Optimization of a Photocatalytic Flow Reactor to measure the Hydrogen evolution reaction activity of Water-Splitting Photocatalysts

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Introduction

Photocatalytic water splitting is a sustainable method of producing hydrogen fuel using sunlight, water, and a semiconductor photocatalyst like TiO_2. When exposed to light, the catalyst generates electron-hole pairs that drive the water-splitting reaction, releasing hydrogen and oxygen. First demonstrated in 1972, this process offers a clean alternative to fossil fuels. Current research focuses on improving efficiency by enhancing light absorption and charge separation through catalyst modification.

Goal for the Project

- Commissioning of the Photocatalytic flow reactor and authorship of the standard operation procedure

Improvements from the Previous Setup

The integration of a CoolLED [pE-4000] light in the revamped flow reactor marks a first for our setup, offering stable, low-heat illumination.
Unlike Xe lamps, which were previously used, LEDs consume less power, generate minimal heat, and provide consistent light output.
This upgrade improves experimental accuracy and reproducibility, setting a more reliable standard for future photocatalysis and hydrogen production studies

Reaction Scheme

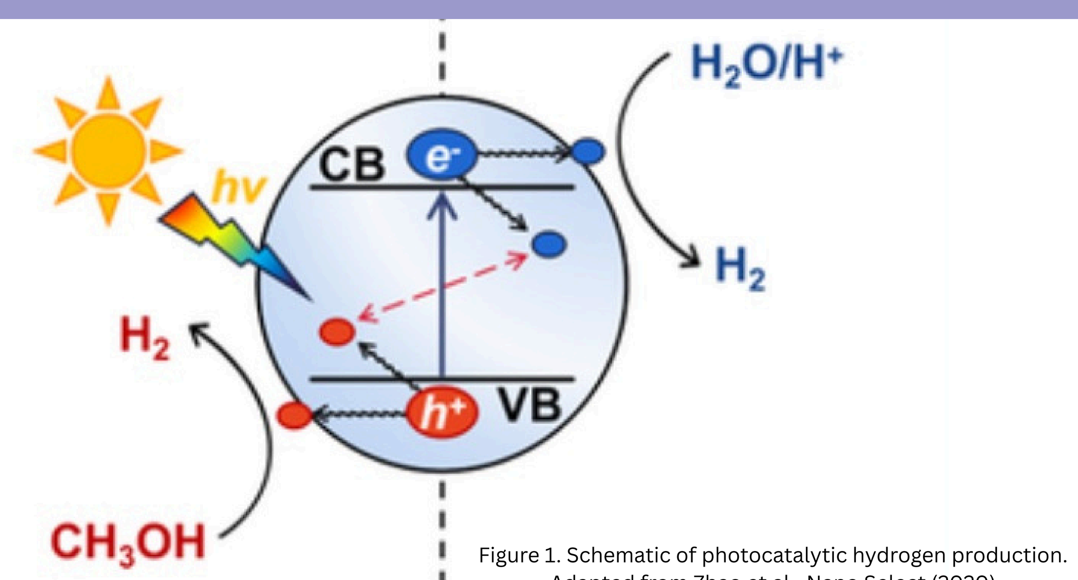
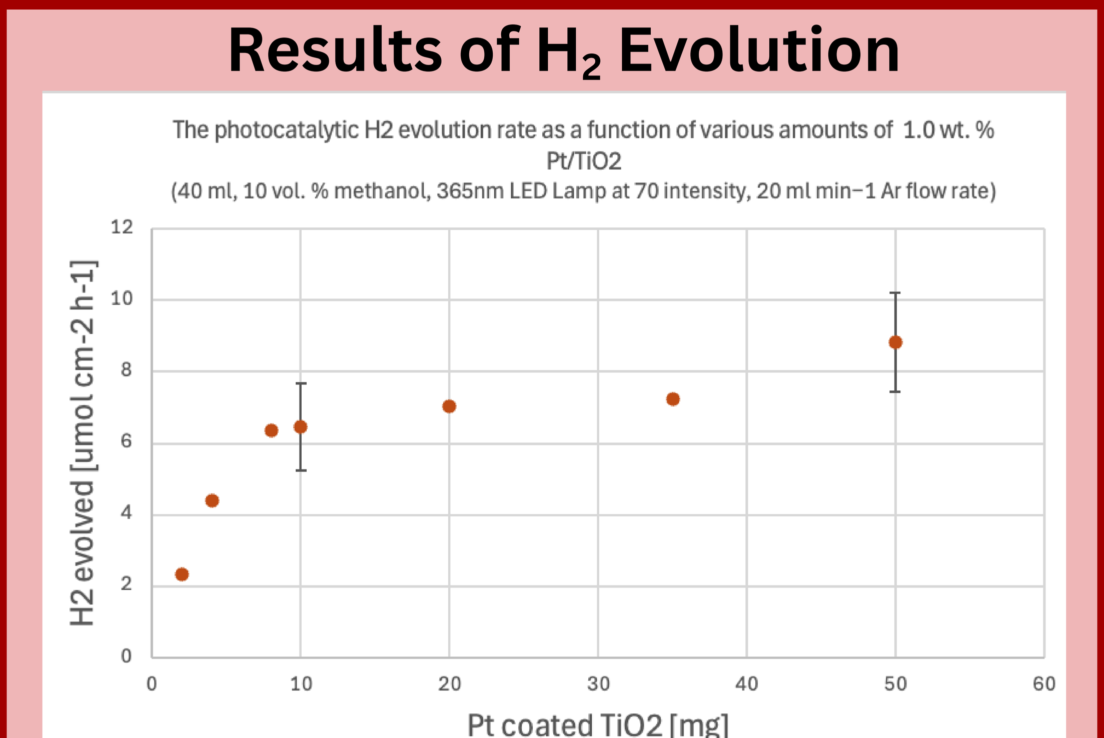
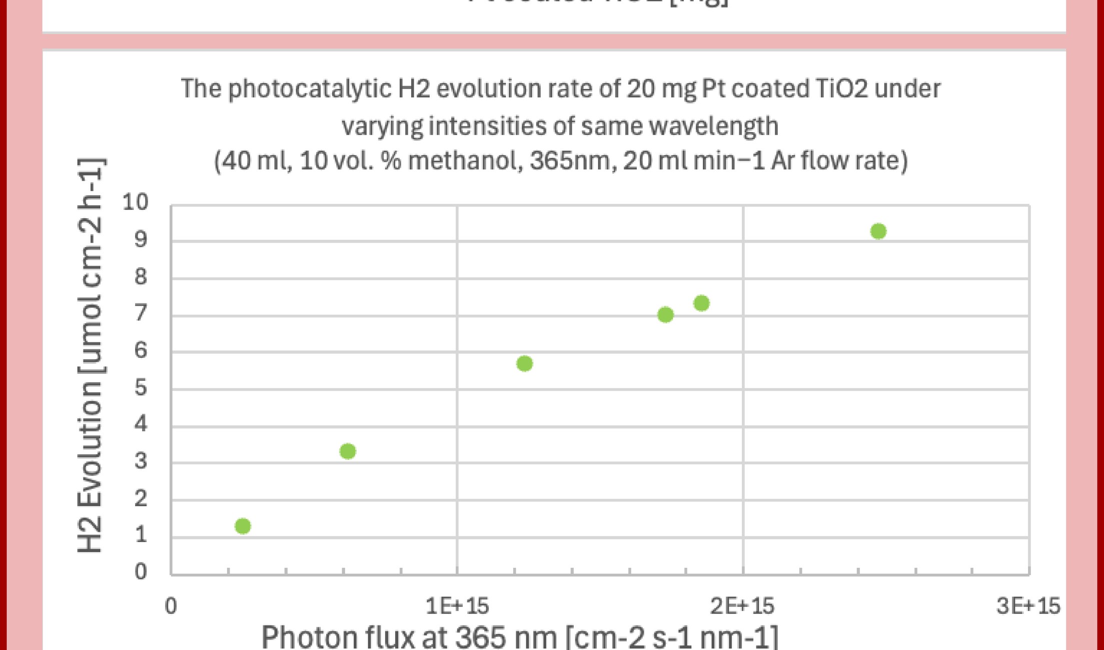
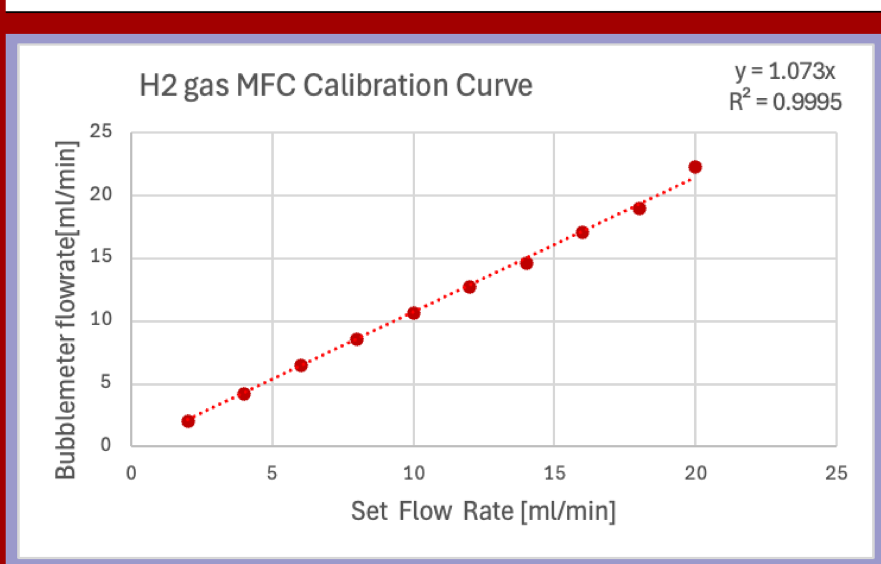


