

Solstice in STEM Education Cyclic Chemistry Driven by Light

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Abstract: This article presents an incredible simple version of the Photo-Blue-Bottle (PBB) experiment that works in sunlight. The fact that a considerable amount of green hydrogen is produced, as well as the interpretation of the reaction cycles taking place in spatially separated reaction sites, and the emerging equilibria make the experiment of high educational value with regard to the STEM education for sustainable development. The desired goal is a technosphere that works according to the model of the biosphere.

Keywords: biosphere, technosphere, cyclic chemistry, Photo-Blue-Bottle (PBB) experiment, chemical (thermodynamic) equilibrium, photosteady state, photochemical contents, chemistry curricula, STEM education, sustainable development education, Sustainable Development Goals SDGs

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1. Introduction

The climate crisis is pushing us to rethink our relationship with our "mother star", the Sun. We need to use sunlight in a more versatile and efficient way than in the past and today. "A solstice is forthcoming" [1] in science and technology.

In this context, *Henning Hopf*, the former president of the German Chemical Society stated in a recent interview: "The energy problem is not our most important problem; it is ultimately solvable because we have the sun. But the material problem is serious: everything that we are surrounded by comes from our own resources, from the earth's resources. ... We chemists need to think much more about this, Where is a division on 'Cyclic Chemistry'? High time to set one up." [2]

Actually, the singularity of our planet among all known cosmic objects is its **biosphere**, the sum of all living organisms. A key characteristic of the biosphere is the fact that it only works with carbon-containing compounds. The unique properties of carbon compounds make them particularly suitable for **cyclic chemistry** with a perfect balance of both energy and atoms. The biosphere is in itself sustainable, but only if we do not include *homo sapiens*. He is damaging the sustainability of life on earth by the technosphere he has established. Although anthropogenic impacts on the sustainability of the biosphere date back to ancient times in human history, they have been accelerating since the beginning of the industrial revolution at the end of the 18th century.

A technological "solstice" would correspond to the realization of the vision illustrated in [Figure 1](#).

According to this vision, energy storage systems such

as hydrogen, methane and other carbon compounds would also be produced from the raw materials water and carbon dioxide in the human-made **technosphere** in the same way as in the biosphere, and would be managed in material cycles.

At present, we are far away from this scenario. However, within the last 10 years, research in *photocatalytic hydrogen* production and *artificial photosynthesis* has become more intensively focused than in almost any other fields of research. A paper published in the scientific journal *Nature* in 2024 considers the large-scale, fossil-free production of hydrogen and carbon-containing fuels, as well as basic chemicals and polymers made from water, carbon dioxide, biomass and plastic waste to be practicable for the years after 2050 [3].

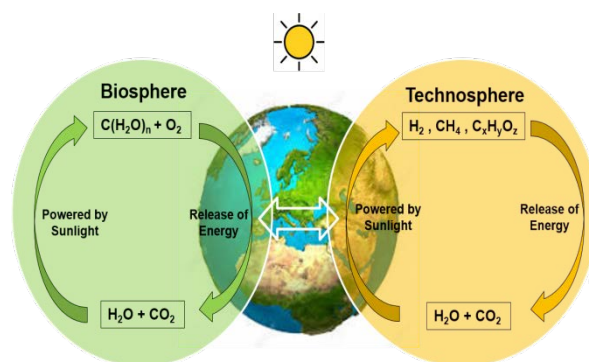


Figure 1. Model of a sustainable technosphere based on the principle of cyclic chemistry powered by solar light

So far, remarkable successes have already been achieved in researching various photocatalytic systems, for instance with covalent organic frameworks (COV) [4], graphite-phase carbon nitride (g-C₃N₄) [5] and with

chalcogenide-based semiconductors [6]. Different methods of solar reforming are discussed and tested, in particular photocatalytic, photo-electrochemical and photovoltaic-electrochemical methods [7]. Since the main aim is the photocatalytic production of green hydrogen, but since overall water splitting (OWS) is difficult to realize on the oxidation side (where water molecules would need to be oxidized to oxygen molecules and protons), *sacrificial donors* are used to increase the efficiency of hydrogen production [5,7,8,9]. If, in this way, an increase in the economic value of the used donors can be achieved simultaneously with the production of hydrogen, for example by oxidizing alcohols to aldehydes [9] or by reforming biomass and plastic waste [5], one would come closer to vision shown in Figure 1. The key research questions to be solved up to this point are mentioned in the articles cited here [3,4,5,6,7,8,9].

