

Hydrogen Evolution Reaction with Sunlight for School Chemistry Education

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Abstract In current research, molecular light-driven catalytic units are linked to hierarchically structured soft matter matrices and used to convert solar radiation into chemical reactivity for photocatalytic water splitting. This article describes the development of a simplified hydrogen evolution reaction that transfers selected results to the student laboratory in a phenomenon-oriented manner. For this purpose, a photocatalytically active system - consisting of the catalyst titanium dioxide, the dye Eosin Y and the electron donor triethanolamine - is combined with two polymers as suitable matrices. A newly developed, low-cost hydrogen detector is used to quantify the hydrogen concentration.

Keywords: photocatalysis, hydrogen evolution reaction, photochemistry, low-cost detection, water splitting

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1. Introduction

Chemical reactions with (solar) light form the basis of numerous processes in the biosphere. They also find broad applications in everyday life, science and technology, ranging from solar collectors over photocatalytic wastewater treatment to air-cleaning wall paint [1] and further. Because of their strong connection to everyday life, their great importance in the present and future, and the possibilities for interdisciplinary considerations and contexts, they offer equally great potential for teaching in formal and nonformal educational settings. From a didactic point of view, they offer a wide range of interdisciplinary learning opportunities and numerous links to the basic concepts *energy*, *donator-acceptor* and *structure-property*. In addition, there are numerous links to many classic contents and content areas of chemistry teaching at secondary level II - just one example of how this can be implemented in an everyday-oriented manner is described in the literature [2].

For these and other reasons, the subject area "Chemistry with Light" is the object of research of several didactic research groups and is systematically described in a recent publication for teaching in class [3]. Nevertheless, the potential is currently only partially exploited in the classroom, since some curricula do not yet take the topic into account (enough) or are still limited to superficial considerations and a few examples of application. More

in-depth possibilities and learning opportunities are given, for example, by the use of light for photocatalytic reactions with consideration of the underlying mechanisms [2], the synthesis of organic LEDs (OLEDs) [4], quantum-dot based LEDs (QLEDs) [5], photochromism [6], artificial photosynthesis [7] or photocatalytic water-splitting with UV light [8]. Equally interesting with regard to future societal importance and climate change are application-oriented questions in the context of bionics, *i.e.* how the potential of these materials can be used to achieve, for example, artificial photosynthesis with the highest possible efficiency.

However, current scientific research on this socially highly relevant objective has not yet been sufficiently communicated in schools and student laboratories, nor has the contribution that the corresponding technologies can potentially make to society as a whole (*e.g.* energy supply, mobility, climate change) been considered enough.

In the CataLight collaborative research center of the universities of Jena, Ulm, Mainz and Vienna, these issues are being researched in an equally fundamental, application-oriented and interdisciplinary manner [9]. In addition to the development of new photocenters and catalysts, the interactions with a surrounding soft matrix are also being systematically examined for the first time. By combining these elements - photosystem with a matrix - it is intended to address central challenges that currently prevent transfer to application, such as stabilization, regulation, and regeneration of the materials. Corresponding findings offer great opportunities to synergistically link

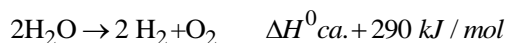
current research contexts with classical contents of chemistry education and societal challenges. Contexts that are currently omnipresent in the media, such as mobility or the energy supply of the future also offer numerous opportunities for discussion ("Fridays for Future").

A central objective of the project is the regenerative and efficient generation of hydrogen with sunlight. Compared to photocatalytic water splitting with high-energy UV light [10], the implementation with lower-energy sunlight offers significantly more challenges - but ultimately also far more potential for a broad, efficient and sustainable energy supply.

In this article, new findings from scientific research are briefly presented followed by a demonstration, how these results can be didactically communicated in schools and student laboratories through an experimental approach. For this purpose, a series of experiments on the production and detection of hydrogen is presented, which can be carried out with simple and harmless chemicals and equipment.