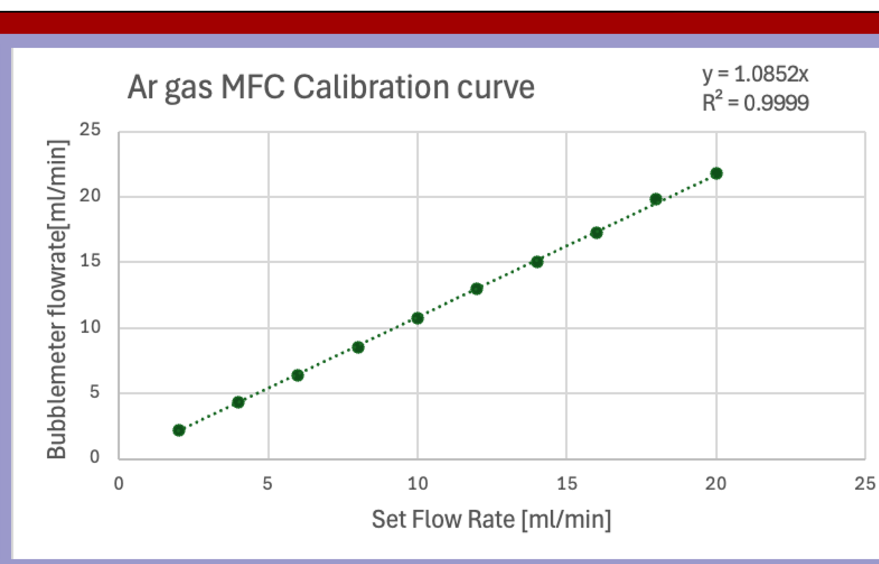
Figure 1. Schematic of photocatalytic hydrogen production. Adapted from Zhao et al., Nano Select (2020).

