UNIVERSITY OF
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PRAGUE

# New trends in photo and electro catalysis

## Proceedings of the 12th Czech-Austrian workshop

Josef Krýsa
EDITOR

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## PREFACE

The Czech-Austrian workshops "New Trends in Photo and Electro Catalysis" represent a successful series of conferences organized jointly by the University of Chemical Technology (UCT) Prague and the Technical University of Vienna. The first Czech-Austrian workshop took place in November 2006 in Hnanice, Czech Republic. Other workshops were held annually or bi-annually due to the COVID pandemic.

The 12$^{th}$ Czech-Austrian workshop with the title "New Trends in Photo and Electro Catalysis" will take place from 1$^{st}$ to 3$^{rd}$ October 2025 in Hnanice, Czech Republic. Proceedings of the workshop contain abstracts of participant contributions from various university institutions and scientific research organizations from the Czech Republic, Austria, Slovakia and Slovenia.

The workshop aims to bring together leading academic scientists, researchers, postdoctoral fellows and PhD students to share their knowledge and research expertise and to discuss obtained results in the area of semiconductor photocatalysis and photoelectrochemistry including new semiconductor materials, batteries, fuel cells, water electrolysis and other electrochemical processes.

When selecting presenters, priority is given to PhD students, some of whom are attending the workshop for the first time, to allow them to gain experience in presenting their research results in an international forum. At the same time, there are many experienced researchers among workshop participants including supervisors of PhD students. The meeting thus allows to meet colleagues and discuss further possible collaborations. Finally, let us wish that all the participants of the annual conference will have a wonderful and fruitful time at the workshop.

Josef Krýsa and Guenter Fafilek

**Acknowledgement**

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# 12th Czech-Austrian workshop:
# New trends in photo and electro catalysis

CONFERENCE VENUE: HOTEL HAPPY STAR, HNANICE, CZECH REPUBLIC
(www.hotelhappystar.cz)
Date: ***1st October – 3rd October 2025***

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**Main Austrian partner:**
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Dr. Gregor Žerjav

# Programme

## Wednesday, 1st October 2025

**12.30 – 13.00** registration of participants
**13.00** Welcome talk (Josef Krýsa)

*Chair: Josef Krýsa*
**13.10** ***Š. Slapničar, G. Žerjav, A. Pintar***
Utilization of Plasmonic Effects for Visible-light-driven Photocatalytic Environmental Applications
**13.40** ***M. M. Ballari, M. Filip Edelmannová, R. Ricka, M. Reli, K. Kočí***
Photocatalytic $CO_2$ Reduction in $TiO_2$ Slurries: Kinetics and Carbon Species Equilibrium
**14.00** ***M. Baudys, J.Rusek, J. Krýsa***
Photocatalytic Reduction of $CO_2$ on $TiO_2$ Based Materials
**14.20** ***R. Ricka, B. Trindade Barrocas, M. F. Edelmannová, M. Reli, M. Łapinski, M. Conceição Oliveira, A. C. Marques, K. Kočí***
Mo-Modified $CeO_2$ and Microscafs® Composite as an Efficient Photocatalysts for $CO_2$ Photoreduction

**14.40 – 15.10** coffee break

*Chair: Albin Pintar*
**15.10** ***H. Krýsová, Z. Hubička, T. Imrich, M. Neumann-Spallart, J. Krýsa***
*In-situ* and *Ex-situ* Sn Doping of Hematite Photoelectrodes
**15.30** ***Z. Venkrbcová, Z. Hubička***
ECWR-Assisted Pulsed Reactive Sputtering for Deposition of Semiconducting Oxide Thin Films Suitable for Photocatalysis
**15.45** ***T. Imrich, M. Neumann-Spallart, H. Krýsová, H. Tarábková, J. Krýsa***
Extension of the (Photo)Electrochemical Working Range of $WO_3$ Electrodes to pH 8 by ALD Coverage with $TiO_2$
**16.05** ***T. Blecha, P. Dzik, M. Králová, M. Veselý***
Dual-phase $WO_3$ Films with Enhanced Macroporositythrough Biotemplating
**16.20** ***B. Radová, T. Imrich, Michael Neumann-Spallart, J. Krýsa***
$WO_3$ And $CuWO_4$ Photoanodes for Photoelectrochemical Applications
**16.35** ***J. Jablunka, N. Kotulková, M. Filip Edelmannová, B. Trindade Barrocas, K. Kočí***
Advanced $TiO_2$ and $CeO_2$-based Photocatalysts for $CO_2$ Reduction
**16.50** ***D. Varga, S. Pfaffel, J. Blaschke, H. Rabl-Wolff, D. Apaydın, D. Eder***
Bimetallic and Alloy Catalysts for Electrocatalytic Conversion of $CO_2$ to Formic Acid
**17.05** End of the session

**19.00** Dinner

## Thursday, 2nd October 2025

*Chair: Ladislav Kavan*
**8.30** ***P. Dzik, M. Veselý***
Structure-function Optimalization of Printed Photoelectrochemical Cells
**9.00** ***H. Krýsová, T. Imrich, H. Tarábková, P. Janda, J. Krýsa***
Impact of Variations in ALD Procedure on Nanomorphology, Protecting Properties and Chemical Stability of Thin $TiO_2$ Films
**9.20** ***J. Rusek, M. Neumann-Spallart, H. Krýsová, T. Ohno, J. Krýsa***
$CuBi_2O_4$ Photocathodes for Solar-driven Reduction Reactions
**9.40** ***J. Štěpánek, T. Bystron, Š. Paušová***
Two-step Synthesis and Characterization of $CuFeO_2$ Thin Layers for Photoelectrocatalytic Applications
**9.55** ***S. Pfaffel, N. Parak, D. Varga, H. Rabl-Wolff, S. Myakala, J. Blaschke, D. Apaydın, D. Eder***
Formic Acid Photoelectroreforming over Bismuth Vanadate – Proof of Concept
**10.10** ***D. Vojvoda, J. Hnát, K. Bouzek***
Electrochemical Deposition of Nickel on Porous Electrodes

**10.25** – **11.00** coffee break

*Chair: Kamila Kočí*
**11.00** ***M. Paidar, V. Masojídek, V. Šram***
Importance of Water Quality for Water Electrolysis
**11.20** ***J. Volák, K. Vydrželová, P. Hladík, Š. Paušová***
Development and Optimization of BiOX Photocatalysts for Environmental Pollutant Degradation
**11.40** ***Z. Huang, Z. Wang, H. Rabl, S. Naghdi, Q. Zhou, Y. Yu, D. Eder***
How Powerful is Ligand Engineering in Enhancing (Photo)Electrocatalytic Activity of Metal-organic Frameworks***?***
**12.00** ***A. Karafiátová, J. Rusek, M. Baudys, J. Krýsa***
Photocatalytic Oxidation of Organic Compounds in Air on Various $TiO_2$ Structures
**12.15** ***M. Králová, M. Veselá, M. Veselý, P. Dzik***
Resazurin Assay for Determining the Antimicrobial Activity of Photocatalytic Surfaces: Optimization and New Findings

**12.35** End of the session

**13.00** Lunch

**14.30 – 19.00** Walk to the Thaya National Park, individual discussions

**19.00** Dinner

## Friday, 3rd October 2025

**8.30 – 9.00 check out**

*Chair: Guenter Fafilek*
**9.00** ***L. Kavan***
Dual Ion (Zn/Li) Battery with Water-in-Salt Electrolyte: The Emerging Alternative of Li-ion
**9.30** ***J. Hnát, M. Plevová, K. Bouzek***
Development of Catalyst-coated Membrane for Alkaline Water Electrolysis
**9.50** ***M. Carda, S. Janda, D. Budáč, M. Paidar, L. Novák, K. Bouzek***
Optimizing Tape Casting and Sintering for Advanced Solid Oxide Cell Components
**10.10** ***D. Budáč, V. Miloš, M. Carda, M. Paidar, K. Bouzek***
Monte Carlo Model for Simulation of Porous Composite Ceramics
**10.30** ***P. Janda, H. Tarábková***
Surface "Ageing" – Hidden Process Deteriorating Interfacial Properties of Solid Electrodes

**10.50** – **11.25** coffee break

*Chair: Martin Paidar*
**11.25** ***G. Žerjav, A. Pintar***
Platinum on $TiO_2$: How the Schottky Barrier Influences Photocatalytic Pathways
**11.45** ***D. Dvoranová, M. Malček Šimunková, K. Lušpai, M. Férová, V. Slovák, V. Lazić***
Indirect Techniques of EPR Spectroscopy as an Effective Tool in Detection of Transient Paramagnetic Species
**12.05** ***A. Brüger, G. Fafilek, M. Neumann-Spallart***
Phase Changes in $WO_3$ Films upon Heating and Cooling
**12.25** ***X. Li, I. Venkrbcová, D. Lundin, M. Čada, Z. Hubička***
Plasma Diagnostics for Reactive HiPIMS Process Control of Vanadium Oxides
**12.45** ***S. Patakyová, M. Kubát, P. Dzik***
Hybrid Layers of Polymeric and Graphitic Carbon Nitride for Advanced Photoactive Coatings

**13.00** Final remarks (Josef Krýsa)

**13.15** Lunch

# Abstracts

# UTILIZATION OF PLASMONIC EFFECTS FOR VISIBLE-LIGHT-DRIVEN PHOTOCATALYTIC ENVIRONMENTAL APPLICATIONS

***Š. Slapničar, G. Žerjav, A. Pintar***

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This research focuses on heterogeneous photocatalysis for environmental applications, using visible light to activate photocatalysts. Titanium dioxide ($TiO_2$) is a key material due to its high photocatalytic activity, low toxicity and water stability. However, due to its wide band gap, its activation is limited to UV light [1,2]. To address this limitation, plasmonic metals (PM) such as Au, Ag and Pt were deposited on titanate nanorods (TNR) to enhance visible-light-driven photocatalysis by localized surface plasmon resonance (LSPR). In addition, an influence of different PM on the properties of TNR+PM photocatalysts was investigated.

