

A batch photoreactor for small-scale laboratory research - a technical note

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ABSTRACT

Photocatalysis for organic synthesis has experienced rapid progress over the last decades, which serves as an alternative to conventional synthetic routines. However, the photochemical reactor setup of many published works remains ambiguous due to missing standardized protocols. Therefore, a general design of batch photoreactor is required to circumvent the problem of difficult reproducibility and experimental inconsistencies. In this report which we have termed a technical note, a novel batch photoreactor with temperature feedback control and modulated light intensity was assembled from several low-cost, commercially available components, which will assist other researchers to reproduce this standardized reactor for use in their own research.

Keywords: photocatalysis, batch photoreactor, all-in-one reactor, temperature control, flexible design

1. Introduction

The utilization of photons as green, sustainable and cost-free reagents to drive chemical transformations with the visible-light-excited photoredox catalysts has experienced a great deal of progress over the last decades¹⁻⁵. Consequently, a dizzying array of photochemical test benches have been introduced by different research groups, yet no clear standardization is disclosed. Despite the rapid development of photocatalysis, this achievement faces some problems of reproducibility and inconsistency of results because of the use of the traceless reagent "photon" as a reaction reagent. The discrepancies are often a result of missing detailed descriptions with respect to the photoreactor setup. Important information such as light intensity is not clearly stated and generally absent in the literature, but vaguely provides macroscopic indicators of irradiated powers and distances from the light source^{6,7}. To address the above-mentioned issues, a standardized batch photoreactor with the following features is imperatively needed as partially recommended by Schiel *et al.*⁸ and Bonfield *et al.*⁹

1. Fixed vial and Lamp (or LED) position: a given distance between the light sources and the reaction containers (vials, test tubes, etc.) provides a constant light intensity.
2. Dimmable light intensity: since the reaction is initiated and dominated by the photon absorption process, a dimmable light intensity benefits conducting a kinetic study and enables the reacting system to perform without changing the spectral emission distribution of the light, thus increasing the reproducibility.
3. Replaceable and wide choice of high energy density light sources: the light source should be replaceable with a wide range of options for discovering the optimal excitation wavelength and possess a high energy density to initiate and propagate the reaction.
4. Effective in heat removing and broad operating temperature: most of photoreactions are carried out under ambient temperature and atmospheric environment. The reaction should be motivated only by the photon excitation and free of the thermal path,

therefore the effect of temperature should be minimized. As a result, air or liquid cooling is necessary.

5. Flexibility in the use of different types of reaction containers and ease to work with the flow conditions: the reaction containers in use can be arbitrarily assigned and the test rig can work in flow without too much additional effort.
6. Running reactions in parallel: the photoreactor should be able to perform several reactions simultaneously and rapid reaction condition screening is applicable.
7. Electronic display and controllable input data: the operation conditions can be specified by researchers.
8. Built-in magnetic stirring system: the photoreactor needs to have its own stirring function to assemble the all-in-one reactor.
9. Portable design with good safety: the ideal photoreactor should be all-in-one and well assembled in a closed box to avoid exposing light radiation to the environment.
10. Affordable price, and made of commercially available components: the photoreactor should be readily available and inexpensive.

In fact, some companies already offer several standardized commercial photoreactors with partially pre-described features¹⁰⁻¹⁷, but the prices are relatively high. The more functions a photoreactor can achieve, the more expensive it will be. In addition, several studies have revealed the design of the photoreactors and their principle,^{9, 18} even using the thriving 3D printing technology.^{8, 19} However, none of the aforementioned photoreactors achieve the full functions discussed, especially external stirring equipment is often required.^{8, 12, 13} Salley *et al.* has introduced a homemade stirring system in their nanomaterial discovery robot using stirring fan array and Neodymium magnets.²⁰ Inspired by this design, we realized that a built-in stirring system in a photoreactor is practical. Therefore, we have decided to build an all-in-one, open-access, standardized photoreactor that covers all the criteria mentioned before, which is rarely unveiled in recent publications. The technical note here describes the assembly of the

photoreactor in detail and researchers who want to build a similar one on their own may get some clues from this note.