It should be emphasized that these are research topics opening up chances for Nobel prizes for our today's students. The best catalyst in this sense would be more light-driven processes in science education.

2. Experiment: Hydrogen Production Directly by Visible Light Irradiation

Not only the simplicity of the experimental setups shown in Figure 2, but also the following additional features make the PBB experiments recommendable for teaching purposes:

- All chemicals needed for the different versions of the Photo-Blue-Bottle PBB experiments, i.e. the components of the PBB-solution as well the reduction catalyst are commercially available (see below). They are non-toxic, harmless from the safety point of view, cost effective, and recyclable.
- Here are the Product Numbers for chemicals available by Merck/Sigma-Aldrich: ethylviologen (1,1'-Diethyl-4,4'-bipyridinium-dibromid) Product No. 384097; proflavine (Proflavin-hemisulfate (Salt) Product No. P2508; sacrificial donor: EDTA

(Ethylenediaminetetraacetic acid disodium salt dihydrate, Product No. E6635; 5%Pt@Al₂O₃, and (5% Nano-Platinum on alumina powder) Product No. 311324.

- The PBB-solution required for all versions of the PBB experiments is prepared simply by dissolving 1 g of EDTA, 561 mg of ethylviologene, and 12 mg of proflavine in 500 mL of distilled water. The solution should be stored in the dark in a glass flask.
- 7 - 10 mL of hydrogen can be obtained within 30 min starting from 50 mL of reaction mixture. This consists of 50 mL of a very diluted aqueous solution, and 50 mg of the solid catalyst suspended in the PBB-solution (see SEM-Photograph in Figure 3a).
- The hydrogen produced can be convincingly proofed and applied, e.g. by the hydrogen-oxygen explosion test, by the glow up on the 5%Pt@Al₂O₃ (5% Nano-Platinum on alumina) catalyst, or by powering a fuel cell car (in Figure 3b).

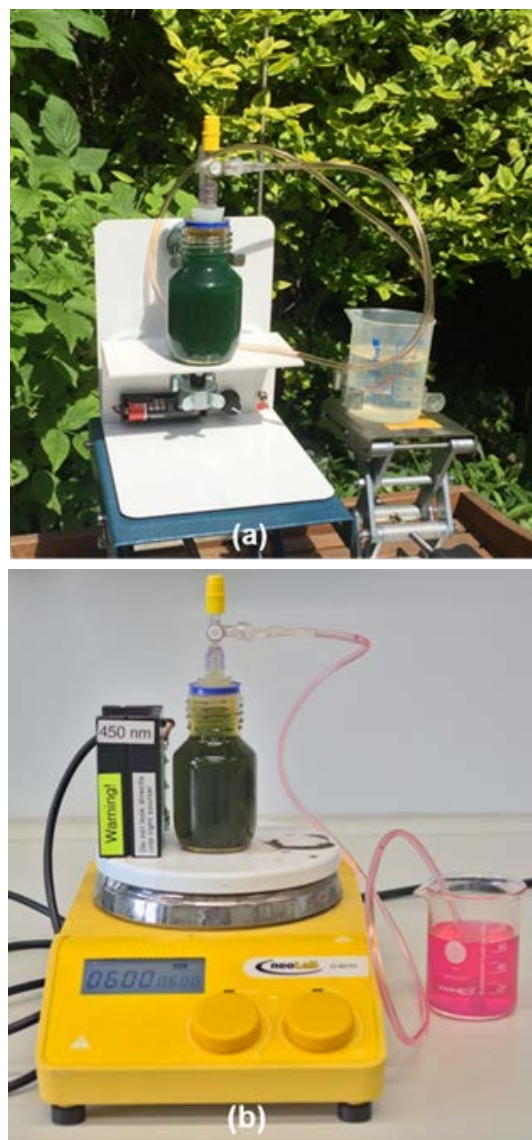
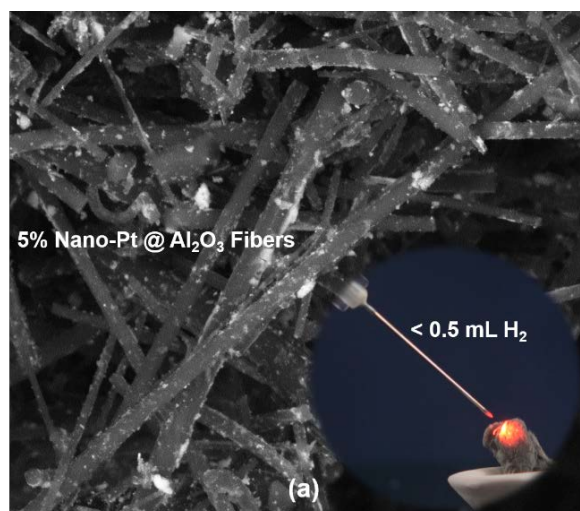