TNR+PM catalysts were prepared using a wet impregnation method, where in-house prepared TNR were stirred in a PM precursor/ethanol solution for 2 h, followed by calcination at 300 °C in an atmosphere of 5% $H_2$/95% $N_2$ with a heating ramp of 150 °C/h [3,4]. The structural and optoelectronic properties of the materials were characterized using various techniques such as UV-Vis DR, solid-state PL, TCSPC, $N_2$ physisorption, SEM-EDXS, XRD and TEM. The photocatalytic performance of the materials under visible-light illumination was evaluated by measuring the generation of reactive oxygen species (ROS) and the degradation of bisphenol A (BPA).

SEM-EDXS analysis confirmed that the actual PM loading was approximately 1.0 wt.%, which is consistent with nominal values. The TNR+PM samples showed a uniform distribution of PM, with particle sizes varying according to metal type: Pt exhibited the smallest average particle size (1.5 nm), while Au had the largest (45 nm). These differences were influenced by the pH of the PM precursor solutions relative to the isoelectric point of TNR. The addition of PM to TNR resulted in an extended fluorescence lifetime, which improved the catalytic properties. Solid-state photoluminescence (PL) measurements revealed that TNR-Pt had the lowest charge carrier recombination rate among all tested materials. ROS generation tests using various water-soluble target molecules (e.g., DPPH, $ABTS^{\bullet+}$ and coumarin) confirmed that the photocatalysts were able to generate superoxide anion ($O_2^{\bullet-}$) and hydroxyl ($OH^{\bullet}$) radicals under visible light. Additionally, all TNR+PM catalysts tested were shown to generate ROS due to the LSPR effects of the plasmonic metals when illuminated at 550 nm and using coumarin dissolved in water as the target molecule. Among the catalysts, TNR-Au showed the lowest charge carrier generation and BPA degradation rate. In contrast, TNR-Pt with its smallest nanoparticles and the most suitable energy band distribution showed the highest ROS production and BPA degradation efficiency. These results indicate that $O_2^{\bullet-}$ radicals are the primary drivers of BPA oxidation and degradation on TNR+PM catalysts, with TNR-Pt outperforming the other catalysts due to its enhanced ROS generation and superior photocatalytic activity [5].

Recently, we have pioneered the synthesis of nanoflower-shaped Au photocatalysts (Au NFs) on $TiO_2$ nanorods (TNR) and systematically investigated their structural, optical and electronic properties [6]. Size- and shape-dependent LSPR effects were observed, which are reflected in the superior activity of the (Au NFs)/TNR photocatalyst for $H_2$-assisted $NO_2$ reduction at ambient conditions, compared to Au/TNR solids with spherical Au nanoparticles.

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# PHOTOCATALYTIC $CO_2$ REDUCTION IN $TiO_2$ SLURRIES: KINETICS AND CARBON SPECIES EQUILIBRIUM

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Nowadays, one of the main environmental challenges is clean energy production, as well as the control of $CO_2$ emissions from fossil fuel combustion [1]. Photocatalytic $CO_2$ reduction has emerged as an attractive technology to both lower atmospheric levels of this greenhouse gas and provide an alternative renewable fuel resource. However, the efficiency of this reaction is still low, and the identification of the optimal operating conditions remains the focus of ongoing research [2]. The dissolved $CO_2$ species play a crucial role in photocatalytic reduction. $CO_2$ exists in equilibrium with bicarbonate and carbonate, its concentration being governed by pH, and it has been shown to be the primary specie that can be photoreduced [3]. This study investigates the influence of the carbon species equilibrium on product yields in a $TiO_2$ suspension system under UV irradiation.

Experiments were conducted in a stirred batch photoreactor illuminated by an 8 W Hg lamp ($\lambda = 254$ nm) containing suspensions of $TiO_2$ (Aeroxide P25) at varying loadings (0.1-1g $L^{-1}$). The photocatalyst was suspended in NaOH or $NaHCO_3$ solutions with a concentration of 0.2 M. $CO_2$ or He was bubbled into the suspension before irradiation, ensuring saturation and air removal. The reactor was sealed at 120 kPa, and gas samples were analysed using gas chromatography with a barrier discharge ionization detector (BID). pH measurements were taken before and after saturation, and total dissolved inorganic carbon (DIC) was quantified via TOC (Total Organic Carbon) analysis.

The obtained results show that $NaHCO_3$ solutions saturated with $CO_2$ produce the highest yields of CO, $CH_4$, and $H_2$. Upon $CO_2$ saturation, both the $NaHCO_3$ and NaOH systems stabilize at pH 6.7, with the carbonaceous species equilibrium comprising approximately 38% dissolved $CO_2$ and 62% $NaHCO_3$. The enhanced yield correlates with higher DIC availability from both $NaHCO_3$ and $CO_2$ capture. In contrast, $NaHCO_3$ solutions saturated with He result in higher pH (7.9) and contain only 2% dissolved $CO_2$ together with lower DIC, leading to lower yields.

Based on these results, a kinetic model was proposed to correlate the equilibrium dissolved $CO_2$ concentration with the formation rates of by-products, also accounting for the dependence on the radiation field in the $TiO_2$ suspension. To determine the concentration of $CO_2$ in the different suspensions as a function of pH, the acid-base equilibrium of the carbonaceous species was considered in the carbon balance. A good agreement between the experimental data and the mathematical model predictions was observed.

In conclusion, enhancing the availability of dissolved inorganic carbon through $CO_2$-saturated $NaHCO_3$ solutions, together with the stabilization of carbon species at lower pH, increases the efficiency of photocatalytic $CO_2$ reduction.

**Acknowledgment**

This research was funded by the European Union under Research Excellence For REgion Sustainability and High-tech Industries (REFRESH) project No. CZ.10.03.01/00/22_003/0000048 via the Operational Programme Just Transition and supported within the project “Waste as an alternative source of energy”, reg. nr. CZ.02.01.01/00/23_021/0008590 under the Programme Johannes Amos Coenius. Experimental results were accomplished by using Large Research Infrastructure ENREGAT supported by the Ministry of Education, Youth and Sports of the Czech Republic under project No. LM2023056.

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# PHOTOCATALYTIC REDUCTION OF $CO_2$ ON $TiO_2$ BASED MATERIALS

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Semiconductor photocatalysis in gaseous phase is mainly associated with oxidation reactions where various types of contaminants such as traces of aldehydes [1], toluene[2] can be decomposed. On the other hand, in the presence of oxidizable compound (typically water [3]) which acts as electron donor photocatalysis could be also utilised in reduction processes [4]. This contribution deals with initial experiments of photocatalytic reduction of $CO_2$ in batch photoreactor according to the standard DIN 91457.

Experimental apparatus is illustrated in Fig. 1, it consists of photoreactor (Fig. 2.), humidified nitrogen purge system, $CO_2$ cylinder (50 ppm in $N_2$), manometer and GC septa for sampling. Detailed description of the photoreactor was reported recently [5]. At first, reactor and tubing was purged by nitrogen (relative humidity 50%, flowrate 1 l/min) to remove oxygen and to set humidity. Than the reactor is filled with $CO_2$ (concentration 50 ppm in $N_2$, pressure 1.750 mbar). As a photocatalytic material pellet of $TiO_2$ CG 300 (Precheza) optionally covered by graphitic carbon nitride (g-$C_3N_4$) and immobilised by Nafion dispersion was used. Samples of the reaction mixture were taken through GC septa (Hamilton syringe) and analysed using FID GC equipped with Jetanizer nozzle.

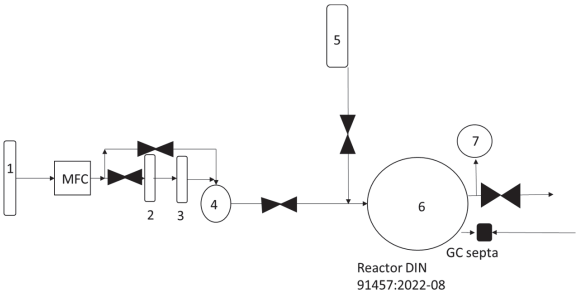


Fig 1: Experimental setup (1 nitrogen cylinder, 2 bubbling flask with water, 3 bubbling flask without water, 4 RH meter, 5 $CO_2$ cylinder (50 ppm in N2) 6 photoreactor according DIN 91457, 7 manometer)

Fig 2: Photoreactor according DIN 91457

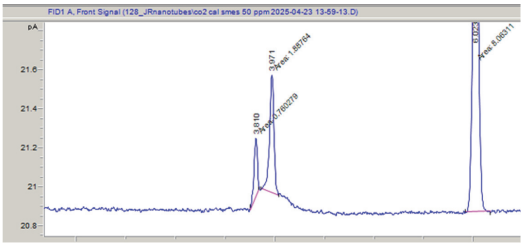


Fig 3: Typical chromatogram of the reaction mixture after 1 hour of UV irradiation (CO (3.8 min.), $CH_4$ (4.0 min.) and $CO_2$ (6.0 min)).

