.

It is important to note that both the installation printed on PLA and the two alcohol thermometers could be reused by other students, thus reducing costs. For the calculation of the individual costs of the thermometers and the gloves, the prices available on Labbox’s website have been used (Labbox, 2020), while the price of the PLA used in the installation has been obtained from Farnell’s website (Farnell, 2020).

Students will perform two different experiments using this installation. In the first one, they will place room temperature water in the outer cavity and hot water in the inner cavity. In the second experiment, they will exchange the positions of the hot and cold fluids. To determine the mass of hot fluid and cold fluid introduced, the students will measure with a ruler the height of water in each of the cavities. Using the measurements of the installation plans shown in Fig. 2, they will geometrically determine the volume of water added to each cavity. Finally, using bibliographic values of water density as a function of temperature can calculate the mass of hot fluid and cold fluid added.

Once the two fluids are introduced, students will begin taking temperature data from hot and cold fluids every 30 s for 18 min using the alcohol thermometers. In Fig. 4, the experimental temperature profiles in both experiments are shown. The initial temperature of the hot fluid was approximately 80 °C, while the initial temperature of the cold fluid was around 20 °C. From the experimental values of temperature and the calculated masses of hot fluid and cold fluid from the geometry, students will calculate the enthalpies of hot and cold fluids at each time, using a correlation of the specific heat of liquid water as a function of temperature and setting the initial temperature of the cold fluid as the reference temperature:

\[ h_i = mC_{p,i}(T_i - T_{ref}) \]  

From the initial enthalpy values of both fluids, the students will calculate the enthalpy transferred from the hot fluid (Eq. 2) and the enthalpy losses to the outside of the installation (Eq. 3). Both parameters as a function of time are also shown in Fig. 4.

\[ h_{\text{transferred}} = (h_{i,\text{hot}} - h_{i,\text{hot}}) \]
Comparing the results in both experiments, the students will see how the percentage of losses is much higher if the hot fluid is placed in the outer cavity, even cooling both fluids after 15 min of the experiment. They will also see that a greater quantity of enthalpy is transferred from the hot fluid when the hot fluid is placed in the outer cavity due to the greater volume of this cavity.

The results obtained will also be applied to approximately determine the conductivity of the PLA used in the construction of the installation with 3D printing. For this purpose, the heat flow transferred from the cold fluid in each of the 30-second increments (Q)

\[ h_{loss} = (h_{\text{transferred}} - h_{i,cold} - h_{0,cold}) \]  

(3)
will be calculated, and the product of the heat flow by the thickness of the wall ($\Delta x$), divided by the logarithmic mean area of the wall that separates both fluids ($A_{lm}$) will be represented versus the difference in temperature existing in that time interval ($\Delta T$). According to the following expression, the conductivity will be calculated as the slope of the line of adjustment of the representation previously described:

$$\frac{Q \Delta x}{A_{lm}} = K \Delta T$$  \hspace{1cm} (4)

Fig. 5 in the Supplementary Material shows the approximate determination of the thermal conductivity of PLA in the experiment where the hot fluid occupied the inner cavity. As can be seen in the fit equation, the $K$ value obtained was 0.261 W/mK. Students should compare the value obtained with the literature value for polyactic acid, 0.193 W/mK (Lebedev et al., 2017), discussing the simplifications made and proposing improvements for the determination of conductivity with higher reliability.

6.2 3D printed concentric tube heat exchanger

In the second of the experiments designed for the thermal engineering subject, students will use a concentric tube heat exchanger 3D designed and printed. The design procedure is analogous to that described above for the conductive heat transmission study facility. First, the installation has been designed using the Rhinoceros 7 software. The hot water will be placed in the inner tube of the heat exchanger, and cold tap water will circulate through the outer tube, entering and leaving continuously the concentric tube heat exchanger. This installation represents a jacketed reactor in which the reaction medium is cooled through a jacket through which cold water circulates.