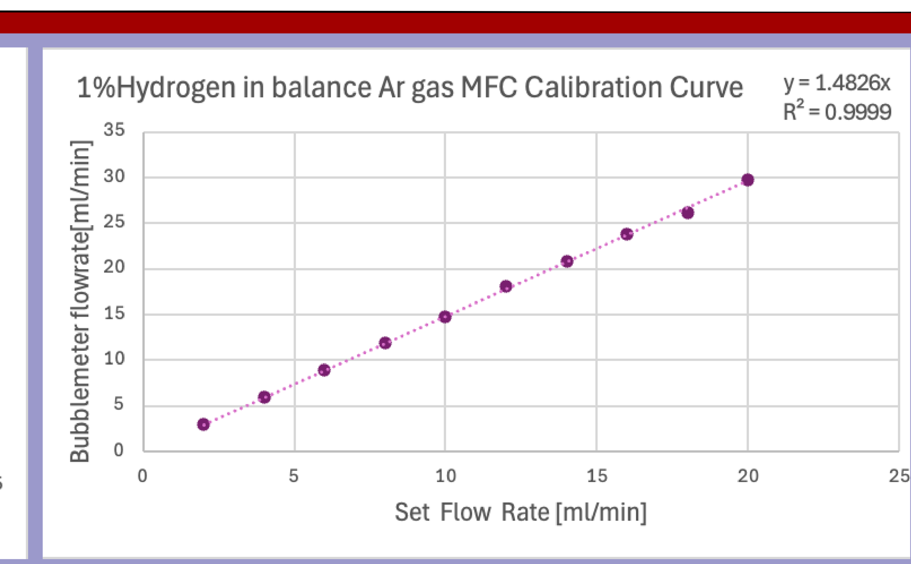
Results of H_2 Evolution





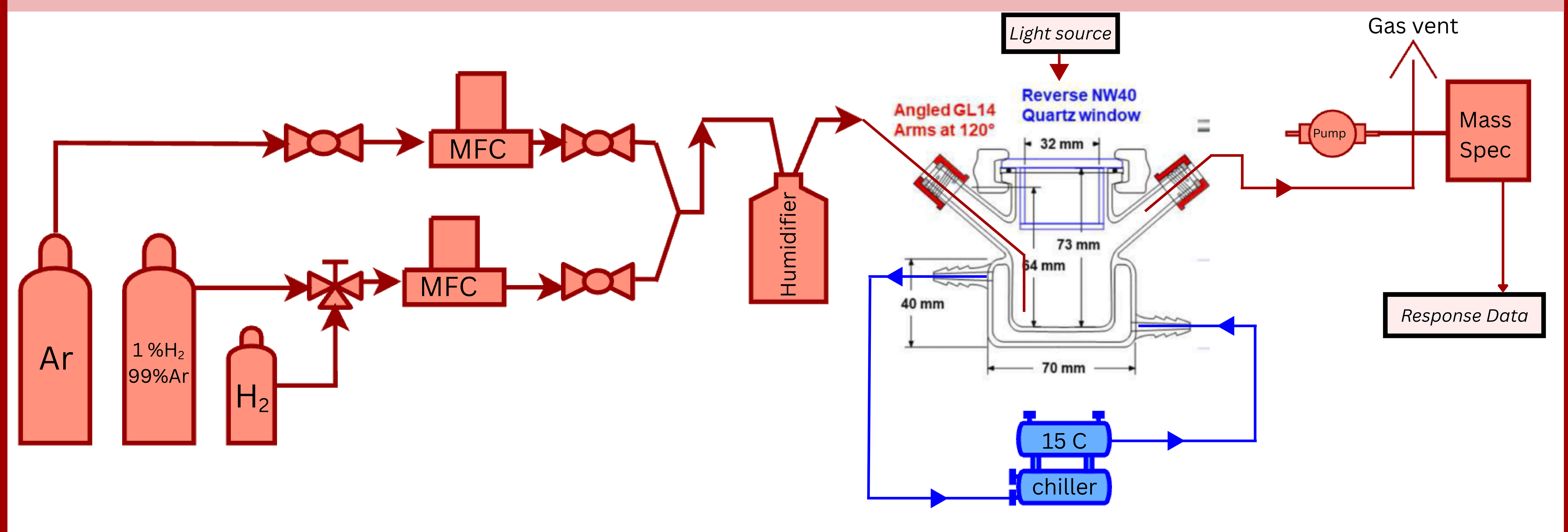


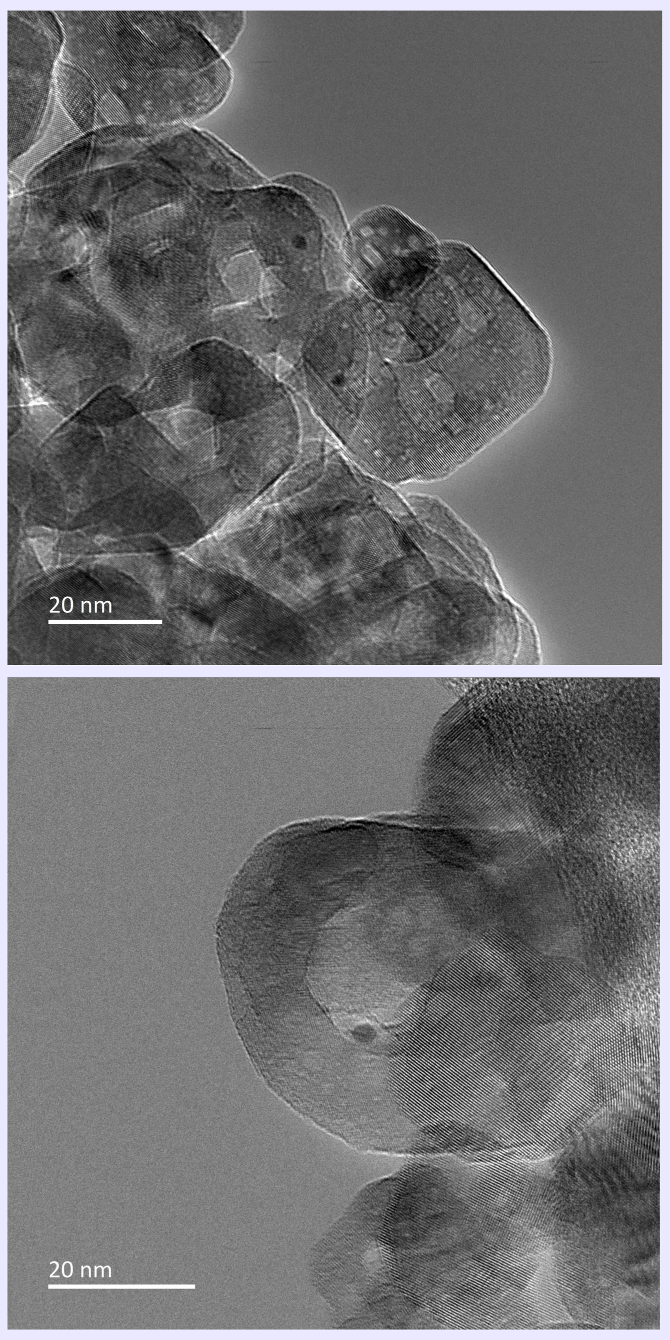




Mass Flow Controller Calibration Curves [Used to calculate