Fig. 3 shows the typical chromatographic signals for CO, $CH_4$, and $CO_2$. Under the given analysis conditions, the CO and $CH_4$ peaks are clearly separable. The molar amounts of the individual compounds is expressed as a function of UV irradiation time in Fig. 4). The reduction of one molecule of $CO_2$ to methane requires eight electrons (Eq. 1), while reduction to CO requires two electrons (Eq. 2)[6]. The experimental data in Fig. 4 show that the amount of $CH_4$ is negligible, and that CO is the preferential reduction product. Its concentration in the reaction mixture increases with irradiation time. In addition to the increase in CO, the concentration of $CO_2$ also increases with irradiation time. This increase is generally attributed to the oxidation of organic impurities present either in the photocatalyst pellet (e.g., Nafion, g-$C_3N_4$) or in air. In our previous work [7], it was demonstrated that UV irradiation of g-$C_3N_4$ leads to the formation of organic compounds (OC). A detailed analysis is provided to explain the origin of the observed $CO_2$.

$$CO_2 + 8H^+ + 8e^- \rightarrow CH_4 + 2H_2O \qquad \text{(eq. 1)}$$

$$CO_2 + 2H^+ + 2e^- \rightarrow CO + H_2O \qquad \text{(eq. 2)}$$

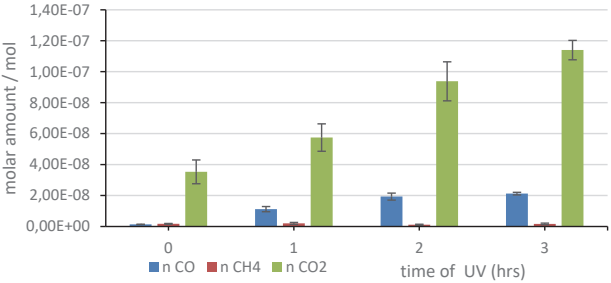


Fig 4: Molar amounts of CO, CH4 and CO2 as a function of irradiation time during photocatalytic experiment in the batch reactor, photocatalyst: TiO2/g-$C_3N$.


## Acknowledgment

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# Mo-MODIFIED $CeO_2$ AND MICROSCAFS® COMPOSITE AS AN EFFICIENT PHOTOCATALYSTS FOR $CO_2$ PHOTOREDUCTION

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Global warming and climate change, driven by rising $CO_2$ emissions in the atmosphere, represent the most critical environmental issues facing modern society. These problems are underscored by the World Meteorological Organization´s declaration of 2024 as the warmest year in recorded history. Based on these facts, there is an urgent need to improve the efficiency of current $CO_2$ utilization technologies or to find alternative strategies for processing $CO_2$ emissions [1, 2],

One of the most promising approaches is photocatalytic $CO_2$ reduction, which enables not only the reduction of $CO_2$ molecules, but also their conversion into valuable solar fuels. However, despite the great potential of this technology, its efficiency needs to be improved. Fortunately, material engineering offers an effective path toward this aim, as the design and preparation of suitable photocatalytic materials can significantly influence the overall performance of this reaction [3].

Within this research, a molybdenum (Mo)-modified $CeO_2$ photocatalyst was synthesized and investigated both in nanoparticle form and after immobilization on porous MICROSCAFS® microspheres. Furthermore, the prepared samples were comprehensively characterized to reveal their physico-chemical properties and finally tested for $CO_2$ photoreduction in a batch-stirred photoreactor operated under UV-C irradiation ($\lambda_{max}$ = 254 nm).

Our findings demonstrated the significant impact of successful Mo doping and introduction of oxygen vacancies in modified $CeO_2$ and MICROSCAFS® photocatalysts on enhancing photocatalytic performance in $CO_2$ photoreduction, relative to their unmodified counterparts. These modifications not only improved overall activity, but also significantly influenced product selectivity toward the formation of $H_2$, CO, and $CH_4$. Enhanced charge separation emerged as a key contributing to the observed improvements in photocatalytic efficiency.

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# *IN-SITU* AND *EX-SITU* Sn DOPING OF HEMATITE PHOTOELECTRODES

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$Fe_2O_3$ with a hematite structure is a promising n-type semiconductor with a suitable band gap (Eg ≈ 2.1 eV), which allows absorption of a significant portion of sunlight, resulting in a theoretical maximum energy conversion efficiency of 32 % under AM 1.5G illumination. It is also photoactive in the visible region [1]. Hematite is being intensively investigated as a non-toxic material for photoanodes for light-assisted water electrolysis. Its main drawbacks are: (i) low majority carrier mobility, (ii) limited stability in acidic solutions [2], and (iii) low minority carrier diffusion length. Low majority carrier mobility can be overcome by doping with elements such as Si [3], Ti [4], or Sn [5, 6], which results in an increase in conductivity.

In the case of Sn, doping can also be provided via thermal diffusion from a fluorine-doped tin oxide (FTO) substrate, which is used as a transparent conductive electrode. Therefore, it is important to distinguish between: i) in-situ doping, where an Sn precursor is added during deposition, and ii) ex-situ doping, where Sn diffuses from the FTO substrate during high-temperature annealing at temperatures exceeding 700 °C.

Two techniques for hematite deposition were used in the present work: aerosol pyrolysis (AP) and reactive high-power impulse magnetron sputtering (HiPIMS). Fluorine-doped tin oxide (FTO) on glass was used as the substrate. For the HiPIMS deposition, in situ doped hematite films were prepared by co-sputtering from an Fe target and an additional Sn target. In the case of aerosol pyrolysis, in situ doping was achieved by adding $SnCl_4$ to the AP precursor. Thin films prepared by both techniques were characterized using X-ray diffraction, SEM and profilometry, and their photoelectrochemical properties (photocurrents, IPCE) were compared. Emphasis was placed on the origin of Sn doping.

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# ECWR-ASSISTED PULSED REACTIVE SPUTTERING FOR DEPOSITION OF SEMICONDUCTING OXIDE THIN FILMS SUITABLE FOR PHOTOCATALYSIS

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Thin-film oxide semiconductor films with p-type conductivity, based on $Cu_2O$ and CuO [1], were deposited using reactive pulsed magnetron sputtering assisted by an RF ECWR plasma. An RF ECWR plasma is a special type of inductively coupled plasma placed in a static magnetic field. This field can be controlled to generate an electron cyclotron wave in the depositing plasma. Changing the magnetic field tunes the resonance of the electron cyclotron wave, resulting in maximum plasma density [2]. For the deposition process, a copper (Cu) magnetron target was used. The reactive gas used was a mixture of argon (Ar) and oxygen ($O_2$). Additionally, two pulsed magnetron systems with copper (Cu) and iron (Fe) targets, as well as an additional RF-ECWR plasma source, were employed for reactive co-sputtering to create $CuFe_xO_y$ thin films [3,4] with various stoichiometries and crystalline structures. The goal was to create a $CuFe_2O_4$ with spinel structure and p-type conductivity and an apparent photoresponse. Photoelectrochemical measurement and van der Pauw methods were used to evaluate the light and dark conductivity of the $Cu_2O$/CuO and $CuFe_xO_y$ films. XRD measurements were used to evaluate the thin film structure. ECWR plasma significantly influences parameters such as ion density, ion flux, and electron temperature. ECWR plasma also partially enhances the ionization of sputtered particles, as well as the dissociation, excitation, and ionization of the reactive gas [2]. These parameters strongly affect the reactive deposition process and the properties of the deposited oxide films. The process and plasma parameters were monitored using a planar RF probe installed at the substrate position [5]. The probe determined the time evolution of the electron temperature, ion density, and substrate ion flux depending on the flow of reactive oxygen gas and the RF power of the ECWR high-density source. There is a correlation between the measured plasma parameters and the thin film structure, as well as the semiconductor and optical properties.

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# EXTENSION OF THE (PHOTO)ELECTROCHEMICAL WORKING RANGE OF $WO_3$ ELECTRODES TO pH 8 BY ALD COVERAGE WITH $TiO_2$

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In our previous work [1], we developed $WO_3$ photoanodes with stable photocurrent-time behaviour in acidic media (perchloric acid). Such $WO_3$ photoanodes could have an application in the photoelectrochemical degradation of organic pollutants dissolved in water if they are stable in the neutral range (pH 6–8). However, $WO_3$ dissolves at pH around 6 and above [2]. A possible solution could be the application of a protective overlayer consisting of a chemically more resistant semiconductor ($TiO_2$ or $SnO_2$) with a wider bandgap acting as window material in the front side illumination mode. The present work aimed to check whether the above considerations are valid by experimentation with ALD coverage of $WO_3$ photoanodes with protective $TiO_2$ films of various thicknesses. Besides monitoring the photocurrents of as-obtained and covered photoanodes, it was the purpose of capping with $TiO_2$ to see whether in phosphate buffer (pH 8) stabilization against dissolution and photocorrosion can be achieved.

Table 1. Dissolution rates and Faradaic efficiencies of $WO_3$ photocorrosion in 0.1 M phosphate buffer (pH 8). The initial $WO_3$ layer thickness was 4300 nm.

| Coverage by $TiO_2$ ALD | Conditions | Dissolution rate /nm·h$^{-1}$ | Thickness decrease /nm | Charge /C | Faradaic efficiency |
|---|---|---|---|---|---|
| none | 4 h dark | 252 | 1008 | - | - |
| none | 4 h 0.9 V* light | - | 2112 | 3.04 | 0.27 |
| none | 1 h 0.9 V* light | - | 1314 | 2.25 | 0.35 |
| ------- | ------- | ------- | ------- | ------- | ------- |
| 20 nm | 4 h dark | 0.58 | 2.3 | - | - |
| 20 nm | 1 h 0.9 V* light | - | 28 | 0.95 | 0.021 |
| 50 nm | 1 h 0.9 V* light | - | 9 | 0.12 | - |
| 20 nm annealed$ | 1 h 0.9 V* light | - | 61 | 1.8 | 0.025 |
| 50 nm annealed$ | 1 h 0.9 V* light | - | 16 | 1.1 | 0.011 |

*potential *vs*. Ag/AgCl, electrode area 1 cm$^2$; $annealed at 500 °C for 1 h in air

In contrast to α-$Fe_2O_3$|$TiO_2$ photoanodes where photocurrents are strongly decreased with increasing thickness of ALD $TiO_2$ overlayers [3], in the case of $WO_3$|$TiO_2$ such capping layers led to much less decrease of photocurrents. This is ascribed to a more favourable relative position of valence band energies of the two semiconductors in the latter case, allowing easier passage of photogenerated holes from the absorber material to the capping layer. The coverage of $WO_3$ by protecting $TiO_2$ layers using ALD was shown to decrease the Faradaic efficiency of photocorrosion in pH 8 solutions by a factor of 20. Consequently, the presence of 20 nm thick capping layers extended the "lifetime" of photocurrents in pH 8 solutions from 3 to 25 hours. This is a proof of concept as to the use of capping layers for the suppression of photocorrosion.