Fig. 5 shows the 3D design of the heat exchanger, including the lid with two holes that will allow the measurement of the cold fluid outlet temperature and the temperature of the hot fluid present in the inner tube as a function of time. In this installation, the two alcohol thermometers previously described in the installation for studying heat transfer by conduction will be also used.

Once the design was made on Rhinoceros 7, it was saved as STL files and sent to the Ultimaker 3 Extended printer, where the concentric tube heat exchanger was printed also using a filament of polylactic acid (PLA). Fig. 6 shows the installation printed in 3D. The STL files with the 3D design are available as supplementary material. As seen, a plastic funnel has been placed on a tap, whereas the funnel and the heat exchanger have been joined using a silicone tube. To measure the flow of cold fluid, the students will use a plastic graduated cylinder with which they will be able to measure the volumetric flow of cold fluid that is coming out of the tap and through the exterior tube of the exchanger.

Table 3 shows the unit costs of the material used per student in this experiment. The costs have been obtained from the Labbox’s website (Labbox, 2020), except for the cost of PLA that has been obtained from the Farnell’s website (Farnell, 2020), considering the consumption of PLA in the printing of the concentric tube heat exchanger. It is important to note that 90% of the cost of the materials has been spent on materials that could be reused by several students. It should also be emphasized that the alcohol thermometers indicated in Table 3 are the same thermometers used in the other thermal engineering practice described above. To the cost of the materials previously described, the cost of sending the materials from the university to the students’ homes should be added. In the facilities designed for thermal engineering, the total weight of both installations is less than 500 g. All materials have been protected with bubble wrap paper to avoid damage during transport and have been placed in a cardboard box. The cost of transport in the case of shipping to locations near Madrid was less than 5 euros, and the students used the same materials and shipment methodology to return the facilities to the university.

In this work, the results obtained in three experiments are shown, in which the flow of cold fluid that circulates through the exchanger was varied. First, 15 mL of water was heated with a conventional microwave, and the inner tube of the heat exchanger was filled with this hot water using a plastic syringe. Then, the tap was opened to start passing cold fluid through the external tube.
of the heat exchanger. The temperature values of both fluids were measured for 8 min every 20 s. Finally, the volumetric flow of cold fluid that was circulating through the installation was determined using a graduated cylinder and a timer. The inlet temperature of the cold fluid in the heat exchanger was determined by measuring the temperature of the tap water.

Fig. 7 shows the temperature of the hot fluid as a function of time for three different values of cold fluid flow through the heat exchanger. Using the experimental values of the experimental temperatures for the hot water and the initial mass of hot water added to the internal tube, the enthalpy transferred from the hot fluid \( h_{\text{transf}} \) was calculated as a function of time employing the following equation:

\[
h_{\text{transf}} = m_{\text{hot}} C_{P,\text{hot}} (T_{0,\text{hot}} - T_{i,\text{hot}})
\]

where \( m_{\text{hot}} \) is the mass of the hot fluid calculated from the volume added to the interior tube and density of the water at the initial temperature, \( C_{P,\text{hot}} \) is the specific heat of the hot water at each temperature value expressed in J/kg K calculated with a correlation as a function of temperature, \( T_{0,\text{hot}} \) is the initial temperature of the hot fluid, and \( T_{i,\text{hot}} \) is the temperature of the hot water at each time.

From the enthalpy values of the hot fluid, students can determine the heat flow \( Q \) in W that has been transmitted in each 20-second increment, using the following expression:

\[
Q (W) = \frac{\left( h_{i+1,\text{hot}} - h_{i,\text{hot}} \right)}{\Delta t}
\]

As can be seen in Fig. 7, the maximum heat flow was obtained in the initial moments for the highest volumetric flow of cold fluid, while the decrease in the \( Q \) value was slower using the lowest volumetric flow of cold water. Using the heat exchanger design equation (Eq. 7), students will determine the overall heat transfer coefficient \( \langle U \rangle \) for one of the experimental times at each flow rate, calculating from the experimental temperature data the value of logarithmic mean temperature difference \( \Delta T_{\text{lm}} \) and the heat exchange area referred to the hot fluid \( A \) from the installation geometry provided in the plans:

\[
Q (W) = UA \Delta T_{\text{lm}}
\]