and set the desired amount of gas output from the MFCs]

Experimental Setup / Schematic Diagram



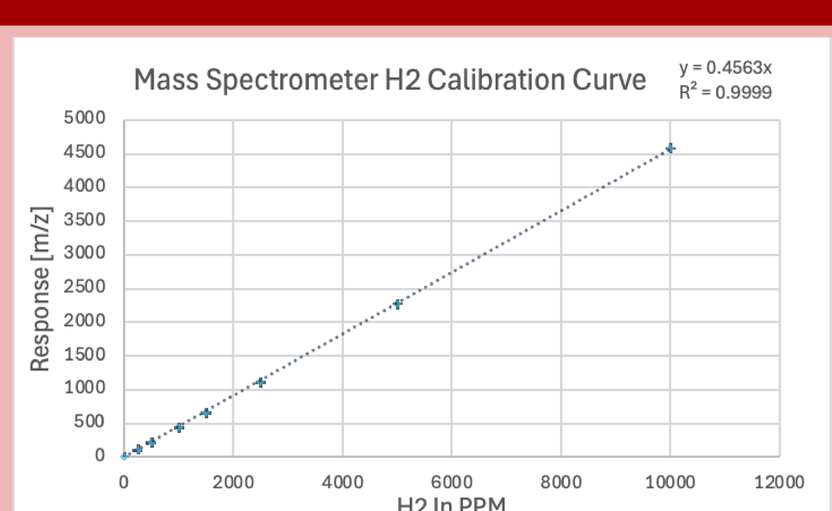


Transmission Electron Microscopy[TEM] of Pt-modified TiO2 via photodeposition.