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# DUAL-PHASE $WO_3$ FILMS WITH ENHANCED MACROPOROSITY THROUGH BIOTEMPLATING

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The aim of this work was to prepare layers of tungsten oxide using different deposition techniques from aqueous $WO_3$ composite to accommodate a greater need for environmentally conscious materials. Metal oxide composites and deposited thin films are usually prepared using complicating and often environmentally unfriendly methods [1]. To combat this, non-toxic materials and simple and inexpensive material deposition methods are crucial for further development. In addition, utilisation of renewable energy from sunlight is necessary for environmental photocatalysis-based applications. Tungsten oxide is a non-toxic semiconductor with lower bandgap of under 2.8 eV, which allows it to be photocatalytically active under not only ultraviolet, but also visible light irradiation [2]. This sets $WO_3$ apart from other widely used metal oxide semiconductors, such as titanium oxide, which shows photocatalytic activity only under ultraviolet irradiation.

Semiconductor photocatalysts are generally synthesized in two different ways: disintegration of crude material by mechanical processing (top–down), or assembly of atoms and molecules into desired nanoparticles (bottom–up). Each of these methods have their own advantages and disadvantages. Combining these two fundamentally different methods into one could bring out advantages and reduce disadvantages of each, while creating new properties. Such dual-phase material would be consisting of top–down prepared nanoparticles ("bricks") and bottom–up prepared amorphous matrix ("mortar") [3].

Such composite was prepared using ball-milled $WO_3$ nanoparticles and solution of amorphous $WO_3$ precursor. If this composite was deposited onto support and annealed at high temperatures, the thin layer would crack into smaller fragments, which were dense and connected only by the supporting material, often used glass with conductive doped tin oxide layer on top in contact with deposited semiconductor layer. An addition of small templating agent into the prepared composite before annealing could generate small pores after calcination, which might reduce the compartmentalisation into smaller fragments and increase the stability of prepared thin films. However, complete erasure of tearing inside the layer isn't optimal either, since it might seal the inside of thicker layers and reduce the specific surface area, reducing its photocatalytic activity. An addition of larger templating agent into the prepared composite could generate uniform porous structure, which should increase the specific surface area and photocatalytic activity with it.

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# $WO_3$ AND $CuWO_4$ PHOTOANODES FOR PHOTOELECTROCHEMICAL APPLICATIONS

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This work was aimed to prepare photoelectrochemically active photoanodes based on tungsten trioxide ($WO_3$) and copper tungstate ($CuWO_4$). Such materials are promising n-type semiconductors for various photoelectrochemical applications such as light-assisted water electrolysis [1], synthesis of fine chemicals [2] or water remediation [3]. Their relatively smaller band gaps (2.7 eV for $WO_3$ [4] and 2.2 eV for $CuWO_4$ [5]) compared to that of the widely studied $TiO_2$ (3.2 eV) [6] enables utilization of a larger part of the solar spectrum – 12 % of solar energy for $WO_3$ and 28 % for $CuWO_4$ instead of only 4 % for $TiO_2$ [7].

Both types of semiconductor layers were deposited by spray pyrolysis on FTO glass substrates, in the case of $WO_3$ using an aqueous solution of peroxotungstic acid; in the case of $CuWO_4$, using an equimolar mixture of peroxotungstic acid and $CuCl_2$. The deposition was carried out at temperatures ranging from 250 to 570 °C, followed by post-calcination in air at 550 °C for 2 hours to obtain the desired phase composition [8]. Mechanical stability and high photoelectrochemical response of $WO_3$ and $CuWO_4$ films was achieved through the optimisation of two parameters: (*i*) deposition temperature and (*ii*) layer thickness. Photoelectrochemical measurements were performed using a solar simulator equipped with an AM1.5 G filter and an irradiance of 1 SUN (100 mW/cm$^2$). Irradiation was applied from the front side.

Deposition temperatures above 350 °C were necessary to obtain the monoclinic crystalline phase. $WO_3$ films deposited at 250 °C were amorphous. Post-annealing at 550 °C for 2 h resulted in the formation of a monoclinic crystalline structure. The highest improvement in crystallinity, along with the highest photocurrent response, was found for $WO_3$ layers deposited at 250 °C and subsequently post-annealed at 550 °C. For an optimised layer thickness (~4.2 µm), the photocurrent density reached 1.9 mA/cm$^2$ in 0.1 M $HClO_4$ at 1.6 V *vs*. Ag/AgCl (1.31 mA/cm$^2$ at 1.23 V *vs*. RHE) under irradiation with a solar simulator (AM 1.5, 100 mW/cm$^2$). For $CuWO_4$ the highest photocurrent response was also achieved for deposition at 250 °C followed by post-annealing at 550 °C. For the optimal layer thickness (~3.9 µm), the photocurrent density was 0.54 mA/cm$^2$ at 1.45 V *vs*. Ag/AgCl in 0.1 M $Na_2SO_4$ (0.049 mA/cm$^2$ at 1.23 V *vs*. RHE).

The photocurrent stability of the optimised photoanodes was tested for 12 hours in various electrolytes. $WO_3$ photoanodes exhibited stable photocurrent values in all tested electrolytes at pH 1 (0.1 M $HClO_4$, $CH_4O_3S$, and $H_2SO_4$). A similar result was obtained for $CuWO_4$ photoanodes, but the tests were performed in near neutral pH (0.1 M $Na_2SO_4$).

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# ADVANCED $TiO_2$ AND $CeO_2$-BASED PHOTOCATALYSTS FOR $CO_2$ REDUCTION

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Human society currently faces a number of serious challenges, the resolution of which is important for maintaining global stability and sustainable development on Earth. Many threats to terrestrial and marine ecosystems are closely related to climate change and pollution caused by chemicals released as a result of anthropogenic activities. One group of these pollutants are greenhouse gases, such as carbon dioxide, which contribute significantly to environmental damage and climate change [1].

Heterogeneous photocatalytic reduction of $CO_2$ appears to be a promising technology for the future by utilizing sunlight, valuable products such as methane, carbon monoxide, methanol, and formic acid are generated, which can be reused. This method has the potential to reduce the impact of $CO_2$ emissions on the environment and to support ecological and sustainable development. Photocatalytic $CO_2$ reduction uses ultraviolet to visible light to create photoinduced electron-hole pairs that participate in redox reactions. Current research focuses on the synthesis of new photoactive materials and increasing the activity of photocatalysts to improve $CO_2$ conversion efficiency [2].

In this work, we focused on the photocatalytic reduction of $CO_2$ in the presence of $TiO_2$ and $CeO_2$-based photocatalysts. The tested materials included pure $TiO_2$, $CeO_2$, a $TiO_2/CeO_2$ composite, and $CeO_2$ nanoparticles surface-modified with diethylenetriamine (DETA/$CeO_2$) and ethylenediamine (EDA/$CeO_2$). $TiO_2$, $CeO_2$, $TiO_2/CeO_2$ photocatalysts were prepared by the hydrothermal method, while DETA/$CeO_2$, EDA/$CeO_2$ were prepared by post-synthetic surface modification of pure $CeO_2$. The photocatalytic experiments were carried out in a batch stainless steel photoreactor under UVC irradiation (Hg pen-ray, $\lambda = 254$ nm). The gaseous products were detected $CH_4$, CO, and $H_2$, which were analysed using gas chromatography with a barrier ionization discharge detector (GC/BID).

The results show that $TiO_2$ exhibited the highest selectivity towards $CO_2$ photoreduction products, primarily $CH_4$ and CO. In contrast, $CeO_2$ and especially its surface-modified forms (DETA/$CeO_2$ and EDA/$CeO_2$) showed higher activity for photocatalytic water splitting, favouring $H_2$ production. The $TiO_2/CeO_2$ composite photocatalyst proved effective in maintaining photocurrent stability and promoting efficient charge carrier separation. This work provides insight into the $CO_2$ reduction behaviour of different types of photocatalysts and confirms their potential for future applications, especially if these systems are optimised for operation under solar irradiation.

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# BIMETALLIC AND ALLOY CATALYSTS FOR ELECTROCATALYTIC CONVERSION OF $CO_2$ TO FORMIC ACID

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Rising atmospheric carbon dioxide ($CO_2$) from human activities, mainly fossil fuel use, deforestation, and agriculture, drives global warming, extreme weather, rising sea levels, and ocean acidification. Reducing emissions is essential, but converting carbon dioxide ($CO_2$) into useful products offers a complementary solution. One promising route is the electrochemical reduction of carbon dioxide ($CO_2$) to formic acid (HCOOH), a versatile liquid organic acid with high energy density that can serve as a renewable energy carrier, industrial feedstock, and indirect hydrogen source. Unlike hydrogen gas ($H_2$), formic acid is stable, safe to transport, and compatible with renewable-powered production[1,2].

This project focuses on using p-block metals (Sn, In, Bi) as catalysts for carbon dioxide ($CO_2$) reduction to formic acid (HCOOH). Due to the high overpotential for hydrogen ($H_2$) evolution reaction of these metals, they are considered as efficient catalyst to reduce carbon dioxide ($CO_2$) [3]. Two primary pathways are proposed: one through COOH* intermediates and another via OCHO* intermediates[4]. However, single-metal catalysts often require large overpotentials and may produce competing products such as carbon monoxide (CO), methane ($CH_4$), or methanol ($CH_3OH$).