2. Methods

2.1 Commercial components

The covers of photoreactor were made of stainless steel, which is a very common material in additive manufacturing. To make the photoreactor easily reproducible, all the commercially available material were in use as received. Table 1 summaries the main materials and resources for the batch photoreactor.

Table 1 Materials and equipment used and key dimensions

Materials	Source	dimensions	Reference
Axial cooling fan	Mouser Electronics	120 mm × 120 mm	21
Axial stirring fan	RS components	40 mm × 40 mm	22
Bosch Profile	RS components	20 mm × 20 mm	23
PFA tubing	Häberle	i.d. 6 mm & 14 mm	24
Aluminum rod	Conrad Electronics	30 mm × 30mm	25
Heat exchanger	Amazon	Tube o.d. 20 mm	26
High power LEDs	Mouser Electronics	/	27-29
Neodymium magnets	Supermagnete	20×4×3 mm	30
Arduino Board	Amazon	/	31
Tubing adapters	Swagelok [®]	/	32
Temperature/humidity sensor	Amazon	/	33
AC/DC converter	Amazon	/	34
DC/DC Buck converter	Amazon	/	35
OLED displays	Amazon	/	36
Adjustable Module			
Buck Converter	Amazon	/	37
Resistor status indicators	/	/	/

Note: i.d. inside diameter; o.d. outside diameter

2.2 Procedure

Section 1 Prepare the stainless steel plate cover

The built photoreactor should be assembled in a closed box to avoid light exposure to the environment and meet the requirements of safe operation. Stainless steel plate is a suitable, readily available and economical material for constructing photoreactors. In order to properly cover all components in the photoreactor, the following metal plate should be prepared:

1. Cut two pieces of stainless steel plate with a dimension of 325 mm × 325 mm × 2 mm as the top and bottom covers of the photoreactor
2. Cut four pieces of stainless steel plate with a dimension of 290 mm × 200 mm × 2 mm as the side covers of the photoreactor
3. Drill the holes of two side metal plates for mounting the axial cooling fans according to the parameters detailed in the supplementary .stl file.
4. Drill the holes of another side metal plate for installing the Arduino Board controller according to the parameters detailed in the supplementary .stl file.

Fig 1 shows the final design of the cover plates. The square holes ($\square=30$ mm) in Fig. 1a and 1b represent the position of the aluminum rod where the high-power LEDs will be installed. The large circular holes ($\Phi=22$ mm) in Fig. 1a represent the position of the test tubes while the small circular holes ($\Phi=2$ mm) in Fig. 1b indicate the position of the screws for fixing the magnetic stirring system. Fig. 1C shows the square holes ($\square=80$ mm) reserved for installing axial fan cooler. The bigger square area ($\square_1=30$ mm × 20 mm) in Fig. 1d will install the Arduino digital display screen, and the small squares ($\square_2=25$ mm × 8 mm) stand for indicators and controllers. The square holes ($\Phi=2$ mm) reserved for securing the indicators and controllers. One should note that all these sheet metal parameters should correspond to the dimensions of the electronic components used.