2. Scientific Background

Nature uses the process of photosynthesis to produce energy storage materials, *e.g.* sugar, from sunlight, water and carbon dioxide. Photosynthesis thus converts solar energy into chemical energy. The energy is subsequently stored in chemical bonds. Research groups around the world are pursuing this goal in studies on so-called artificial photosynthesis. The aim of this process is to use sunlight to split water into its elements, *i.e.* hydrogen and oxygen. Looking at the underlying reaction:



At first glance, these appear to be quite simple. In detail, however, both redox half-reactions, *i.e.* the water oxidation to oxygen and the water reduction to hydrogen, are quite complex, since they are so-called proton-coupled multi-electron reactions. For the water reduction, for example, it can be seen that two protons and two electrons have to be reacted together to produce one molecule of hydrogen. This requires complex and highly specific catalysts. Of great interest at present are molybdenum sulfide solids, which combine high reactivity, stability, and low initial cost. In the context of this article, we use the molecular analogues of molybdenum sulfides, the so-called thiomolybdates.

If endothermic water splitting is to be carried out with sunlight, a photosensitive molecule must also be integrated into the reaction system as a light collector. In fundamental studies, ruthenium complexes are frequently used, since they combine high light absorption with good electron transfer properties.

Since the light collector and catalyst can only interact if they are in close spatial proximity, the joint deposition of both molecules on solid materials such as semiconductors or polymers is used. The method results in a photosensitive, catalytically active solid, also known as a hybrid material due to the combination of often inorganic and organic components. The incorporation into a polymer matrix achieved in this way improves the solubility

(dispersibility) of many catalysts or sensitizers in an appropriate medium (in this case, water), on the one hand, and on the other hand, this route ensures sufficient proximity of both components for efficient subsequent photocatalysis.

As a further advantage, the solid can be easily separated from the liquid reaction mixture after the reaction is complete and reused. In this article, a simplified model system consisting of a metal oxide solid substrate (TiO_2), a model catalyst (thiomolybdate, $(\text{NH}_4)_2[\text{Mo}_3\text{S}_{13}]$) and the light collector Eosin Y is used to didactically reconstruct on light-driven water reduction and hydrogen formation in such a system.

3. Didactic Reconstruction

For a successful transfer of these findings to schools, there are three central challenges in particular:

(1) Didactic reconstruction: Due to its high degree of specialization, current (basic) research is usually very demanding and difficult for learners to comprehend. Especially in the understanding of light, there are inaccurate preconceptions or misconceptions on the part of the learners [3], which have to be addressed.

(2) Experiments and analytics: Current research often relies on equipment and chemicals that cannot be used in schools for cost reasons and/or have a high hazard potential. In addition, schools and student laboratories typically do not have access to scientific analytics.

(3) (Perceived) barriers from teachers: Many teachers are motivated to teach new subject areas in their classes, but often reach and/or perceive multiple barriers to teaching new subject areas in their classes. These include, in particular, a lack of specialist knowledge, a lack of teaching materials and a lack of teaching time [11].

In the experimental-conceptual didactics of chemistry, various research concepts are applied to the development process of subject-specific chemical contents in order to implement corresponding projects successfully [12]. Widely approved models are for example the model of Curricular Innovation Research [13] or the model of Didactic Reconstruction [14]. Special chances regarding the quality and the practice connection result, if the reconstruction process includes the expertise of didactics, chemistry and teaching practice. In this way, on the one hand, the chemical and practical expertise can be used and, on the other hand, barriers to the transfer into school practice can be better addressed already during the development.

Due to their key position between research and school, student laboratories are the ideal learning location for this, as all these actors come together in this place. Their direct connection to the university (1) offers material degrees of freedom for the development and testing of teaching concepts and materials, (2) allows subject-specific research and research groups to be directly involved on site, and (3) can significantly accelerate the practical testing and optimization of teaching offers in the sense of "rapid prototyping". Thus, student laboratories have the potential to act as a catalyst for the transfer of current research content into formal education.