To address this, alloy catalysts have emerged as a powerful strategy. By combining these aforementioned metals, alloys can fine-tune binding energies and stabilize critical intermediates, especially OCHO*, leading to higher efficiency and selectivity. Bi- and trimetallic systems provide additional flexibility, enabling better intermediate stabilization and selectivity[4,5]. This research therefore investigates how alloy composition and the influence of mixed oxide surfaces influence carbon dioxide ($CO_2$) reduction performance, with the goal of advancing selective and sustainable formic acid production.

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# STRUCTURE-FUNCTION OPTIMALIZATION OF PRINTED PHOTOELECTROCHEMICAL CELLS

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A photoelectrochemical cell is a device where at least one working electrode consists of a semiconductor supported on an electrically conductive substrate. The photocatalytic activity of immobilized semiconductor photocatalysts can be enhanced by applying an external electrical bias, which improves electron–hole separation and increases the quantum yield of redox reactions at the electrodes.[1] This enhancement requires the photocatalyst to be deposited on a conducting substrate. In n-type semiconductors, the working electrode functions as a photoanode with a positive bias, typically comprising a conducting base coated with a semiconducting metal oxide like titanium dioxide. The counter electrode's material must offer adequate electrical conductivity and corrosion resistance.

The photocurrent produced in an irradiated cell depends on factors such as electrolyte composition, irradiance, electrode materials, and cell design. These cells can be used for diverse applications, including sensing, remediation, and energy harvesting.

Material printing is an emerging microfabrication technique suitable for producing planar layered devices, such as photoelectrochemical cells[2]. It involves sequentially layering patterned functional materials using modified traditional printing methods. Among these, inkjet printing holds a prominent position due to its versatility. Despite constraints like viscosity and particle size, it is ideal for lab-scale prototype development since it eliminates the need for hardware printing forms. Patterns designed digitally can be printed directly, and the process is easily scalable to industrial levels using large-scale inkjet printers.

This contribution will provide a summarizing overview of various designs of photoelectrochemical cells [3-5] and demonstrate the benefits of material printing for their rapid prototyping, upscaling and possibly mass production of such cells. We will show that once the material printing approach is capable of direct patterning of insulating, semiconducting and conducting layers, we gain access to some very interesting direct additive fabrication pathways yielding fully printed cells suitable for various applications such as sensing, remediation, and energy harvesting.

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# IMPACT OF VARIATIONS IN ALD PROCEDURE ON NANOMORPHOLOGY, PROTECTING PROPERTIES AND CHEMICAL STABILITY OF THIN $TiO_2$ FILMS

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Surface coverage by thin films is of great technological importance, because it improves mechanical, optical, and electrochemical properties of solid surfaces. We have shown, that atomic layer deposition (ALD) of titanium dioxide protects the semiconducting electrode against corrosion and photocorrosion [1–3].

The present contribution deals with thin $TiO_2$ films deposited on FTO and $Si/SiO_2$ substrates by ALD with different deposition temperatures, 150 °C (LT-ALD $TiO_2$) and 250 °C (HT-ALD $TiO_2$) focusing on the influence of deposition conditions on the nanomorphology (AFM), phase composition (XRD), chemical stability, protective and photoelectrochemical performance. ALD deposition parameters strongly affect $TiO_2$ film properties. While both types of as-grown ALD $TiO_2$ were amorphous, subsequent annealing at 500 °C /1 hour led to the formation of anatase crystalline structure only in the case of LT-ALD $TiO_2$. HT-ALD procedure formed non-stoichiometric titanium oxide, with presence of low-valence Ti. Its annealing decreased doping level, but did not induce crystallization. The exposure to 0.1 M $HClO_4$ for 72 h led to the dissolution of $TiO_2$ and the formation of pinholes in $TiO_2$ film with depths matching film thickness. In alkaline buffer (pH 8), films remained stable. Barrier properties of $TiO_2$ films were evaluated via electrochemical reaction[4] of redox couple $[Fe(CN)_6]^{3-/4-}$. LT-ALD $TiO_2$ layers (8–50 nm) effectively blocked redox reaction, whereas such evaluation method was not applicable for HT-ALD films due to presence of self-doped sites. As-grown HT-ALD $TiO_2$/FTO showed higher (4 times) photoelectrochemical conversion efficiency than LT-ALD $TiO_2$/FTO, arising from $Ti^{3+}$ self-doping.

**Acknowledgment**

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# $CuBi_2O_4$ PHOTOCATHODES FOR SOLAR-DRIVEN REDUCTION REACTIONS

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The development of p-type oxide semiconductor photoelectrodes is gaining increasing attention as they may play an important role in sustainable energy conversion, particularly photoelectrochemical (PEC) water reduction [1, 2]. Among various candidates, $CuBi_2O_4$ (CBO) has been considered as a promising material for solar-driven reduction reactions (e.g. green hydrogen generation or hydrogen peroxide ($H_2O_2$) production) due to its narrow band gap (~1.8-1.9 eV) [1, 3] and favourable photocurrent onset potentials around 1.1 V *vs.* RHE [4, 5].

In this work, thin films of CBO were synthesized by spin coating on FTO glass and subsequent annealing to achieve uniform, crystalline films of 450 and 800 nm thickness. Structural and morphological characterization was performed by XRD, SEM, and EDS, while optical properties were evaluated by UV-Vis spectroscopy. Photoelectrochemical properties were studied in an aqueous electrolyte (0.1 M $Na_2SO_4$) under simulated solar illumination (1.5 AM, 100 $mW \cdot cm^{-2}$). The effect of various oxygen concentrations (saturation with $O_2$, air, $N_2$) and the presence of $H_2O_2$ and methyl viologen ($MV^{2+}$) on the photoelectrochemical response was investigated.

A band gap of 1.8 eV was determined from IPCE (incident photon-to-current efficiency) measurements. The position of the valence band was estimated from the open circuit potential under illumination to lie at 0.515 V *vs.* Ag/AgCl at pH 6 (1.08 V *vs.* RHE), which could be considered close to the flat band potential ($E_{FB}$). The chemical stability in acidic and alkaline pH was evaluated. In 1 M $H_2SO_4$ complete dissolution within 10 seconds occurred, whereas, in contrast, the electrodes remained stable after immersion in 1 M NaOH for 24 hours.

Photocurrents were stable under $O_2$ or air saturation for at least 24 h, whereas $N_2$-saturated conditions led to rapid degradation (noticeable change of the color of CBO photocathodes from brown to black) due to self-reduction. $H_2O_2$ served as an efficient electron acceptor, enabling stabilization of photocurrents even under $N_2$ bubbling, while $MV^{2+}$ resulted in much lower photocurrents and electrode instability. Oxygen reduction led to $H_2O_2$ formation with a Faradaic efficiency of around 20 %, indicating a dominant four-electron reduction pathway of oxygen to water. The obtained results show the potential of $CuBi_2O_4$ for solar-driven reduction reactions and its application in sustainable energy conversion and highlight the necessity of suitable electron acceptors to maintain long-term stability.

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# TWO-STEP SYNTHESIS AND CHARACTERIZATION OF $CuFeO_2$ THIN LAYERS FOR PHOTOELECTROCATALYTIC APPLICATIONS

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The main trends in the field of energy pertain to its production without the utilization of fossil fuels and its efficient storage. A potential collective solution lies in harnessing hydrogen as an environmentally friendly energy carrier, produced through electrolysis from renewable energy sources. [1] On the other hand, the process of generating electrical energy and subsequently utilizing it for water electrolysis incurs losses throughout the entire conversion process. Consequently, there is a prospect of direct photoelectrolysis of water into hydrogen and oxygen using semiconductors capable of harnessing solar radiation and inducing water splitting, akin to the mechanism observed in plants [2]. Hydrogen produced via solar-assisted water electrolysis can be utilized as a storage medium for renewable sources. The usage of copper-iron oxide is possibility, since $CuFeO_2$ (a delafossite) [3] naturally display p-type conductivity. Preparing phase pure delafossite on conductive FTO glass substrate is a challenging process since most of the syntheses are performed at high temperatures, which leads to the decrease of the conductivity of the substrate itself. Copper ferrite, due to its attractive properties, like narrow indirect bandgap in the range of 1.54−1.95 eV, belongs to the group of possible photocathode materials. [4]

This study investigates the utilization of the spray pyrolysis method to deposit thin films of mixed iron and copper oxides from aqueous solutions of nitrates onto fused silica or conductive FTO glass (F-doped tin oxide on borosilicate glass) substrates. Following deposition, thermal treatment was conducted to achieve the delafossite phase, $CuFeO_2$. The annealing process was optimized to produce phase pure crystalline delafossite on a conductive substrate and we report the successful preparation of $CuFeO_2$ on FTO through vacuum annealing at 700 °C. To compare influence of used conditions on delafossite films performance, the photoelectrochemical properties of the resulting films were evaluated using linear voltammetry, amperometry and electrochemical impedance spectroscopy in $O_2$-saturated electrolyte solutions. The optimized layers exhibited photocurrents of around −0.2 mA/cm$^2$ at −0.35 V vs. Ag/AgCl. After 30 min, the photocurrent dropped to −0.09 mA/cm$^2$ and remain stable for following 7 h.

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# FORMIC ACID PHOTOELECTROREFORMING OVER BISMUTH VANADATE – PROOF OF CONCEPT

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Hydrogen storage materials are a key factor in implementation of a sustainable hydrogen economy.[1] Among many options, liquid organic hydrogen storage (LOHC) materials are of high interest due to their easy handling and implementation to existing infrastructure. A possible candidate for LOHC is formic acid (FA), a low toxicity, water miscible compound that can also be found in nature.[2] Furthermore, a key advantage is the possibility of implementing a carbon neutral cycle by coupling carbon dioxide ($CO_2$) reduction to FA with selective FA oxidation to $CO_2$.[3]

For the oxidative part of aforementioned cycle, most research up to now has focused on FA photoreforming over titanium dioxide ($TiO_2$)[4], a UV absorbing semiconductor, but little effort has been made to implement a semiconductor with absorption in the visible light range, such as bismuth vanadate ($BiVO_4$), which recently has gained attention due to its capabilities for oxygen evolution reaction.[5] This contribution focuses on utilisation of $BiVO_4$ as a photocatalyst for FA oxidation under applied external potential.