Figure 2. Two Setups for the photocatalytic production of hydrogen by irradiation with sunlight (a), and with blue light from LED (b); the reaction mixture is stirred magnetically, and the evolving hydrogen is pneumatically collected in a flexible tube of silicone



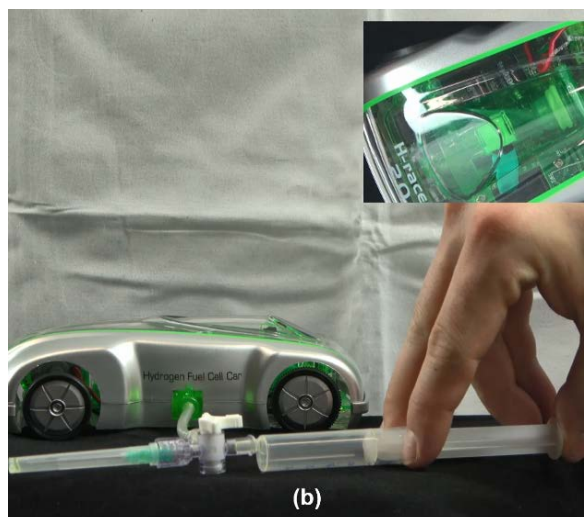


Figure 3. SEM-photograph of the catalyst with excerpt showing the detection of very small amounts of H_2 by the glow up (a), and fueling of the fuel cell toy car with approx. 5 mL of hydrogen (b)

These new versions of our well explored PBB experiment [10,11,12] are designed in such a way that the critical reactions are spatially separated according to the scheme in Figure 5.

In the *homogeneous phase*, the light-driven reduced form EV^+ of the redox mediator is generated in the PBB solution, and on the *heterogeneous phase* of the Pt-nanoparticles protons H^+ are reduced to molecular hydrogen H_2 . Simultaneously, the monocations EV^+ of the redox mediator get oxidized back to dications EV^{2+} . It should be noted that in the non-irradiated yellow PBB solution, the concentration of the *redox mediator* ethylviologen EV^{2+} is in the range of 10^{-3} mol/L, that of the *photocatalyst* proflavine PF^+ around 10^{-5} mol/L, and that of the *sacrificial donor* around 10^{-1} mol/L. These concentrations of the components and the corresponding redox potentials of the redox pairs involved ensure that the experiments work.

In the homogeneous part of the reaction system, the first step is the absorption of a light quantum ($\lambda = 450$ nm), resulting the electronic excitation in the proflavine $\text{PF}^+ \rightarrow \text{PF}^{*+}$. This leads to a dramatic change in the redox potential of the photocatalyst from $E^\circ(\text{PF}^+/\text{PF}^{2+}) = +1.1$ V to $E^\circ(\text{PF}^{*+}/\text{PF}^{2+}) = -0.6$ V. This difference is as big as that between a noble and an ignoble redox pair, for example $E^\circ(\text{Ag}/\text{Ag}^+) = +0.8$ V and $E^\circ(\text{Zn}/\text{Zn}^{2+}) = -0.76$ V). The electronically excited proflavine monocation can reduce an ethyl viologen dication from the PBB solution by photoelectron transfer: $\text{PF}^{*+} + \text{EV}^{2+} \rightarrow \text{PF}^{2+} + \text{EV}^+$.