As an example, the overall coefficient of heat transmission at 60 s has been calculated for the three experiments, obtaining the highest value \( \langle U \rangle = 197.2 \text{ W/m}^2\text{ K} \) for the highest cold fluid flow: \( Q_{\text{cold}} = 5.26 \text{ mL/s} \) and the lowest value of the overall coefficient \( \langle U \rangle = 98.2 \text{ W/m}^2\text{ K} \) for \( Q_{\text{cold}} = 0.47 \text{ mL/s} \). The values obtained from the overall heat coefficient could be compared by the students with those estimated from predictive correlations of the individual coef-
Fig. 5. 3D view and plans of two pieces conforming the concentric tube heat exchanger designed using Rhinoceros 7. The measures indicated are expressed in mm.
coefficients and using the PLA conductivity value determined in the other practice designed in this work.

7. Lab at home: experimental installations for separation processes

7.1. Liquid-liquid extraction of rhodamine B dye from aqueous solutions using limonene

In the first experiment designed for the subject of separation processes, students will perform at home the liquid-liquid extraction of the dye rhodamine B from an aqueous solution with 20 mg/L of dye, using limonene as the solvent. The materials sent to the students are described in Table 4, together with the estimated cost of each material obtained from Labbox and Sigma Aldrich’s websites (Labbox, 2020; Sigma Aldrich, 2020). As can be seen, the unit cost of the material used in the experiment is less than five euros, to which it should be added the cost of sending this material from the university to the confined student’s home. The shipment of the materials of the experiments designed for separation processes was made by wrapping all the materials in bubble wrap paper and introducing them in a plastic container to retain a possible leak of liquid if any damage occurs. The plastic container was then placed in a cardboard box and sent to the students’ homes. Once the experiments were finished, the students introduced the liquid waste generated in a polypropylene bottle and sent it together with the materials used to the University, where the waste management was carried out. The cost of sending these installations was similar to that previously described for the thermal engineering experiments, being less than 5 euros, due to the low weight and volume of the materials used.

Students will receive 7 glass vials with different volumes of limonene inside (4.0, 3.0, 2.0, 1.0, 0.5, 0.2, and 0.1 mL) to avoid having to handle this solvent. The liquid-liquid extraction will be done in these glass vials, adding, with a plastic Pasteur pipette, the necessary amounts of the aqueous solution of rhodamine B (20 mg/L) to work at different volume of solvent-to-feed ratio in volume between 0.05 and 2.0. Using latex gloves, students will manually shake the vials for five minutes to reach the liquid-liquid equilibrium at room temperature. About ten minutes after shaking, perfect separation of the extract and raffinate phases will be achieved, obtaining two clear liquid phases, as can be observed in Fig. 8. Subsequently, the quantity of rhodamine B present in the raffinate phase after the extraction will be quantified.

To quantify the final concentration of rhodamine B in the raffinate phase after the extraction, students should prepare a colorimetric scale using 10 Eppendorf tubes. From the 20 mg/L solution of rhodamine B, they will make successive dilutions to reach aqueous solutions of the dye between 0.1 mg/L and 20 mg/L. Once this scale is prepared in the ten Eppendorf tubes, they will compare the colors of the raffinate phases obtained in the seven extraction vials with the different values of the colorimetric scale, thus approximately determining the final concentration of the orange in the raffinate. From these final concentration values of rhodamine B in the raffinate, students will determine the extraction yield achieved as a function of the solvent-to-feed ratio using the following equation:

\[ \text{Yld}(\%) = \frac{c_{\text{Rhod,r}} - c_{\text{Rhod,f}}}{c_{\text{Rhod,f}}} \times 100 \]  

(8)

where \( c_{\text{Rhod,r}} \) is the concentration of rhodamine B in the feed (20 mg/L) and \( c_{\text{Rhod,f}} \) is the rhodamine B concentration in the raffinate determined with the colorimetric scale. The obtained results of extraction yield as a function of solvent-to-feed ratio are depicted in Fig. 9.