Based on a published synthesis method for $BiVO_4$ electrodes [6] we examine the behaviour of the material for the desired application, done by coupling classic (photo-)electrochemical examination techniques with both gas and liquid phase analysis methods (GC, NMR) for examination of the photoelectroreforming performance and state-of-the-art material analysis methods (XRD, XPS, SEM) for electrode characterisation.

What was thought to be a straight forward proof of concept, that can be answered with either yes or no, turned out to be a journey full of new insights and pitfalls.

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# ELECTROCHEMICAL DEPOSITION OF NICKEL ON POROUS ELECTRODES

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Improving the performance of alkaline water electrolysis is crucial for advancing sustainable energy. It can be achieved, for example, by improvement in activity of the electrodes. Nickel (Ni) represents a cost-effective and catalytically active electrode material for both electrode reactions, *i.e.* the hydrogen (HER) and oxygen evolution reaction (OER). Because the electrode activity is dependent on the number of the active centres, it is beneficial to use 3D electrodes with enhanced surface. Ni foams, owing to their 3D porous structure, are thus commonly used in zero-gap alkaline water electrolyser configurations. Modifications of the electrodes, such as galvanostatic deposition can further increase the specific surface area of the electrodes, thereby enhancing catalytic activity. Modified 3D electrodes with increased specific surface can be directly employed in zero-gap assemblies or further modified for example by deposition of another electroactive substance.

Electrodeposition represents a simple, cost-effective, and highly controllable technique capable of coating external surfaces as well as the internal volume of 3D structures, making it particularly suitable for modifying Ni foams. The composition of the electrolyte plays a crucial role in determining the properties of the deposited layer. Among the different electrodeposition approaches, galvanostatic deposition has advantage of straightforward control over the amount of deposited Ni. It can be performed either in continuous or pulse mode. The latter promotes rougher surface, which is desirable for increasing the specific surface area of the electrode.

In this work, Ni was deposited under galvanostatic pulses from different electrolyte solutions to assess their influence on electrochemical performance. Sacrificed Ni anode was always used. The effects of the amount of deposited Ni and the type of galvanostatic pulse on HER and OER activity were examined. Scanning electron microscopy was used to analyze the morphology of the deposited layers. Electrochemical characterisation was done by polarization curve measurement in 0.1 mol·dm$^{-3}$ KOH at 30 °C in three-electrode arrangement. Polarisation curve measurements of HER and OER were followed by Tafel analysis to determine kinetic parameters (overpotentials at 10 mA cm$^{-2}$, Tafel slope and exchange current density). Electrochemical impedance spectroscopy was conducted to gain further insights into the electrochemical properties of the modified electrodes.

The results demonstrate that Ni deposition can significantly enhance the activity of Ni foam electrodes. The choice of deposition solution plays a key role, with nickel sulphate proving most effective among solutions tested. Furthermore, the amount of deposited Ni strongly affects performance. An optimal amount of deposited Ni is observed, which improves both HER and OER activity compared to bare foams. However, higher amounts of deposited Ni lead to decrease for OER activity, likely due to the formation of large surface structures that hinder gas bubble release and block active sites.

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# IMPORTANCE OF WATER QUALITY FOR WATER ELECTROLYSIS

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The quality of demineralized water plays a pivotal role in determining the performance, efficiency, and longevity of water electrolysers. Electrolysis demands water that is virtually free of impurities, as contaminants can clog porous electrodes and separators, degrade system efficiency, and increase maintenance frequency and costs. Maintaining high water purity helps ensure stable operation and minimizes the risk of component degradation. However, the specific purity requirements differ depending on the type of electrolyser in use—whether it's an alkaline system, a proton exchange membrane (PEM) electrolyser, or a high-temperature solid oxide electrolyser. Each technology has distinct tolerances and operational sensitivities to water quality, making tailored water treatment strategies essential for optimal performance.

Integrating renewable energy sources like solar and wind introduces variability in water electrolysis, leading to intermittent operation. This affects water demand and reduces the efficiency of purification systems. Additionally, different raw water sources impose varying requirements on demineralization technologies. Customizing water treatment technology to match specific electrolyser types can reduce costs while maintaining performance and durability.

Mechanical Vapor Compression Desalination (MVCD) is well-suited for unattended, intermittent operation. Combined with final purification, it enables the production of ultrapure water from high-conductivity sources such as seawater or brackish water. Waste heat from the electrolyser can be efficiently used in a zero liquid discharge (ZLD) step, ensuring only solid waste remains.

Energy and mass balance simulations confirmed the feasibility of water demineralization with ZLD. Laboratory tests combining a PEM electrolyser and ZLD system were conducted to estimate actual energy requirements.

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# DEVELOPMENT AND OPTIMIZATION OF BiOX PHOTOCATALYSTS FOR ENVIRONMENTAL POLLUTANT DEGRADATION

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Energy and environmental challenges are two significant barriers to the sustainable development of human society. Air and water pollutants are released not only from industrial activities but also from hospitals and households, contributing to the accumulation of persistent pollutants such as pharmaceuticals, pesticides, polycyclic aromatic hydrocarbons, and dyes in water supplies designated for drinking. Photocatalytic processes offer a viable solution for the removal of these pollutants. These processes rely on the excitation of a semiconductor by photons with energy equal to or greater than its bandgap, creating charge carriers that facilitate redox reactions [1]. Key to their effectiveness is designing photocatalysts that are chemically stable, non-toxic, and capable of maximizing the use of photogenerated charge carriers. Various photocatalysts, oxides, or non-oxides, such as $TiO_2$, ZnO, BiOBr, BiOI, and g-$C_3N_4$, have been studied for photocatalytic applications. Among these, BiOX compounds (where X = Cl, Br, I) represent promising materials belonging to a class of layered substances suitable for photocatalytic energy conversion and environmental remediation, drawing extensive research attention [2-4].

This study focuses on the development and testing of BiOX (where X = Cl, Br, I) photocatalysts, recognized for their potential in photocatalytic energy conversion and environmental remediation. BiOX compounds were prepared using various methods: solvolysis, hydrolysis, ultrasonicated hydrolysis, solvothermal, and hydrothermal synthesis. The catalysts were characterized through techniques such as XRD, SEM, BET, and XRF, and their activities were evaluated under UV and Vis light irradiation by determination of the degradation of different model pollutants such as monuron, 4-chlorophenol, Orange II, naproxen and sulfathiazol.

Key factors like precursor concentration, purity, and pre-treatment through stirring were found to significantly influence the formation of crystalline structures, particularly those favouring growth in the (001) plane, which enhanced activity [5]. The most effective BiOCl, prepared via hydrothermal synthesis, removed 27% of monuron in 4 h. The solvothermal method yielded the most active BiOI sample with 8% degradation within the same timeframe, although impurities were present. Solvolysis methods were generally ineffective in producing high-activity BiOX due to low yields and impurities. The most promising results were obtained with BiOBr, especially when synthesized via ultrasonicated hydrolysis, achieving 85% degradation of monuron in 4 h, though still less effective than $TiO_2$, which completed degradation in 2.5 h. While in the case of Orange II, more than 90% was removed using BiOBr and about 70% using $TiO_2$ in 2 h.

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# HOW POWERFUL IS LIGAND ENGINEERING IN ENHANCING (PHOTO)ELECTROCATALYTIC ACTIVITY OF METAL-ORGANIC FRAMEWORKS?

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In this Workshop, I plan to report on three research works I led [1-3], focusing on ligand engineering strategies and their effects on modifying Zeolitic imidazolate frameworks (ZIF). These strategies include the mixed-ligand and the selective ligand removal, applied in photoelectrocatalysis.

First, I introduce the mixed-ligand strategy to enhance the activity and stability of (photo)electrocatalytically active MOFs [1]. Four mixed-ligand variants of Co-ZIF were synthesized, enabling a detailed analysis of the influence of secondary ligands on the reconstruction of ZIF frameworks during the electrocatalytic oxygen evolution reaction (OER). Some secondary ligands preserved the fundamental framework while inducing surface reconstruction to form an in-situ cobalt (oxy)hydroxide layer. This contrasts sharply with the complete reconstruction observed in single-ligand ZIFs. The cobalt (oxy)hydroxide layer, along with its interfacial synergistic effects, enhanced conductivity and catalytic performance while also improving (photo)electrochemical stability.

Further, I continue to introduce how the ligand removal strategy to design hierarchically porous and defect ZIFs [2]. This novel method involves synthesizing mixed-ligand ZIFs with varying ratios of two ligands, followed by controlled thermal treatments to selectively remove the thermolabile secondary ligand. By incorporating open metal sites (OMS) into Zn-ZIF, specifically high-valence HO−Zn−$N_2$ active sites evolved during the reaction, an exceptionally low overpotential of 0.41 V was achieved, enabling a sustained current density of 1.0 A $cm^{-2}$ with 120-hour stability [3]. Moreover, this sample exhibited a 1.5-fold enhancement in current density under visible light, whereas pristine Zn-ZIF showed no such improvement, highlighting the potential of ligand-engineering strategies for photoelectrocatalysis.

These successful cases clearly demonstrate that ligand engineering has a powerful effect for designing advanced functional frameworks with enhanced (photo)electrocatalytic activity, selectivity, and durability for energy applications.