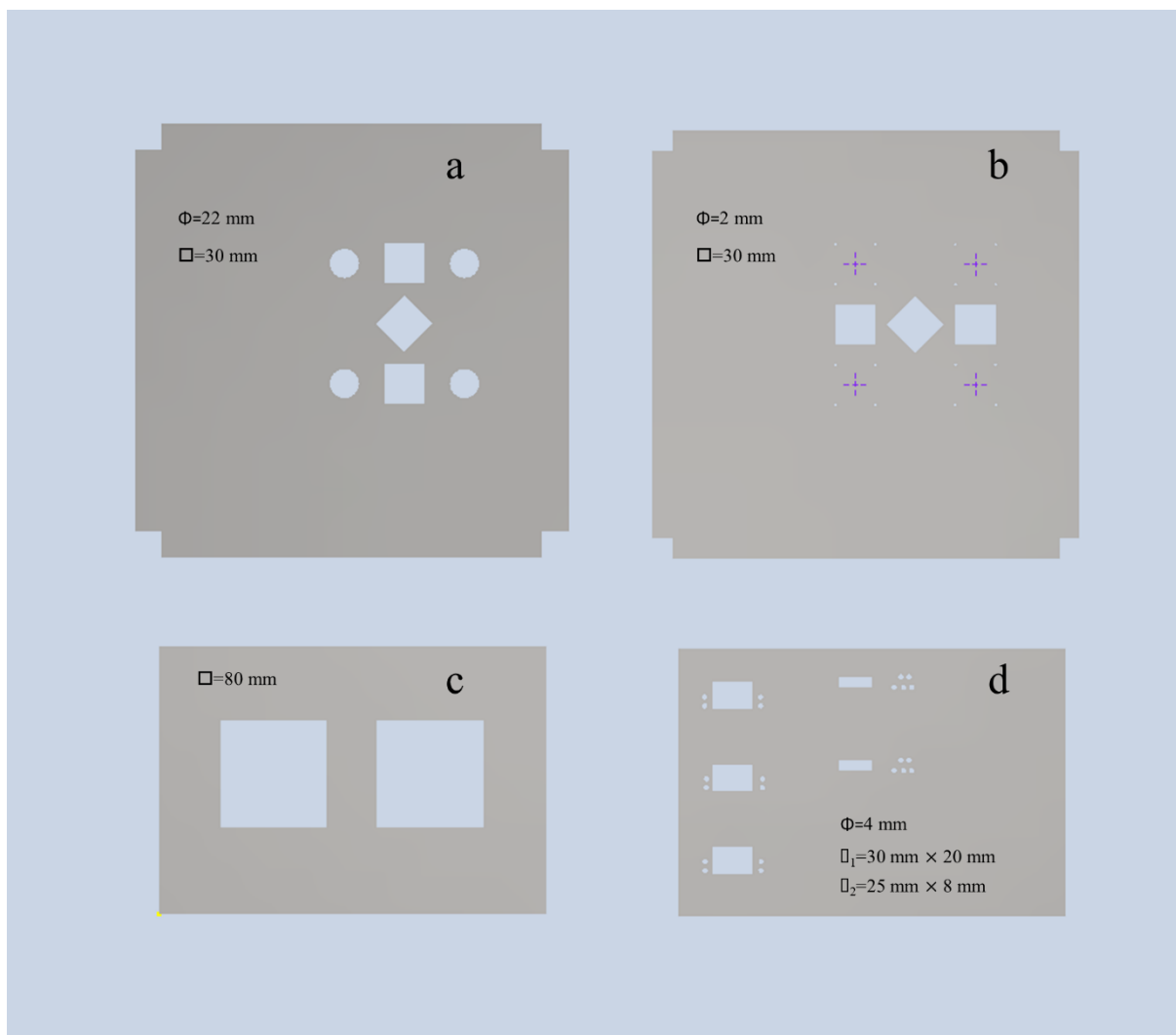


Figure 1 Final design of metal plates for batch photoreactor assembly: a) Top plate, fixing the test tubes and high power LEDs b) Bottom plate, fixing the magnetic stirring system and LEDs c) Left plate, axial fan cooler installed d) Right plate, Arduino Control panel installed

Section 2 Use 3D printing to prepare customized scaffolds

Once the metal plates were ready, the next step is to print some useful components for installing built-in magnetic stirring system and fixing the test tubes. 3D printing is a useful tool for generating bespoke designs. Therefore, it is possible to print simple scaffolds of arbitrary shapes in this photoreactor.

1. Use Autodesk Inventor® 2021 to draw the design (Fig. 2a-d) of the aluminum-cooler holder, test tube guide, test tube holder, stirrer base, stirrer top cover, stirrer mount, etc.
2. Convert the design file into .stl format for 3D printing

3. Use polymer printer “FlashForge Creator Pro” to print the customized support according to the parameters detailed in the supplementary .stl file.
4. All of the aforementioned parts were printed with PLA filament.

Fig. 2 shows the final design of these components. Fig. 2a represents the aluminum-cooler holder, which enables the circulating cooling water to go through the rod. Therefore, it will act as a heat sink to remove the heat generated by the LED irradiation. Fig. 2b represents the tube guide and holder that fit all the test tubes with NS 14/23 standard. This tube holder design is also replaceable depending on the tube used. Fig. 2c represents the customized adapters for building the magnetic stirring system, which should fit with the axial stirring fans incorporated into the system. Fig. 2d is the base for the magnetic stirring system, whose height is adjustable according to the position of the LED used. Similarly, it is important to note that all these parameters should correspond to the design of the photoreactor that researchers want to build by themselves.