During continuous irradiation, a constant concentration of monocations EV^+ is established in the blue PBB solution over time. They serve as an intermediate energy storage for the absorbed photons. Due to the buffering effect of excessive EDTA in the PBB solution, this is slightly acidic ($\text{pH} \approx 4.5$). The concentration and quantity of H^+ ions are therefore more than high enough to produce 10 mL of hydrogen. At $\text{pH} = 4.5$, the reduction potential for the formation and evolution of hydrogen is $E^\circ(\text{H}_2/2\text{H}^+) = -0.265$ V. The reduction of the H^+ ions to molecular hydrogen on the nano-platinum particles of the reduction catalyst occurs according to the equation $2\text{EV}^+ + 2\text{H}^+ \rightarrow \text{H}_2 + 2\text{EV}^{2+}$. As the redox potential of the ethylviologen

pair $\text{EV}^+/\text{EV}^{2+}$ is pH-independently $E^\circ(\text{EV}^+/\text{EV}^{2+}) = -0.449$ V, only molecular hydrogen is produced if no oxygen is available in or above the PBB solution. For this reason, all of the air is removed from the system in the experiment before the irradiation begins. The remaining oxygen from air that has not been completely removed and the oxygen dissolved in water is consumed after only a few minutes of irradiation.

One of the advantages of the experiments presented here is the use of light within the *visible range* of the solar spectrum to drive the production of hydrogen. It would therefore make sense to investigate the reactions in the PBB experiment in more detail (see Figure 4).



Figure 4. In direct sunlight and blue LED light, the yellow PBB solution turns blue (see reactions in the yellow box in Figure 5); however, there is no blue coloration when irradiated with red or green light

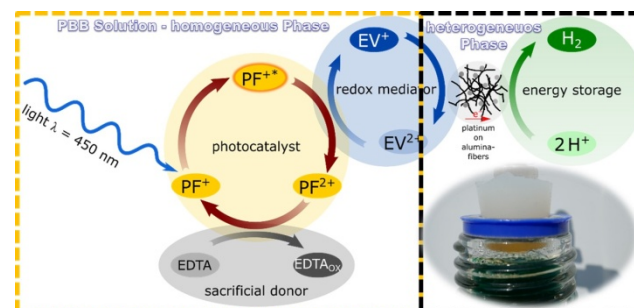


Figure 5. Coupled reactions in the homogeneous and the heterogeneous phase in experiments from Figure 2; the image section shows evolving hydrogen from the bottle neck of the 50 mL reaction flask

The main observation in the basic version of the PBB experiment shown in Figure 4 is an easy to repeat cycle of *phenomena* consisting of the blue coloration when the initially yellow PBB solution is irradiated and the re-coloration from blue to yellow when oxygen from the air is introduced into the blue solution, e.g. by simple shaking of the small glass vial. Micro versions in 5 mL screw cap vials show the color cycles yellow $\rightarrow$ blue $\rightarrow$ yellow more than 20 times when irradiated with sunlight or blue light and after that shaken. The color change from blue to

yellow upon shaking no longer occurs when the oxygen from the air above the solution has been exhausted.

The macro version in a 500 mL screw cap bottle placed on a module of 3 LED lamps provides fascinating images for show lectures [11]. The supplement at the end of this article provides some examples.

The abundance of phenomena provided by the different versions of the PBB experiment inspire further investigations not only by the experimenting schoolchildren, but also by researching students, and by teaching experts. The investigations may refer to the wavelengths of the active light in the PBB system, the influence of the amount of air above the solution on the number of cycles which can be realized, and the energy conversion and storage upon the reduction from EV^{2+} to EV^+ (i.e. during the blue coloration of the PBB solution) [12]. To mention just one example of how school students have further developed the PBB experiments: Three girls from a Bavarian high school, who used a flower extract of a plant (*Hypericum Perforatum*) as a photocatalyst and triethanolamine as a sacrificial donor in the PBB experiment, were very successful at a prominent scientific youth competition in Germany (Jugend Forscht) thanks to their creative approach to experimental work and their excellent results [13].