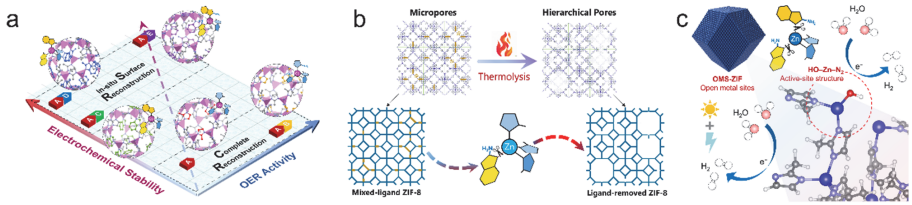


Figure: Schematic diagram of the mixed-ligand strategy (a) [1], selective ligand removal strategy (b) [2] and open metal sites (b) [3] for the ZIF frameworks.

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# PHOTOCATALYTIC OXIDATION OF ORGANIC COMPOUNDS IN AIR ON VARIOUS $TiO_2$ STRUCTURES

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This contribution deals with photocatalytic oxidation of volatile organic compounds (VOC) in air on $TiO_2$ nanostructures. At first photocatalytic degradation rate of acetaldehyde on $TiO_2$ nanotubes (TNT) according to ISO 22197-2 was investigated. This method is based on oxidative degradation of 5 ppm of acetaldehyde in stream of air in flow through reactor under standard conditions (flow rate 1 $dm^3$/min, relative humidity 50% and UV light intensity 1 mW/$cm^2$). Photocatalytic oxidation takes place on the surface of the $TiO_2$ based layer, where absorbed VOC are oxidized by $\cdot OH$ and $\cdot O_2^-$ radicals forming $H_2O$ and $CO_2$.TNT layers were prepared by anodic oxidation of titanium sheet, achieving thickness from 2.3 to 13.2 μm. After calcination at 450°C, the nanotubes exhibit an anatase $TiO_2$ structure as was proved by XRD analysis.

The study revealed that photocatalytic activity increases with increasing TNT thickness, peaking at 4 μm, where over 80% of UV radiation was absorbed [1]. Another studied parameter was the gas flow rate. The degradation rate increases with increasing flow rate (up to 5 l $min^{-1}$), further increase in the flow rate had no significant influence on the degradation rate and the system reaches its performance plateau. In addition to flowrate the effect of UV intensity was also investigated. Typically, the degradation rate is proportional to UV intensity; as photon flux increases, more reactive species are generated. However, beyond a certain threshold, all active sites on the photocatalyst surface become saturated. As a result, the relationship between degradation rate and UV light intensity becomes sublinear – following approximately a square root dependence [2]. At the lower flow rate of 1 $dm^3$/min (as prescribed by ISO standards), degradation rate increases linearly with UV light intensity up to 1 mW/$cm^2$, while at the higher flow rate of 5 $dm^3$/min, degradation rate increases linearly with UV light intensity up to 2 mW/$cm^2$.

In practical application of photocatalytic air treatment, it is important to reach reasonable degradation rate also for higher volumes of treated air and to oxidise present organic compounds (typically acetaldehyde) without forming of significant by-products. For this reason, attention was also payed to the investigation of photocatalytic properties of foam ceramics covered with $TiO_2$,. Such material seems to be applicable in air purifier thanks to its high surface area. Samples with dimension suitable for ISO photoreactor (10 x 5x 1.5 cm) were spray coated with commercial $TiO_2$ suspension containing 40% of photoactive $TiO_2$. Two samples of foam ceramics where placed in ISO reactor in such a way that the testing gas passed through their inner surface. The results of degradation rate expressed in molar amount of removed acetaldehyde related to geometric surface area and time (μmol/$cm^2$/h) show that degradation rate increases with increasing flowrate up to 6 $dm^3$/min. Further increase in flow rate has no influence on degradation rate peaking its maximum about 0.7 μmol/hour/$cm^2$.

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# RESAZURIN ASSAY FOR DETERMINING THE ANTIMICROBIAL ACTIVITY OF PHOTOCATALYTIC SURFACES: OPTIMIZATION AND NEW FINDINGS

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The aim of this work is to present the resazurin method as a tool for investigating the antimicrobial activity of photocatalytic surfaces and to share the latest findings of our work. The resazurin assay could one day be a highly effective alternative to the standard tests (according to ISO 27447:2019, [1]) currently in use which carry a number of disadvantages. The main disadvantages of existing methods for evaluating the antimicrobial properties of surfaces include their time-consuming nature, as determining viable bacteria through cultivation and colony counting usually takes longer than 48 hours. In addition, the recovery rate of the test is usually very low (only about 20–50 %), which results in considerable inaccuracies. These procedures also generate a large amount of plastic waste due to single-use laboratory equipment. A further limitation is that testing can only be carried out on very small surfaces and exclusively under laboratory conditions. All these shortcomings together highlight the need to develop a new, more straightforward methodology that will be in accordance with internationally recognized standards for evaluating antimicrobial effects and will eliminate all the disadvantages mentioned above.

The methodology is based on generally accepted principles for evaluating antimicrobial effects enshrined in international standards [2]. The principle of the new method consists in applying and anchoring a bacterial suspension on a hydrogel carrier and transferring the cells together with the carrier directly to the surface under investigation, whereby five different situations are examined and compared: (1) the number of surviving cells on the irradiated photocatalytic surface, (2) and on the reference, (3) the number of surviving cells on the non-irradiated photocatalytic surface (4) and on the reference. The fifth situation (5) is determining the initial amount of cells that was actually applied to the hydrogel carrier. The number of surviving cells is evaluated using the resazurin test, in which blue resazurin is reduced to pink resorufin, which fluoresces under green light [3]. This is based on the fact that the time required to achieve maximum resorufin fluorescence is inversely proportional to the number of surviving cells [4].

The proposed methodology is a suitable tool for studying the antimicrobial properties of various photocatalytic and photoactive surfaces, ranging from non-porous and porous materials to various fibrous materials.

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# DUAL ION (Zn/Li) BATTERY WITH WATER-IN-SALT ELECTROLYTE: THE EMERGING ALTERNATIVE OF Li-ION

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Aqueous dual-ion (Zn/Li) batteries offer a compelling balance of safety, cost, and sustainability, positioning themselves among promising alternatives to conventional Li-ion batteries [1]. By coupling the abundance and intrinsic safety of the zinc/water interface with the high energy density of lithium oxides or phosphates, these systems embody a hybrid strategy with significant potential. During operation, $Zn^{2+}$ and $Li^{+}$ ions simultaneously participate in charge storage: zinc dissolves to $Zn^{2+}$ at the negative electrode while $Li^{+}$ is extracted from the cathode during discharge, with the process reversed upon charging.

The inherent drawbacks of aqueous electrolytes, i.e. hydrogen/oxygen evolution, corrosion, and Zn dendrite formation, can be largely mitigated using water-in-salt electrolytes (WiSE) [2]. In these solutions, the mass ratio of salt to water exceeds unity. At the extreme end, molten hydrates can be formed, in which the molar ratio of salt to water can drop below three. Zinc chloride, one of the most soluble inorganic salts ever, is of particular relevance in this context. ($ZnCl_2 \cdot 2.33H_2O$ melts above 10 °C).

The paper will present the first systematic study of electrochemical materials, including substrates, active electrode compounds, and electrolyte components, relevant to Zn/Li dual-ion batteries employing $ZnCl_2$-based WiSE. Among the investigated substrates, titanium offers the widest electrochemical stability window. By contrast, carbon, commonly used as an additive and substrate, shows instability at potentials above ~2.2 V vs. $Zn^{2+}/Zn$, raising concerns about its suitability for high-voltage electrode materials such as $LiMnPO_4$. On the other hand, carbon-coated $LiFePO_4$ (olivine) is a highly stable positive electrode material in WiSE, exhibiting both good charge capacity and robust cycling performance. Its formal potential shifts positively with increasing $Li^{+}$ concentration in the electrolyte.

Our study also highlights the often-overlooked impact of electrolyte impurities. Trace amounts of $Mn^{2+}$, commonly present in commercial-grade $ZnCl_2$, can mimic the electrochemical signatures of $LiMnPO_4$, potentially leading to misinterpretation of results [3]. Conversely, the intentional addition of $Mn^{2+}$ to WiSE gives rise to a new battery chemistry reminiscent of redox-flow systems. In this case, $Mn^{2+}/Mn^{3+}/MnO_2/MnO_4^{-}$ species participate in charge storage at carbon electrodes. Interestingly, WiSE containing $AuCl_4^{-}$ displays nearly identical electrochemical behavior.

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# DEVELOPMENT OF CATALYST-COATED MEMBRANE FOR ALKALINE WATER ELECTROLYSIS

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Deposition of the catalyst directly onto the surface of an anion-selective polymer membrane, forming thus a catalyst-coated membrane, represents a significant step forward in the development of alkaline membrane water electrolysis. Catalyst-coated membrane approach enables a substantial reduction in the concentration of circulating liquid electrolyte, thereby increasing the flexibility and safety of the technology.

In alkaline water electrolysis, state-of-the-art approach involves depositing the catalyst layer onto the surface of an electron conductor (e.g. metal or carbon). In this configuration, all catalyst particles have good electronic contact, but ionic contact in diluted circulating solutions is limited to the points, where catalyst particles directly touch anion-selective polymer membrane. Ionic contact in the catalyst layer thus must be ensured by circulating electrolyte of sufficiently high conductivity/concentration. When the catalyst layer is deposited directly on the membrane surface and fixed there by anion-selective polymer binder, the ionic contact within the catalyst layer is ensured independently on the conductivity of the circulating electrolyte. The performance of catalyst-coated membrane under alkaline membrane water electrolysis is determined by the properties of the deposited catalyst layers. Key parameters influencing these properties include catalyst load, polymer binder content, catalyst layer thickness, porosity and catalyst layer conductivity.