In addition to calculating extraction yields, students will determine the composition of both phases using mass balances in each of the vials. First, they will calculate an overall mass balance, considering the added masses of solvent (S) and feed (F), which will be calculated from the volumes of solvent and feed added to each vial.

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**Table 3**

List of materials and estimated cost for the concentric tube heat exchanger printed in 3D.

| Material                                      | Quantity | Cost (€) |
|-----------------------------------------------|----------|----------|
| PLA in 3D printed installation *              | 85 g     | 1.65     |
| Alcohol thermometer (-10 – 110 °C) *          | 2        | 9.00     |
| Plastic funnel 50 mm diameter *              | 1 funnel | 0.39     |
| Silicone tube *                               | 0.5 m    | 0.63     |
| Plastic graduated cylinder 25 mL *           | 1 cylinder | 0.95   |
| Plastic syringe (20 mL) *                    | 2 syringes | 0.40   |
| Latex gloves *                               | 8 gloves | 1.06     |
| **Total cost for each student**              |          | **14.08**|

* This material can be reutilized.
and the literature density for the solvent and water at the experimental temperature. The sum of the solvent and feed masses should be equal to the final mass of extract (E) and raffinate (R) phases:

\[ F + S = E + R \]  \hspace{1cm} (9)

Then, the students will perform a solvent balance and a water balance in both raffinate and extract phases, considering the amounts added to the vial of water and limonene:

\[ S = E w_{\text{lim}}^{\text{ext}} + R w_{\text{lim}}^{\text{raff}} \]  \hspace{1cm} (10)

\[ F w_{\text{Water}}^{\text{feed}} = E w_{\text{Water}}^{\text{ext}} + R w_{\text{Water}}^{\text{raff}} \]  \hspace{1cm} (11)

where \( w_{\text{lim}}^{\text{raff}} \) is the mass fraction of limonene or water in the raffinate phase, \( w_{\text{lim}}^{\text{ext}} \) is the mass fraction in the extract phase and \( w_{\text{Water}}^{\text{feed}} \) is the water mass fraction in the initial rhodamine B aqueous solution added to the vials.

To solve the three mass balances above, they will have to consider the literature mutual solubilities of water and limonene at 298.15 K (Tamura and Li, 2005), thus determining the composition of the two liquid phases in mass fractions. From these values, students will calculate the experimental values of the rhodamine B distribution ratio \( D_{\text{Rhod}} \) and the rhodamine B/water selectivity \( \alpha_{\text{Rhod,water}} \) achieved by limonene at each S/F ratio value, using the following equations:

\[ D_{\text{Rhod}} = \frac{w_{\text{Rhod}}^{\text{ext}}}{w_{\text{Rhod}}^{\text{raff}}} \]  \hspace{1cm} (12)

\[ \alpha_{\text{Rhod,water}} = \frac{D_{\text{Rhod}}}{D_{\text{Water}}} \]  \hspace{1cm} (13)