3. Unequal Equilibria: Chemical Equilibrium and Photosteady State

In line with the topic of this article and with reference to the central experiment of this article (Figure 2), the term of *equilibrium* needs to be scrutinized. Beyond chemistry, it belongs to the public domain in everyday language and also to the technical terminology of several natural, economic and social sciences. The tutorial-movie linked in the legend of Figure 6 introduces the topic of this section in a dialog between a scientific layperson and a young researcher using experiments. In the movie, the two protagonists explore the fundamental difference between *chemical (thermodynamic) equilibrium* and the *photosteady state* using the example of the photoactive molecular switch spiropyrane/merocyanine.

A **photosteady state**, is also established and maintained by light irradiation in the blue solution of the PBB system in the two experiments for photocatalytic H_2 production shown in Figure 2. In these cases, the concentration ratio between the blue monocation EV^+ and the colorless dication EV^{2+} is constant in the PBB solution during the entire period of the hydrogen production experiment and $c(EV^+)/c(EV^{2+}) > 1$. In contrast, there is a chemical (thermodynamic) equilibrium in the yellow starting solution. Here again, the concentration ratio of the two species is constant, but in this case $c(EV^+)/c(EV^{2+}) < 1$. The concentration of the dication EV^{2+} is far greater than that of the monocation EV^+ . In the photosteady state, the reduced species EV^+ of the redox mediator, which is continuously generated by light irradiation, predominates due to the coupled reactions shown in Figure 5.

However, in the photosteady state of the PBB system under continuous irradiation the amount of the reduced species EV^+ is oxidatively destroyed by release of

electrons to H^+ ions in the heterogeneous phase on the Pt nanoparticles with same rate as it is produced photocatalytically in the homogeneous phase. Consequently, we can say in terms of *reaction kinetics* that in a photosteady state the formation and decomposition of a photochemically generated species are in balance.

The equilibrium “position” of the photosteady state is generally different from that of the chemical (thermodynamic) equilibrium. Our interactive animations [14] can be used to get in-depth explanations of these two types of equilibria.

At this point, an extremely important photosteady state in nature should also be mentioned. The stratospheric oxygen-ozone cycle (the *Chapman cycle*) is essential for life on our planet because it converts the main part of harmful UV component of solar radiation into heat and thus removes it from the solar radiation reaching the earth's surface. In fact, *the whole biosphere can be considered as a photosteady state of our planet in solar radiation*.

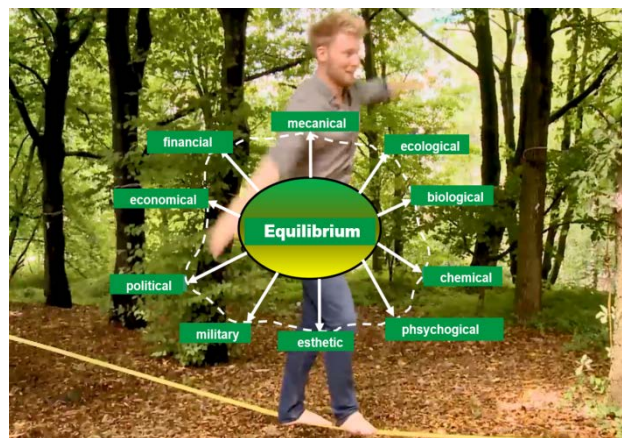


Figure 6. Screenshot from the tutorial-movie „Unequal Equilibria“, <https://chemiemitlicht.uni-wuppertal.de/en/movies-videos/photochromism-solvatochromism-photo-steady-state/unequal-equilibria/>

4. Our Mission: STEM Education with Sustainable Development Goals SDG

In order to realize the vision shown in the model from Figure 1, a priority mission in STEM Education would be the implementation of Sustainable Development Goals

(SDGs) according to the UNESCO Agenda 2030 into chemical and entire STEM education.