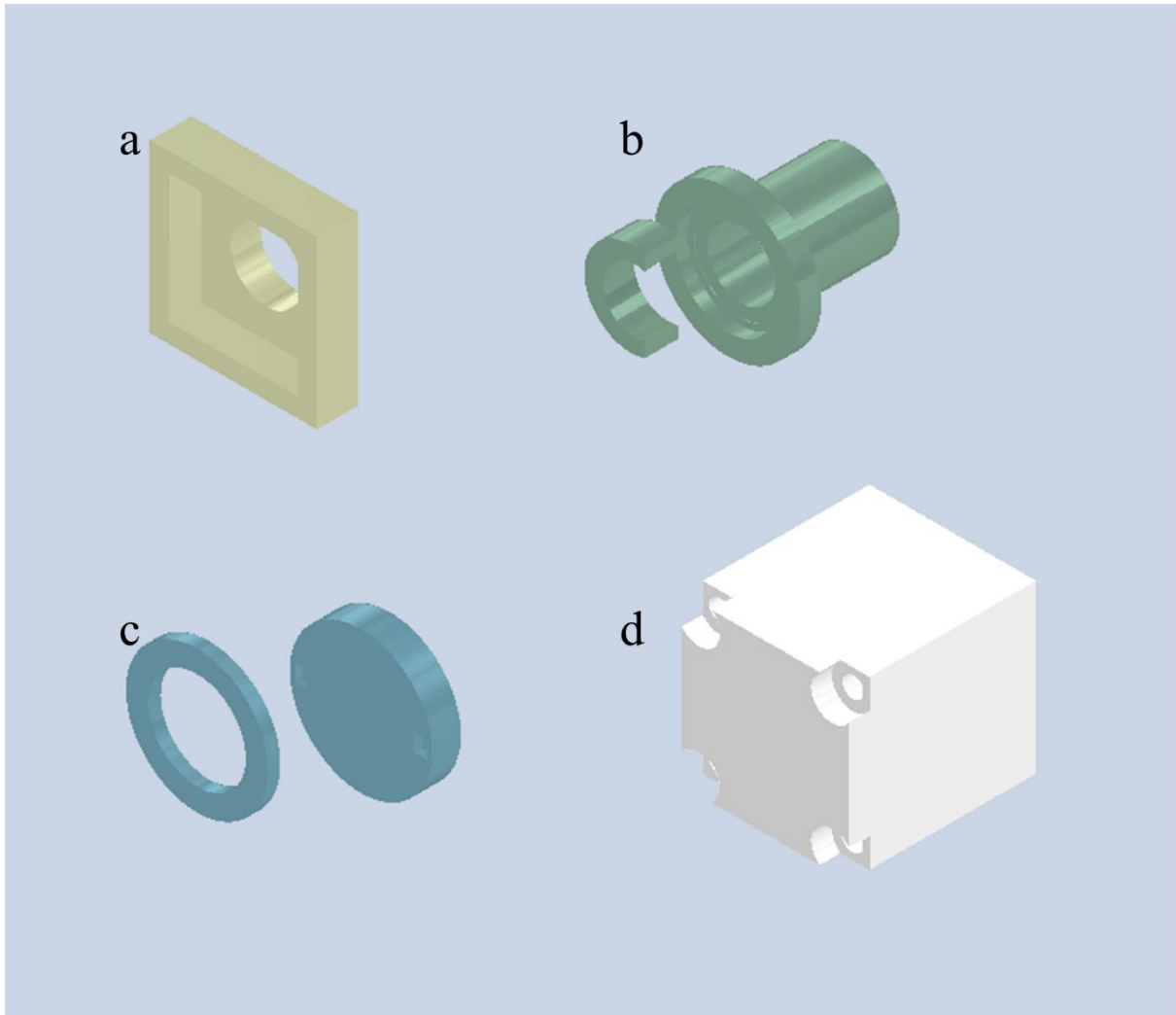


Figure 2 3D printed parts for repositioning and fixing. a) Aluminum-cooler holder b) test tube guide and holder c) stirrer base and top d) stirrer mount