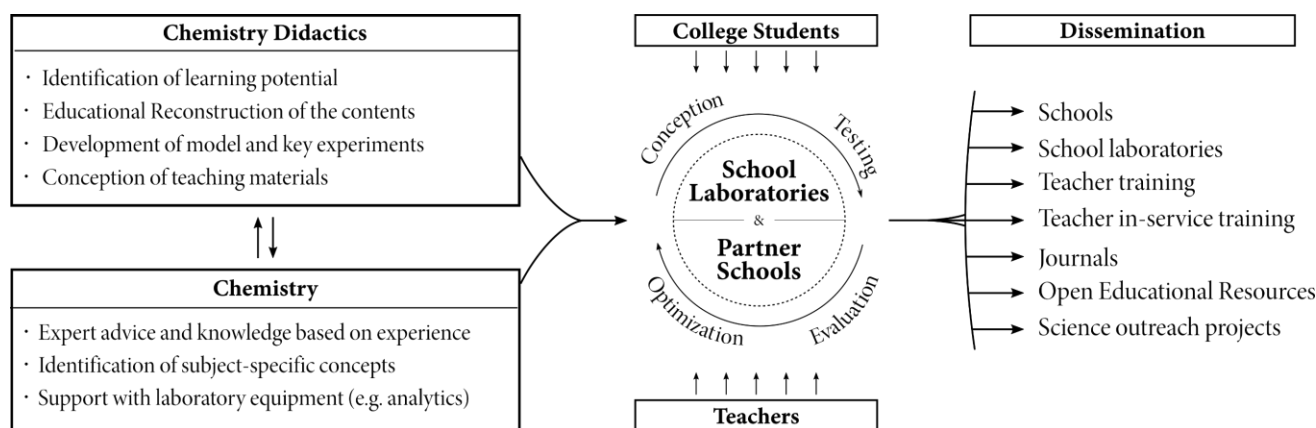


Figure 1. Model of Educational Transfer Research

This potential is reflected in the model of Educational Transfer Research (Figure 1) [15]. In three sections, it describes (1) the didactic reconstruction of the content in collaboration with subject scientists, (2) the development of teaching materials in a cyclical process of conception, testing, evaluation, and optimization with the involvement of teachers and students in student labs and partner schools, and (3) the final dissemination of the tested materials into teaching practice.

As the first building block of this process, we describe a series of experiments on the evolution reaction and detection of hydrogen, that can be carried out in the school laboratory and as part of project work or seminar papers.

4. Experimental Part

As catalysts for hydrogen evolution reaction with sunlight, titanium dioxide (photocatalyst), ruthenium complexes (photosensitive molecules) and thiomolybdate (co-catalyst) have emerged as promising candidates for some time. Current scientific work is focused on maximizing the hydrogen yield of these systems [16,17,18]. One challenge here is the control of electron transfer between the systems involved. In this regard, one current approach is the development of polyelectrolytic graft copolymers [19,20]. These have tailored properties and functional groups that allow immobilization of both the catalyst and the photosensitizer. As a result, electron transfer between the systems is facilitated, which can result in increased

hydrogen production as well as longer lifetime [19].

In the following, this basic principle will be reconstructed within a student experiment. In order for this system to be used in a school experiment, numerous aspects have been addressed and simplified – compared to the procedure used in research: (1) Ruthenium(II) complexes cannot be used in the school laboratory due to their high acquisition cost as well as their sensitivity to oxygen. Instead, the inexpensive dye Eosin Y, which is also used in cosmetics and has already been described several times in chemistry didactics [21], was applied as a photosensitive molecule. (2) The synthesis of custom-made polyelectrolytic graft copolymers [19,20] is also not feasible in schools for a variety of reasons and were substituted with polyacrylic acid (PAA) and polydiallyldimethyl chloride (PDADMAC) as commercially available, inexpensive and non-hazardous alternatives. Both polymers exhibit sufficiently good binding properties for the catalytic system used. (3) In view of any potentially hazardous potential, toxic chemicals (methanol) were waived; (4) also, no inert gas atmosphere is used to enable easier sample preparation for students. (5) It should also be possible to optionally dispense with the commercially unavailable co-catalyst (ammonium thiomolybdate, $(\text{NH}_4)_2[\text{Mo}_3\text{S}_{13}] \cdot 2\text{H}_2\text{O}$). All experiments require only very small amounts of the chemicals. However, if precision balances or microliter pipettes are not available at external sites, higher concentrations can easily be used. Figure 2 summarizes the adjustments made to the system during the transfer from research to the student laboratory.