In a report from 2017, the German Conference of Ministers of Education and Cultural Affairs has formulated general principles for Sustainable Development Education (Bildung für Nachhaltige Entwicklung BNE), and the federal states have followed with concrete content and competence expectations for the subjects involved. However, the photoprocesses that are absolutely indispensable for sustainable development are not adequately represented neither in the ministerial principles nor in the guidelines of the federal states.

Therefore, a *paradigm-shift* from heat and electrochemistry to heat, electrochemistry and photochemistry must be achieved in the near future. In this

sense, chemistry didactics is much faster and more innovative than the educational bureaucracy. Didactically working groups at different universities, for example in Wuppertal, Münster, Potsdam, Tübingen, Karlsruhe, Göttingen and Oldenburg, are researching experimental approaches to *photochemical contents*, developing teaching/learning materials in print and digital formats, and proposing didactic integration into *chemistry curricula* [15,16,17,18] [19,20,21] [22,23].

Sustainability with light for life and technology is possible and achievable through *cyclic chemistry*, which operates in the technosphere according to the model of the biosphere. This technology is completely *fossil-free*, the *raw materials are water* (e.g. seawater, high-purity water as in electrolysis is not required) and CO_2 (e.g. extracted from the air). The products can be long-term energy stores in the form of *green hydrogen*, gaseous and liquid carbon-based *fuels* and valuable *basic chemicals*. These products can be used as fuels in flexible gas-fired power plants, which stabilize the electricity grids when there is a lack of wind and sun, or even to power vehicles in several mobility segments. Their combustion would not produce any increase of CO_2 in the atmosphere, so it would be a *climate-neutral combustion*. If that were the case, the combustion engine in cars would also be facing a renaissance.

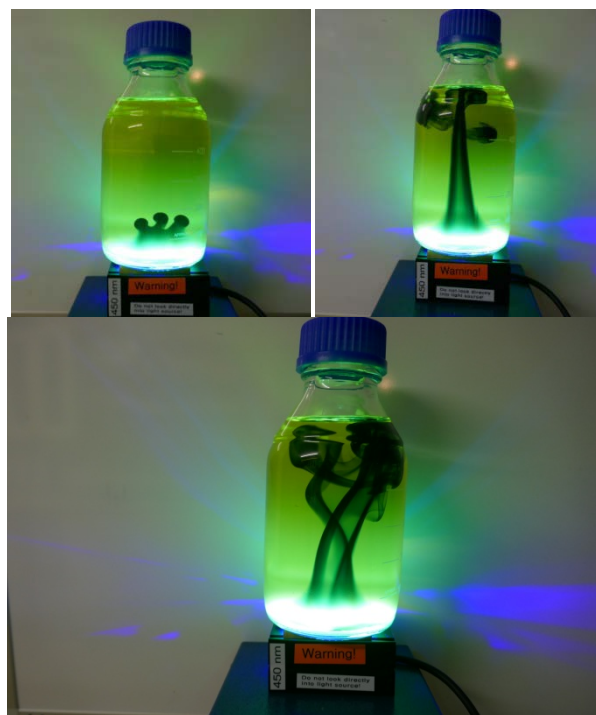
From a photochemist's point of view, the "solstice" in STEM education necessarily and primarily includes photocatalysis for the production of *green hydrogen* directly with sunlight, and the *artificial photosynthesis* of fuels (e.g. methane and liquid hydrocarbons) as well as valuable basic chemicals (e.g. alcohols and other oxygenated carbon compounds). School curricula must be future-oriented from a student's perspective, as they determine what the upcoming generation should learn and be able to do. When defining curriculum contents, guidelines with future relevance such as education for sustainable development must be addressed by including relevant contents in the *obligatory repertoire*. Accordingly, the appeal "More light for sustainable STEM teaching" is a categorical imperative in these times of multiple disruptions and transformations.

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Supplement

When the 500 mL bottle containing PBB solution is irradiated from below with 3 blue LEDs, mushroom-like blue figures initially form, then grow upwards and combine to form fantasy figures, e.g. dancing couples.



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