Experimental results obtained for distribution coefficients and selectivities are shown graphically in Fig. 9. The experimental values shown in this figure could be compared by the students with literature values obtained by other authors in the liquid-liquid extraction of rhodamine B, to determine the performance of limonene in this separation process. In the literature, students will find articles in which rhodamine B has been extracted from aqueous solutions using conventional organic solvents (toluene, benzene, xylene, or hexane) (Elumalai and Muthuraman, 2013), ionic liquids (Fan et al., 2016; Kumar et al., 2020), or soybean oil (Jiao et al., 2016). The students will compare the results obtained using limonene and the other solvents used in literature, considering technical aspects (extraction yields, distribution ratios, selectivities, viscosities, and costs) but also sustainability aspects, comparing the methods of obtaining and synthesizing the different solvents.
The first part of the experiment is focused on studying the adsorption kinetics of rhodamine B. To do this, students will add 1 mL of rhodamine B 20 mg/L solution to four Eppendorf tubes along with a constant mass of activated carbon. Students will receive the Eppendorf tubes with the exact amount of activated carbon to be used in each adsorption experiment, as no precision balance is available at home. The tubes will be stirred for 30, 60, 120, and 300 s, obtaining the results shown in Fig. 10 using 30 mg of activated carbon. As can be seen, the adsorption kinetics of rhodamine B in the activated carbon used in this practice requires at least a stirring time of 300 s for rhodamine adsorption. This time will be used to perform the adsorption tests in batch.

In the second part of the practice, students will determine the isotherm of rhodamine B adsorption in activated carbon at room temperature. For this purpose, they will receive six Eppendorf tubes with activated carbon amounts between 3 and 50 mg. In each of the tubes, they will add 1 mL of the rhodamine B solution at 20 mg/L using a plastic Pasteur pipette, and manually shake each tube for five minutes. After the shaking, the students will obtain the results shown in Fig. 11.

Similar to the procedure described for the extraction experiment, students will prepare a colorimetric scale with rhodamine B concentrations from 0.1–20 mg/L in ten Eppendorf tubes, using plastic Pasteur pipettes and starting from the provided rhodamine B solution of 20 mg/L. Using the colorimetric scale, the students will estimate the concentration of rhodamine B in the aqueous solution after adsorption ($C_r$). These $C_r$ values will be used to determine the rhodamine B adsorption isotherm at room temperature using the
following equation to determine the mass of rhodamine B adsorbed in mg per gram of adsorbent \( (q_e) \), replacing the values of the initial concentration of rhodamine in solution in mg/L \( (C_0) \), the mass of adsorbent in g \( (w) \) and the volume of solution added in L \( (V) \).

\[
q_e = \frac{(C_0 - C_e)}{w} V
\]  

(14)

From the results of the isothermal adsorption, the students will use different isotherm models to fit the results obtained. Fig. S2 in the Supplementary Material shows the results obtained in the fit using the Henry model (Eq. 15), the Langmuir model (Eq. 16), and the Freundlich model (Eq. 17).

\[
q_e = K_h C_e
\]  

(15)

\[
q_e = \frac{q_{as} b C_e}{1 + b C_e}
\]  

(16)

\[
q_e = k_f C_e^{1/n_f}
\]  

(17)

Other models such as the BET, Redlich-Peterson, or Dual-site Langmuir could also be used to determine the different parameters of the models and discuss their validity. In the adjustment to the simplest model, the one proposed by Henry, a correlation coefficient \( R^2 \) of 0.968 was obtained. Using the Langmuir model, a correlation coefficient \( R^2 \) of 0.974 was determined, whereas with the Freundlich model, the correlation coefficient \( R^2 \) in the fitting was 0.984. Therefore, acceptable adjustments were obtained despite the simplicity of the designed experimental procedure. The results obtained by the students in the adsorption of rhodamine B using activated carbon could be compared with the results available in the literature for different types of adsorbents: activated carbon from almond shell \((\text{Abdolrahimi and Tadjarodi, 2019})\), vegetal bark carbon \((\text{Ramuthai et al., 2009})\), waste seeds \((\text{Postai et al., 2016})\), or acid treated banana peel \((\text{Oyekanmi et al., 2019})\).

