

Photocatalytic Production of H₂ from Biomass

Gas-phase Hydrogen Measurements using a Unisense Needle Sensor

The application note is based on the research and article by:

Beckedorf et al.

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Petersen et al., Unisense

Introduction

Hydrogen is widely regarded as a promising alternative to fossil fuels due to its high gravimetric energy density and clean combustion, producing only water as a by-product. However, the sustainability of hydrogen as an energy carrier is strongly dependent on how it is produced. Conventional routes, such as steam methane reforming, remain carbon-intensive, which motivates the development of low-emission strategies.

Photocatalytic hydrogen production through water splitting offers an attractive approach by directly converting solar energy into hydrogen. However, the water oxidation half-reaction is associated with high thermodynamic barriers, which limit the overall efficiency. One strategy to overcome these limitations is the use of a sacrificial electron donor instead of water.

“Unisense’s microsensors have the versatility that helps my research. They are compact and easy to setup, which is critical for us to use them in different types of setups where we can integrate different excitation light and operando optical spectroscopy techniques. The customer service is superb, they give great troubleshooting advice, and are always eager to try to find creative solutions.”

Robert Godin, Assistant Professor,
The University of British Columbia

In a 2025 study, Beckedorf et al. used an agricultural by-product in the form of lignocellulosic biomass as a sacrificial electron donor, which simultaneously facilitates hydrogen evolution and consumes biomass. Carbon nitride serves as the photocatalyst, and the authors aimed to enhance its photocatalytic activity by loading it with the Earth-abundant metal cobalt.

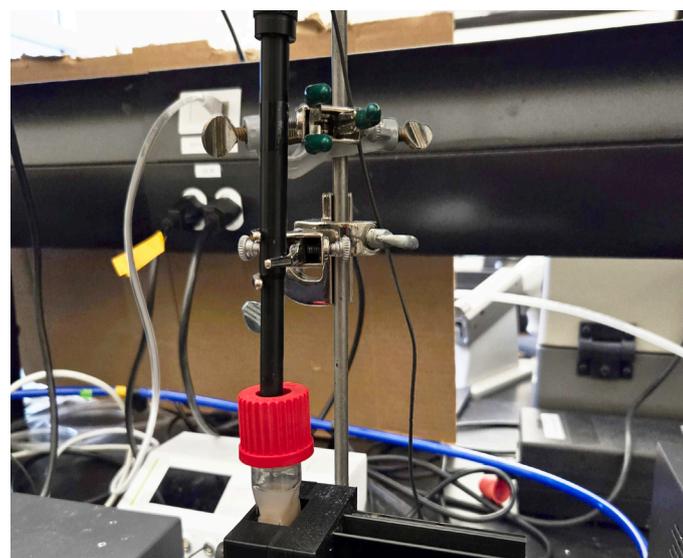


Figure 1: The hydrogen sensor in a needle for piercing inserted through a septum in the lid of the cuvette.

Laboratory setup and methods

Photocatalytic hydrogen evolution experiments were carried out in a 16 mL sealed quartz cuvette under irradiation with a 405 nm LED. The reaction suspension contained the carbon nitride-based photocatalyst and a selected biomass-derived sacrificial electron donor in an alkaline aqueous solution of 10 mL. Prior to illumination, the system was purged with argon to remove hydrogen.

Hydrogen production was quantified by measuring the hydrogen partial pressure in the cuvette headspace. For this purpose, a Unisense Clark-type hydrogen microsensor in a needle for piercing was used. After irradiation, the cuvette was thoroughly shaken to equilibrate hydrogen between the liquid phase and headspace, and the sensor was inserted into the headspace for the measurement. The total amount of hydrogen produced in the cuvette was calculated from the hydrogen partial pressure using the ideal gas law and Henry’s law.



O₂

N₂O

H₂S

NO

H₂

pH

Redox

Temp

EP

Calculations

By thoroughly shaking the cuvette prior to the headspace measurement, equilibrium between the liquid and gas phases was established, resulting in identical hydrogen partial pressures in both phases. The amount of hydrogen in the 6 mL headspace was calculated using the ideal gas law. An example calculation using an arbitrary hydrogen partial pressure of 1500 Pa is shown below.

Gas phase:

$$n = \frac{P \cdot V_1}{R \cdot T} = \frac{1500 \text{ Pa} \cdot 0.006 \text{ L}}{8314 \text{ L} \cdot \text{Pa} \cdot (\text{mol} \cdot \text{K})^{-1} \cdot 293.15 \text{ K}} = 3.69 \cdot 10^{-6} \text{ mol} = \mathbf{3.69 \mu\text{mol}}$$

The amount of hydrogen dissolved in the 10 mL liquid phase was calculated using Henry's law, applying a Henry's constant for hydrogen in water at 298.15 K of $k = 7.8 \cdot 10^{-9} \frac{\text{mol}}{\text{L} \cdot \text{Pa}}$.