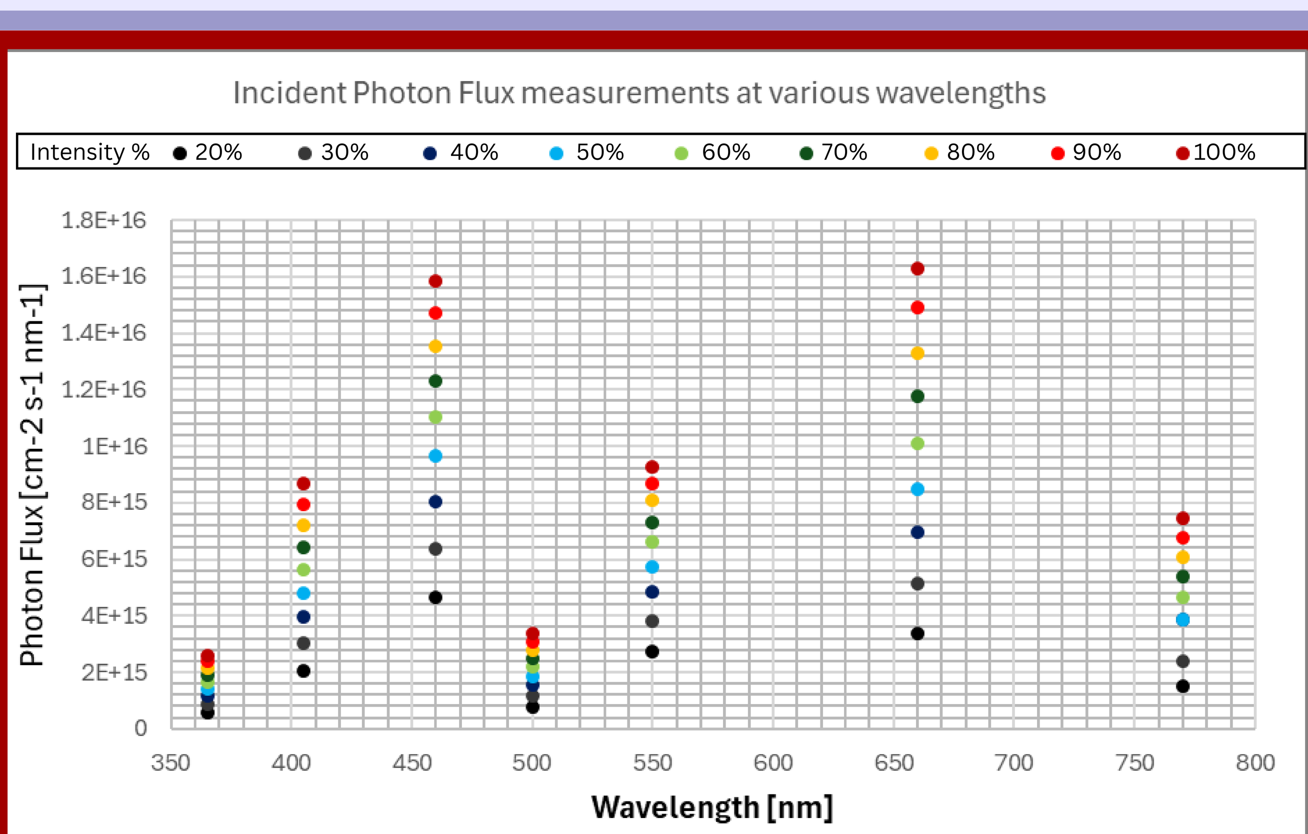
Experimental Protocol

1. Establish appropriate tube and valve connections across all components.
2. Connect the photocatalytic reactor to the chiller unit for temperature control.
3. Calibrate the Mass Flow Controllers (MFCs) to ensure accurate gas flow rates.
4. Calibrate and tune hydrogen detection on the Real Gas Analyzer (RTGA).
5. Perform a complete leak check of the system to ensure airtight operation.
6. Initiate the photocatalytic run under controlled flow and temperature conditions.

Once the system is fully calibrated and tuned, the Argon carrier gas is set to flow at a constant rate of 20 mL/min. The gas first passes through a humidifier containing 10% methanol in *Mili-Q Water*. It then enters the reactor, which contains 40 mL of 10% methanol aqueous solution *[Mili-Q water]*, the desired amount of photocatalyst *[Anatase TiO_2(25nm, Sigma Aldrich)]*, and Potassium Hexachloroplatinate *(K_2PtCl_6)[Sigma Aldrich, ≥99.99% trace metals basis]* as the Platinum precursor.
Downstream, the gas is then pulled by a pump connected to a mass spectrometer (MS) for hydrogen detection. At last, the exhaust gas is safely vented into a fume hood.
Illumination of the reactor under LED light at 365nm initiates the photodeposition of platinum onto titanium dioxide, forming active catalytic sites that drive hydrogen evolution.



H_2 Calibration Curve on the RTGA MS which was Used in umol/hr cm^2calculations



Outlook

This work lays the foundation for wavelength-specific photocatalytic studies using the CoolLED PE-4000 system. The developed SOP will enable future users to operate the system efficiently, safely, ensure reproducibility across experiments, and expand its application to a wide range of photocatalytic reactions, like to perform photocatalytic CO_2 reduction.
Moving forward, this setup can support systematic studies on light-dependent catalyst performance and contribute to the development of more efficient solar-to-fuel technologies.

Acknowledgements

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References

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