In addition to performing discontinuous adsorption experiments, the final part of the experiment will focus on the adsorption of rhodamine B using a fixed-bed of activated carbon. The fixed-bed will be provided to the students already mounted inside an Eppendorf tube, which will contain 150 mg of activated carbon between two small amounts of glass wool. To sample the effluent after passing the fixed-bed, a small hole was done at the bottom of the Eppendorf tube. An image of the fixed-bed is shown in Fig. 12. The experiment will be performed at a flow rate of 1 mL/min using a plastic syringe to dose 0.5 mL of the solution each 30 s to the fixed-bed. At the bottom, samples will be collected using Eppendorf tubes every 60 s, obtaining the samples also shown in Fig. 12. By applying the previously prepared colorimetric scale to the samples obtained in the continuous adsorption experiment, the concentration obtained after crossing the fixed-bed as a function of time will be approximately determined, obtaining the breakthrough curve shown in Fig. S3 of the Supplementary Material.

From the results obtained for the breakthrough curve, the amount of rhodamine B adsorbed by the fixed-bed in mg/g \( (n_e) \) can be calculated using the following expression, considering the flow used \( (Q) \) in L/s, the amount of adsorbent in the bed \( (w) \) in g, the initial concentration of the solution \( (C_0) \) in mg/L, the concentration at the outlet in each time \( (C) \) in mg/L, and the time in which the outlet concentration coincides with the initial concentration \( (t_e) \) in s:

\[
n_e = \frac{Q}{w} \left( C_0 t_e - \int_0^{t_e} C \, dt \right)
\]  

(18)

Applying Eq. 18 to the breakthrough curve shown in Fig. S3, a value of adsorption capacity of rhodamine B in the activated carbon \( (n_e) \) of 0.901 mg/g was obtained. The students should compare the adsorption capacity shown by the fixed-bed in the continuous experiment with the results obtained in batch, proposing improve-
ments in the operating conditions of the fixed-bed to increase the adsorption capacity obtained. Besides, the two separation process practices could be completed by performing a global study of the results obtained by liquid-liquid extraction and adsorption, selecting one of the two technologies for the total elimination of rhodamine B from an aqueous effluent, considering the operating costs and environmental impacts of both separation processes.

8. Conclusions and future perspectives. Extension to other subjects and degrees

This paper has described four experimental laboratory practices designed to be performed in the homes of pandemic-confined students. For the subject of thermal engineering, two installations have been designed and printed in 3D to study heat transmission processes. This methodology of designing small 3D installations could be used in other subjects of the chemical engineering degree and extended to other degrees such as the chemistry degree or other engineering degrees. With these 3D designed installations, we have sought to design an experiment that can be used in all homes of confined students, using tap water as a thermal fluid and small installations that can easily be mailed and reused by several students.

These activities have sought to maintain the same acquired skills and learning outcomes as in previous courses, despite using simpler installations than those available in the university’s laboratories. For this purpose, a combined model of autonomous learning and collaborative learning has been used, obtaining the experimental data individually and writing the laboratory report in small groups.

In the case of the two experiments of the separation process subject, two operations have been selected (liquid-liquid extraction and adsorption) that can be carried out at room temperature, using low toxicity and low-cost materials and without the need to use analytical equipment. This methodology could be used in other subjects of the degree in chemical engineering, such as chemical reaction engineering or fluid mechanics, using colorimetric or pH scales and aqueous solutions to perform experiments at home of chemical reactions, non-ideal flow in reactors, or determination of the circulation regime.

PhD and master’s students have actively participated in the design of the installations, contributing the knowledge they are acquiring in their doctoral and master theses. Also, by surveying the students of the subjects, several of them have participated in the final design of the experiments with valuable suggestions. In this way, undergraduate and doctoral students in chemical engineering have been put in contact, allowing graduate students to participate in teaching tasks and undergraduate students to discover the research work being done in the group on the 3D design and printing and the application of adsorption and extraction in wastewater treatment. This collaborative methodology between professors and undergraduate and doctoral students can be used in the future in the development of new teaching materials and can be extended to any teaching activity of the degree in chemical engineering and other similar degrees. In this way, teaching vocations can be generated and strengthened among doctoral students and research vocations among undergraduate students.

Declaration of Competing Interest

The authors report no declarations of interest.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.ece.2021.02.001.

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