Section 3 Buildup built-in magnetic stirring system

Direct individual stirring was achieved by integrating two 20×4×3 mm Nd block magnets in a custom-designed and 3D printed housing that mounts on the top of a 12 V DC axial stirring fan. Different from placing the magnets horizontally in the literature²⁰, they are installed vertically and inserted into the groove of the stirrer base and top support. A step-by-step assembly is as follows:

1. Carefully remove the fan blades with tweezers and smooth the perimeter with a flat file.
2. Install the adapter shown in Fig. 2c with the axial stirring fan. To achieve a good compact between these two parts, use a glue if necessary

3. Insert Nd magnetics into the adapter and cover it with the top support, use a glue if necessary
4. Secure the stirrer base and stirrer mount with nails and nuts

Section 3 Glue LEDs on the aluminum rod (heat sink)

High power LED will generate lots of heat from illumination, which in turn may make itself burnout. Therefore, a heat sink is required to dissipate the heat effect and guarantee the LED work properly. An aluminum rod is a good choice to act as an effective cooler since a constant surface temperature can be maintained by flowing the cooling water through the center of its shaft. A step-by-step assembly is as follows:

1. Use three types of LEDs in the photoreactor with the peak emission at 365 nm, 460 nm (130 lm per LED), and 523 nm (600 lm per LED)
2. Polish the aluminum rod with sandpaper and clean it with tissue
3. Fix the LEDs to a desired position on the rod and glue it with thermal paste
4. Wait for the curing process to complete
5. Drill holes in the aluminum rod so that the cooling circulating water can pass through
6. Install the appropriate Swagelok adapters at the drilled position

Section 4 Connect all the electronics in the photoreactor

Arduino Boards were used to power all the electronics in the photoreactor. A step-by-step assembly is as follows:

1. Connect the right pins of the LEDs with Arduino Boards (5 pcs required)
2. Connect the 12V DC axial stirring fans (4 pcs required) with Arduino Boards
3. Connect the 12V DC axial cooling fans (2 pcs required) with Arduino Boards
4. Connect the temperature/humidity sensors with Arduino Boards
5. Connect the AC/DC converter (type 24 V, 15 A) with Arduino Boards
6. Connect the DC/DC Buck converters (3 pcs required, LED controller) with Arduino Boards

7. Install adjustable Module Buck converters (2 pcs required, fan controller) with Arduino Boards
8. Install OLED displays (5 pcs required) on the Arduino Boards
9. Install resistor status indicators to disclose the status of electronics
10. Fasten the Arduino Boards to the stainless steel plate as shown in Fig. 1d
11. Use the Arduino Boards to run the self-developed Python code to control the photoreactor

Section 5 Assemble all the components to build the batch photoreactor

Once all the preparative works were done, the final assembly of the photoreactor is quite straightforward. The only thing that needs to do is to place all the subassembly into its preset position. A step-by-step assembly is as follows:

1. Cut four pieces of 3-face Bosch profile to a dimension of 20 mm × 20 mm × 200 mm (4 pcs required)
2. Insert all stainless steel plates as detailed in Section 1 into the cut Bosch profiles
3. Install the heat exchanger in a proper position and seal the perimeter of the heat exchanger with sealing tapes
4. Position the aluminum rods and magnetic stirring system in the preset location
5. Position all the electronics in the preset location
6. Connect the heat exchanger and aluminum rods with suitable Swagelok adapters and silicone tubing
7. Use two of three-way valve to distribute the cooling water from the circulating cooling bath
8. Connect the inlet and outlet of the tubing with circulating cooling bath
9. Cover the photoreactor with top plate (Fig. 1a) and secure the aluminum rods with Aluminum-cooler holder (Fig. 2a)
10. Secure all the parts of the photoreactor with screws and nuts

11. Use spare Bosch Profile to lift the photoreactor in a proper position for use

Section 6 Test the stability and evaluate the performance

1. Run the photoreactor with water
2. Check the light exposure issues
3. Check if the temperature monitoring system is working
4. Check if the light intensity is dimmable
5. Check if the LEDs are still working after long-time operation

Following the previous steps, the photoreactor can be easily assembled. Fig. 3 gives a schematic front view of partially assembled photoreactor.