Liquid phase:

$$c = k \cdot P = 7.8 \cdot 10^{-9} \frac{\text{mol}}{\text{L} \cdot \text{Pa}} \cdot 1500 \text{ Pa} = 11.7 \cdot 10^{-6} \frac{\text{mol}}{\text{L}}$$

The number of moles of dissolved hydrogen was then calculated from the concentration:

$$n = c \cdot V_2 = 11.7 \cdot 10^{-6} \frac{\text{mol}}{\text{L}} \cdot 0.010 \text{ L} = 0.12 \cdot 10^{-6} \text{ mol} = \mathbf{0.12 \mu\text{mol}}$$

The hydrogen present in the gas and liquid phases can be summed to determine the total amount of hydrogen in the cuvette. With 3.69 μmol of hydrogen in the 6 mL gas phase and 0.12 μmol in the 10 mL liquid phase, the liquid-phase hydrogen accounts for approximately 3.25% of the total hydrogen, highlighting the low solubility of hydrogen in water.

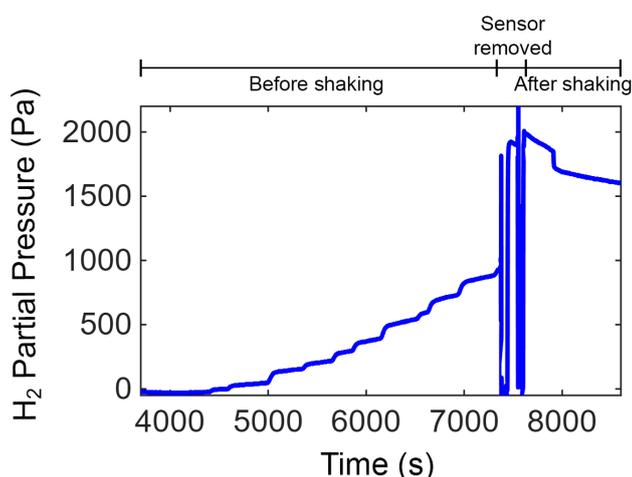


Figure 2: Continuous hydrogen measurements in cuvette headspace.

Results and conclusion

Hydrogen production from the carbon nitride-based photocatalyst in the presence of a biomass-derived sacrificial electron donor in alkaline aqueous solution was quantified via gas-phase measurements. During irradiation, equilibration between the liquid and gas phases was not performed continuously, resulting in a modest increase in the measured hydrogen partial pressure in the headspace (Fig. 2).

Gas-phase measurements obtained after equilibration showed higher hydrogen partial pressures, as hydrogen dissolved in the liquid phase redistributed into the headspace. These end-point measurements were used to calculate the total amount of hydrogen produced for the different photocatalyst compositions.

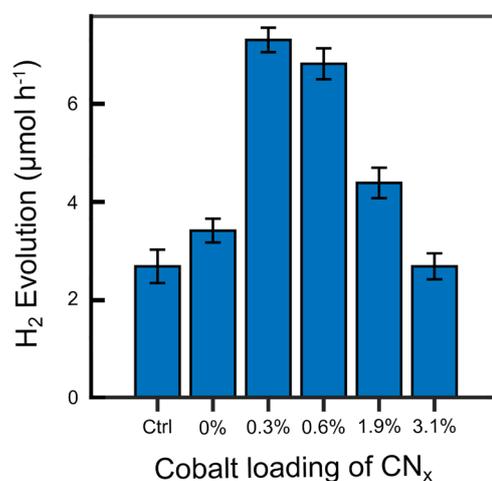


Figure 3: Accumulated hydrogen content per hour using different carbon nitride catalysts.

Carbon nitride photocatalysts loaded with varying amounts of cobalt as a cocatalyst were evaluated for hydrogen production. Photocatalysts containing 0.3% and 0.6% (wt.%) cobalt showed the highest hydrogen yields, reaching nearly three times the hydrogen production of the cobalt-free control (Fig. 3). At higher cobalt loadings, hydrogen production decreased, which is attributed to partial blocking of the limited active sites on the carbon nitride surface and reduced light absorption.

The microsensor-based approach enabled sensitive, real-time detection of hydrogen evolution in the sealed cuvette. In this study, gas-phase measurements were used to calculate the total hydrogen produced; however, the same microsensor approach can be applied to measure hydrogen directly in both the liquid and gas phases simultaneously, enabling validation of phase equilibration and further improving measurement accuracy.

Suggested products

- H₂-NP and H₂-NPLR
- H₂ UniAmp
- Temp UniAmp
- uSense Log



References

Read more in the article by Beckedorf et al. "Cobalt-Loaded Carbon Nitride Demonstrates Enhanced Photocatalytic Production of H₂ from Lignocellulosic Biomass Components", *Artif. Photosynth.* 2025, 1, 50-62.

Related publications

Berglund, Sigrid, et al. "Hydrogen production by a fully de novo enzyme." *Dalton Transactions* 53.31 (2024): 12905-12916.

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