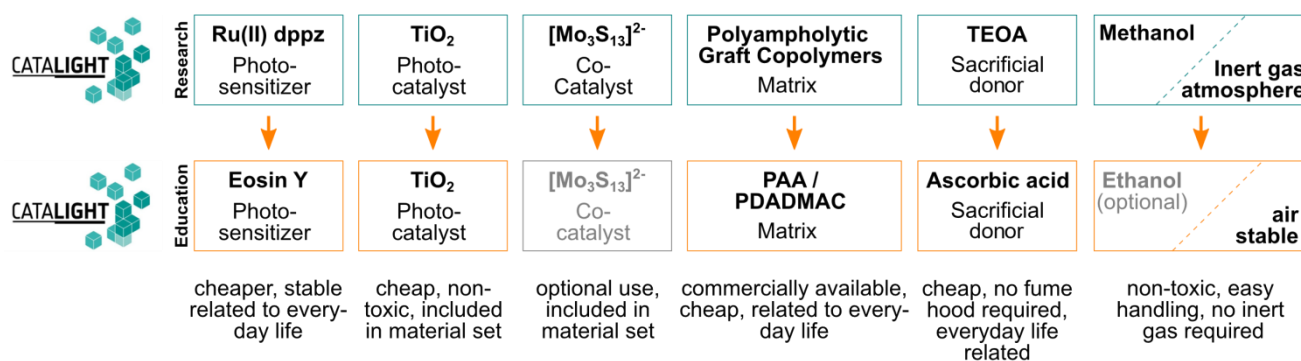


Figure 2. Development of a simple system for photocatalytic water splitting for student laboratories. The changes compared to the system which is used for CATALIGHT research are marked in orange

4.1. Hydrogen Formation with Sunlight

A mixture of polyacrylic acid (PAA) and polydiallyldimethyl chloride (PDADMAC) is selected as the polymer system with polyelectrolytic properties. PAA has multiple cosmetic and pharmaceutical applications, while PDADMAC is used as a common cationic fixative in papermaking, among other applications (see Figure 3). Both chemicals are cheap, widely available and non-hazardous.

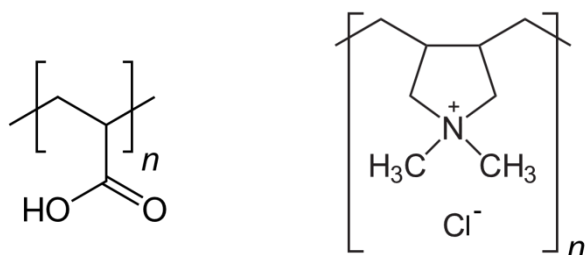


Figure 3. PAA (left), PDADMAC (right)

The polymers are prepared with a molar ratio of $n_{\text{PAA}} : n_{\text{PDADMAC}} = 0.5$. This system forms water-soluble, so-called interpolyelectrolyte complexes of oppositely charged polymers at a pH of 9, which can act as model polyelectrolytes for tailor-made graft copolymers. Specifically, the negatively charged carboxyl groups of PAA can immobilize titanium dioxide; similarly, the positively charged PDADMAC can immobilize the dye Eosin Y (and also thiomolybdate), facilitating electron transfer between the components and enhancing hydrogen generation.

In order to minimize sources of error or distortion and to ensure a comparable standard, a halogen lamp was chosen as the light source in this experiment, since its UV light content is much lower than that of sunlight [22]. Alternatively, LED spotlights or sunlight can be selected as the light source for this experiment.

Chemicals, materials: polydiallyldimethylammonium chloride (Sigma Aldrich, MW = 100,000, 10 g·L⁻¹, “PDADMAC”), polyacrylic acid (Sigma Aldrich, MW = 2,000, 1 g·L⁻¹, “PAA”), titanium dioxide (Sigma Aldrich, 1 g·L⁻¹, “TiO₂”), Eosin Y (Fisher Scientific, 0.692 g·L⁻¹), triethanolamine solution (Sigma Aldrich, 99%, diluted 1:1 with dest. water, “TEOA”), hydrochloric acid (0.5 M), dest. water, 4 vials with lids, microliter pipette (IKA) or syringes with needles, ultrasonic bath, light source (e.g. POWLI023, 500W)

Procedure: To generate the catalytic solutions, the aqueous stock solutions are first prepared according to the concentrations given above. The titanium dioxide-water dispersion is additionally treated briefly in an ultrasonic bath until the TiO₂ nanoparticles are dispersed. The total of four vials are filled as follows:

- **Samples 1 and 2:** 3 mL water, 0.03 mL PAA solution, 0.3 mL PDADMAC solution, 0.3 mL TiO₂ dispersion, 1 mL TEOA solution, 1 mL Eosin Y solution.
- **Samples 3 and 4:** 3 mL water, 0.3 mL TiO₂ dispersion, 1 mL TEOA solution and 1 mL Eosin Y solution.