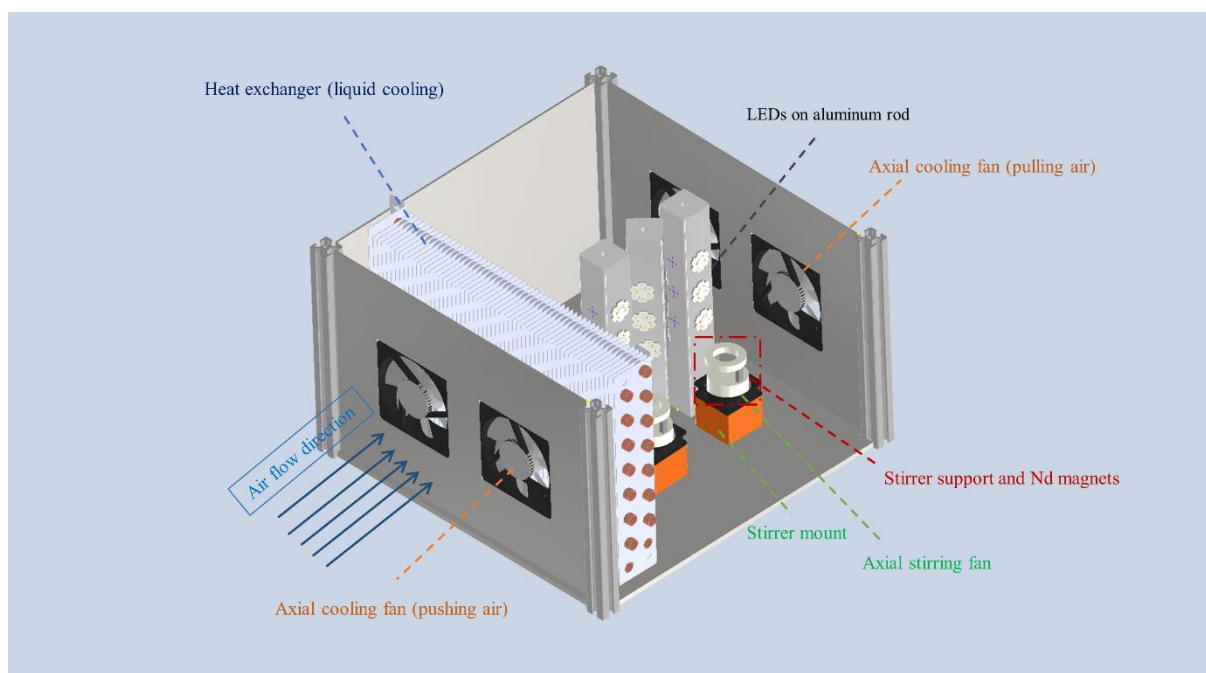


Figure 3 Schematic view of partially assembled photoreactor

2.2 Final design and descriptions

The final design of the photoreactor is shown in Fig. 4. Two axial cooling fans are installed on both sides of the photoreactor to ensure good air cooling. The self-developed Python code enables the fans usually work at 80% power, once the sensor detects the system temperature is above the setting value (in our setup, the value is set at 30 degrees Celsius), the fan will accelerate and work at full power. When the circulating cooling bath is working, the system

temperature can even reach lower values. Therefore, the photoreactor can work at a broad operating temperature range. The distance between the test tube and the LEDs is fixed at around 2.5 cm, and the symmetrical design standardizes the operation conditions because the same position of the tube is used for each independent experiment. The proposed photoreactor setup is able to run 4 reactions in parallel with each test tube illuminated by two LEDs (total irradiation power, 20 W per tube), thereby increasing the efficiency of reaction screening. Of course, one can combine more light sources to increase the total number of reactions that can be run. In the current design, three wavelengths of LEDs are installed for different purposes of reaction screening and can be easily replaced with other wavelengths of interest by inserting new aluminum rods and gluing the LEDs to them. In addition, with the controllable Arduino Board, the research can manually change the output power of the LEDs and monitor the output of the parameters from the digital display screens. Therefore, the corresponding light intensity is tunable. The flexibility of the photoreactor can be achieved by printing different sizes of test tube guide and holder. In addition, the built-in stirring system endows the photoreactor with portability, which can easily transfer from one lab to another lab. Another advantage is that there is no need to include an external magnetic stirring system, as is the case with many batch photoreactors. The total price (without LEDs) of the photoreactor is very cheap (roughly below €400) and the majority of research groups will afford it.