The pH value of all samples is adjusted to 9 with ~ 5 drops of hydrochloric acid. This is followed by treatment

of the samples in an ultrasonic bath for thirty minutes. Samples 1 and 3 are then placed in front of a light source for 45 minutes with the hydrogen sensor placed on top (see Figure 6), while samples 2 and 4 are kept in a dark environment.

Observations: After addition of the orange Eosin Y solution, the colorless samples turned to a pink, fluorescent tone. During exposure, the irradiated samples turn yellowish, whereas the unexposed samples retain their color (see Figure 4).



Figure 4. Various samples during irradiation with sunlight

Analysis: The proposed catalytic cycle for this experiment comprises several steps from irradiation to hydrogen evolution. In the first step, the photosensitive molecule (PS, here: Eosin Y) is brought into the excited state (PS*) through the irradiation with light [23]. The excited PS* then reacts with the sacrificial electron donor (TEOA), this process is called a reductive quenching and reduces the photosensitive molecule (PS⁻). Third, the reduced PS⁻ transfers the electron to the catalyst (Cat, here: TiO₂), which uses this electron to reduce Protons (H⁺).

As described above, the polymers disperse and immobilize the components and thus facilitate the electron transfers between the components (see Figure 5).

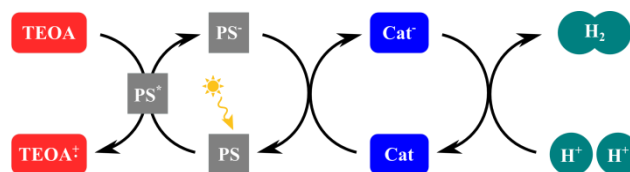


Figure 5. Simplified reaction mechanism from the irradiation of the photosystem to the hydrogen evolution reaction

4.2. Low-cost analytics in the classroom with LabPi

Since analysis by gas chromatography is only available to very few schools and student laboratories, we describe below one way in which this can be carried out inexpensively in school lessons. The digital low-cost measuring station LabPi [24] is used for this purpose, which is based on a Raspberry Pi minicomputer, custom made hardware and free software. A hydrogen sensor - here: Joy-IT Sen-MQ-8 - is connected. These or identical sensors from other manufacturers are widely available, the cost is about 3-4 € (Figure 6). With the adapter board from LabPi, the sensor is connected to the USB-C interface. Without the adapter board, the sensor can also be connected according to the circuit diagram provided here [25].



Figure 6. Low-cost hydrogen sensor (left); the sensitivity can be regulated via the potentiometer (colored in blue with gray screw); simplified hydrogen concentration measurement (right)

When the sensor is connected, the sensitive layer heats up to a constant operating temperature, indicated by a constant base line (here: 24 ppm). If gases are now directed onto the sensor surface, they will interact with the semiconductor sensor. Reducing gases, (such as hydrogen and ethanol) will result in an electron density increase in

the conductive band, while oxidizing gases (such as oxygen) will have the opposite effect. This results in a corresponding increase in voltage or decrease in voltage which is detected. The principle of ionosorption on nanostructured sensor surfaces and related examples and a construction manual for teaching are described in more detail in the literature [26].

In the given experiment, the samples are examined comparatively; due to the good fit of the sensor head to the vial opening, the error due to premature escape of hydrogen can be kept as small as possible. The conversion of the raw data of the sensor (resistance of the active sensing layer, given in Volt) into ppm is done on the basis of the sensor data sheet by a freely available script [27].

All experiments have been conducted multiple times. The combined results show an increasing hydrogen evolution over time in samples 1 and 3, while in samples 2 and 4 - which were not irradiated - no hydrogen above the reference value can be detected, as expected. The results are depicted in Figure 7.

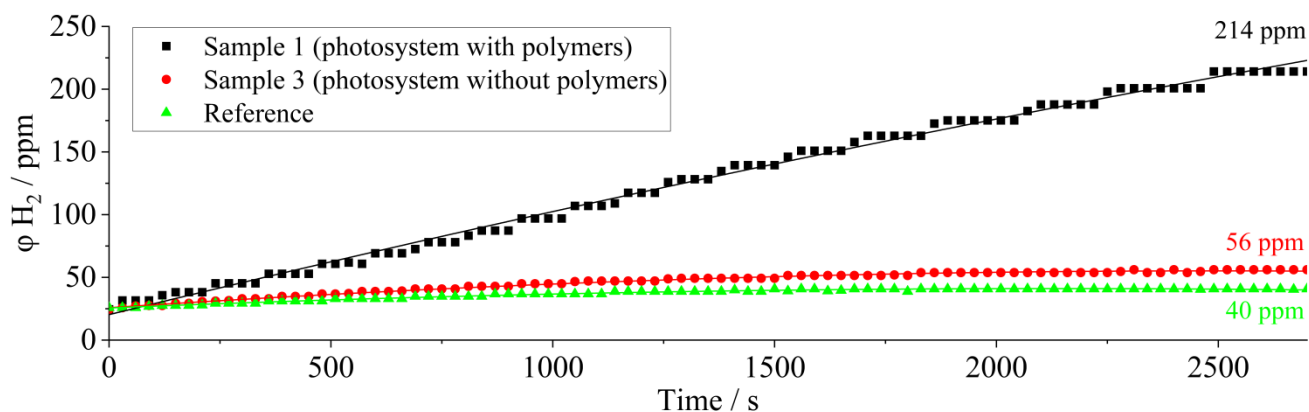


Figure 7. Measurement results of experiment 4.1: depicted are sample 1 (black), sample 3 (red) and the reference (blue). The complete data file can be found in the supporting information

The focal point of this analysis is the comparison between samples 1 and 3, where it is shown that the polymers result in a significant increase in the conversion rate. After 45 minutes, sample 1 shows a concentration of 214 ppm whereas sample 3 shows 56 ppm, which only slightly exceeds the references (samples 3 and 4) which are around 40 ppm.

On this basis, the role of the polymer matrix in the evaluation can be understood as a dispersing aid, which at the same time helps to mediate contact of the reactants. The formation of the interpolyelectrolyte complex between PAA and PDADMAC creates different (electrostatic) attachment points for catalyst and sensitizer. In consequence, both molecular species are tethered in close spatial proximity by the polymer matrix resulting in more efficient light-driven hydrogen evolution.

5. Conclusions and Outlook

In comparison with systems from chemical research, it must be noted that the amount of hydrogen produced is currently still 1-2 orders of magnitude behind. Nevertheless, the evolution of hydrogen could be

conducted in this experimental setup with simple methods in the school laboratory.

In further work, additional parameters are being optimized in order to optimize the conversion rate. These are, on the one hand, the addition of a suitable co-catalyst and, on the other hand, the use of an ethanol-water mixture as solvent, which acts as catalyst in the appropriate concentration. Furthermore, the concentrations of the reactants will be increased, which, in addition to the hoped-for increase in turnover, can at the same time further simplify the preparation of the samples. With cost-effective artificial light sources (*e.g.* Ultra Vitalux daylight lamp, approx. 30 €), the performance can be further increased and the reaction can also be carried out independently of the time of day and other environmental factors (*i.e.* season, angle of sunlight, etc.).

The presented experiment opens a first experimental access for pupils into a future generation of materials, which can generate sustainable hydrogen with sunlight by the interaction of a photosystem with a surrounding matrix. It thus offers an insight into a current field of research in chemistry and materials science and links to challenges facing society as a whole as well as to areas of interest to students. Hydrogen can be generated with sunlight and

subsequently detected using harmless chemicals and inexpensive materials.

Overall, the topic creates references to classical contents of chemistry teaching (*i.e.* catalysis, energy conversion, redox reactions, polymers) as well as basic concepts of chemistry (energy, structure-property) and areas of competence (specialist knowledge, acquisition of knowledge, evaluation). In addition, the connection of photochemistry with aspects of an Education for Sustainable Development, which has already been called for multiple times, is taken up synergistically [28].

The experiment is suitable for student laboratories and seminar papers with interested students. It represents a first step towards opening up this subject area for regular school teaching in the medium term. To this end, the experiments will be further optimized to increase the currently small amount of hydrogen. Subsequently, a teaching sequence for school laboratories will be developed and piloted together with teachers. The results of the evaluation will be incorporated into further optimization, so that in the medium term a transfer to regular chemistry teaching can take place. To this end, there will be accompanying teaching material as an open educational resource as well as a free set of materials for teachers in order to lower the barriers to their use in the classroom.

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Conflict of Interest

The authors Manuel Wejner and Timm Wilke have affiliations with the organization iTUBS mbH which distributes the LabPi System for educational institutions and training laboratories at cost price. No profits were generated.

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