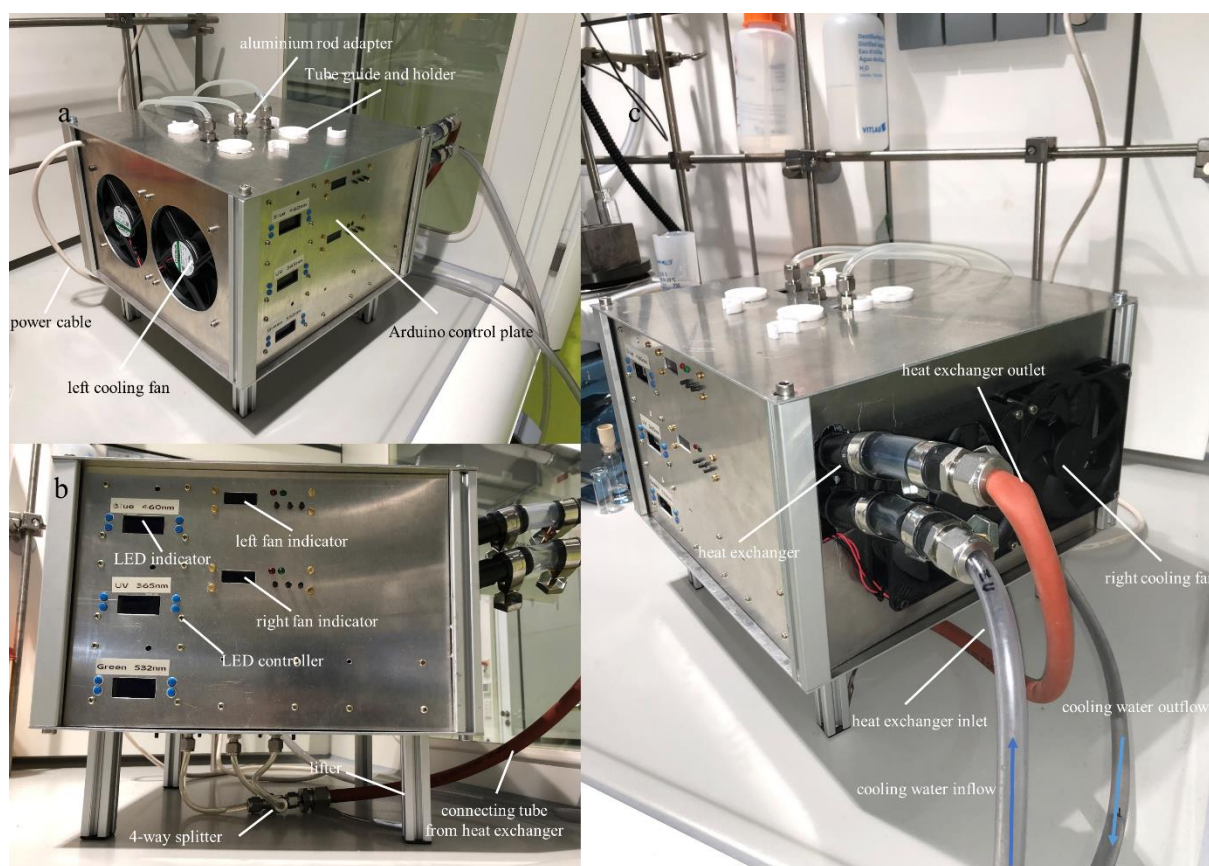


Figure 4 Final design of the batch photoreactor. a) Left side view b) front side view and c) right side view

3. Conclusion

In summary, we have successfully constructed a novel, economical, portable and multifunctional batch photoreactor. The easily reproducible photoreactor design standardizes the reaction conditions in photochemistry and fulfills the criterion mentioned before. All the components for assembling the photoreactor are commercially available and the overall cost is very low. At the same time, the touch-screen operation facilitates its use for amateur researchers. We believe this technical note will help readers who want to build something similar. The corresponding Inventor files and Python code are available in the supplementary.

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Conflict of Interest

The authors declare no conflict of interest.

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