In this work, we investigated different approaches to preparing catalyst-coated membrane for alkaline membrane water electrolysis.

Initially, the catalyst-coated membrane approach was compared with catalyst layer deposition on the electronically conductive substrate. In addition, the effect of the polymer binder content was studied. Optimization of catalyst layer properties included the addition of carbon or nickel particles to enhance the electronic conductivity within the catalyst layer and the selection of solvent for catalyst ink preparation to control polymer binder affinity to the catalyst particles.

All catalyst layers were deposited using a computer controlled sonicated dispersion deposition technique. Morphology of the catalyst layers was investigated by scanning electron microscopy. Electrochemical properties of the catalyst layer were ex-situ investigated on conductivity and in-situ under alkaline membrane water electrolysis conditions. Different concentrations of circulation electrolytes were used at 50 °C to compare performance of various catalyst-coated membranes. Characterisation was performed by load curve and stability at constant current density measurement. The electrochemical impedance spectroscopy measurement was employed to obtain deeper insights into the studied systems.

Results show that catalyst-coated membrane approach can reduce catalyst load (from 10 mg $cm^{-2}$ down to 2.5 mg $cm^{-2}$) while maintaining performance. Moreover, the amount of polymer binder in the catalyst layer significantly affects both the performance and stability. Electronic conductivity of the catalyst layer was identified as the bottleneck of the catalyst-coated approach. Addition of electronically conductive phase and/or using different solvents for catalyst ink preparation can improve the electronic conductivity.

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# OPTIMIZING TAPE CASTING AND SINTERING FOR ADVANCED SOLID OXIDE CELL COMPONENTS

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Solid oxide cells (SOCs) offer an efficient, carbon-free solution for high-temperature energy conversion, enabling reversible operation between steam electrolysis and fuel cell modes [1]. Their performance relies heavily on the properties of the ceramic materials used [2]. The electrolyte—typically yttria-stabilized zirconia (YSZ)—must be gas-tight, mechanically robust, and chemically stable at temperatures up to 1000 °C. Electrodes are commonly based on composites such as lanthanum strontium manganite (LSM) or nickel with YSZ, used for the oxygen and fuel electrodes, respectively. These electrodes must exhibit high electrochemical activity, thermomechanical compatibility with the electrolyte, and long-term operational stability.

While YSZ provides sufficient ionic conductivity, its thickness often limits performance by contributing to ohmic losses. Reducing electrolyte thickness is an effective strategy but introduces fabrication challenges, especially when scaling up the active area. In this work, we focus on the preparation of self-supporting YSZ electrolytes thinner than 300 µm using tape casting. While tape casting is more complex to optimize, it provides more reliable shaping of thin electrolyte layers than conventional powder pressing. Combined with optimized sintering, tape casting yields thin, dense, and planar electrolytes comparable to commercial wafers, demonstrating its potential for high-performance SOC applications.

Sintering is a crucial step not only for densifying YSZ electrolytes but also for shaping the functional microstructure of composite electrodes such as LSM:YSZ [3]. Using in situ SEM with a heating stage, we directly observed the sintering behavior of this system. At 900 °C, LSM particles began to coalesce, forming porous networks essential for gas transport but mechanically fragile. Densification at 1150 °C led to a robust structure with good mixed conductivity. Even minor changes in sintering conditions significantly affected porosity and performance, highlighting the need for precise thermal control across all SOC components [4].

This work demonstrates that reliable fabrication of advanced SOC half-cells requires coordinated control of both shaping and thermal treatment steps. Optimized tape casting yields thin, scalable ceramic layers, while controlled sintering ensures the required microstructure and performance of both electrolytes and electrodes. By combining these approaches, we bridge the gap between laboratory-scale experimentation and pilot-scale production, contributing to the broader implementation of solid oxide technologies for sustainable energy systems.

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# MONTE CARLO MODEL FOR SIMULATION OF POROUS COMPOSITE CERAMICS

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Porous composite materials are used in a large variety of energy applications concerning both fuel cells and batteries. Electrode components based on composites often consist of electron-conducive, ion-conductive and void domains. In the case of oxygen electrode used in the solid oxide cells, these domains accommodate electrons, oxygen anions and gaseous oxygen, respectively. Electrocatalytic performance of such electrode is governed by the amount of active sites – three phase boundary – a simultaneous contact of the three domains [1]. Composite electrode design lies in balancing transport properties of the individual species and the amount of active sites.

While the advantage of composite electrodes is undeniable, their development is usually reliant on trial and error. It is due to the limited ability of predictive methods being time-consuming and usually requiring unavailable input parameters. A simple Monte Carlo-based model is proposed and validated in this work enabling for more efficient development of new composite materials.

The mathematical model was developed based on simple particle packing-building principles [2,3]. Based on user-defined phase composition, the model fills an empty 3D computational domain in a random manner by three types of voxels: electrode material, electrolyte material, and void domain. Subsequently, an equivalent circuit network is generated based on the filled domain – each material is substituted with resistor of corresponding electrical resistance, while a parallel combination of resistor and capacitor is added to each electrolyte-electrode interface representing a simple electrochemical reaction. Based on the 3D network, the model calculates its total conductivity.

Validation was carried out using widely-utilized composite of lanthanum strontium manganite (LSM) and yttria-stabilized zirconia (YSZ). Porous LSM-YSZ samples of different ratios between the solid phases and porosities were fabricated. Their total electrical conductivity was determined by electrochemical impedance spectroscopy (EIS) at 400–800 °C. The model predictions showed excellent agreement in composite material conductivity with experimental data for porosities up to 55% [2]. In the case of higher porosity, void phase was no more randomly distributed and formed larger cavities. The model enables generation of topologic representations of highly porous samples experimental samples [3] by including a geometrical parameter describing the cavity formation. This parameter is fitted to match experimental values of electrical conductivity. Resulting generated structure contains information similar to that provided by FIB-SEM analysis, while demanding shorter experimental time and significantly cheaper equipment.

The proposed model showed reliable and fast operation. Its use will lead to exploration of new composite materials. Due to the simplistic definition, the model is suitable even for material characterized by different flow quantities, e.g. heat transfer.

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# SURFACE "AGEING" – HIDDEN PROCESS DETERIORATING INTERFACIAL PROPERTIES OF SOLID ELECTRODES

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Interfacial properties of solid electrodes in liquid electrolytes influence efficiency of all electrode processes. Factors like electrode wetting, i.e. the quality of interfacial contact between the electrode and the electrolyte solution, is often neglected, though it is essential for efficient performance of mass and charge transfer processes, which are the fundamentals of each photo/electrochemical and electrocatalytic reaction.

So-called surface "ageing" taking place upon exposure of the electrode surface to ambient atmosphere (air) was found to be responsible for significant change of solid-liquid interface composition, manifested by increasing contact angle of water (CAW), explained by decreasing wettability (raised hydrophobicity) of examined surface. This hydrophobization was first observed at HOPG as a CAW difference between freshly peeled and "aged" basal plane. It led to surprising conclusion of intrinsic hydrophilicity of basal plane HOPG [1], while its common hydrophobic properties are caused by "ageing" ascribed to coverage by hydrocarbons-based airborne contaminants.

So-far unclear, slower reaction kinetics found on "aged" surfaces [3,4] compared to fresh ones can be explained by our discovery of surface gaseous nanodomains formation on "aged" basal plane HOPG immersed in aqueous media [2] and their absence on freshly peeled HOPG. It provides plausible explanation of kinetics retardation by existence of gaseous nanodomains, which can occupy up to 80% of immersed "aged" surface, which implies, that only 20% of electrode surface remains in contact with electrolyte solution, available for electrochemical reaction. Our concept of blocking water-immersed aged surfaces by gaseous nanodomains apparently is valid also for other surfaces as it was recently confirmed [5] on aged $Si/SiO_2$ wafer, which repetitive "ageing"- hydrophilization cycles led to consistent appearance/disappearance of gaseous nanobubbles.

This also implies, that the hydrophilization pre-treatment of solid surfaces prior to use in aqueous and hydrophilic media may improve the kinetics of interfacial processes.

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# PLATINUM ON $TiO_2$: HOW THE SCHOTTKY BARRIER INFLUENCES PHOTOCATALYTIC PATHWAYS

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Noble metals such as Au, Ag and Pt exhibit plasmonic effects that enable them to drive chemical reactions in wide bandgap semiconductors such as titanium dioxide ($TiO_2$) under visible-light irradiation [1-4]. In this work, $TiO_2$+Pt catalysts with Pt content ranging from 0.5 to 2 wt.% were prepared by wet impregnation synthesis (samples TP, TP+0.5% Pt, TP+1% Pt and TP+2% Pt) [1]. The study focused on how Pt loading alters the optical, electronic and catalytic behaviour of the materials, with particular attention to the height of the Schottky barrier (SB) at the Pt-$TiO_2$ junction, which was determined by X-ray photoelectron spectroscopy (XPS). Furthermore, we investigated how Pt loading on anatase $TiO_2$ controls the generation of reactive oxygen species (ROS) under visible light and thus the degradation of bisphenol A (BPA) in water. ROS were quantified using two selective probes: i) reduction of $ABTS^{\bullet+}$ (tracking of photogenerated electrons/$O_2^{\bullet-}$), and ii) hydroxylation of coumarin to 7-hydroxycoumarin (tracking generation of $^{\bullet}OH$ radicals).

The bare TP showed no significant ROS generation rates under visible-light illumination, while in the TP+Pt samples, $ABTS^{\bullet+}$ conversion increased with Pt loading and 7-HOCU formation decreased, indicating a shift from $^{\bullet}OH$-dominated to electron/$O_2^{\bullet-}$-dominated pathways with increasing metal loading. Mechanistically, the localized surface plasmon resonance (LSPR) of Pt under visible-light illumination generates "hot electrons" that are injected into $TiO_2$ and "hot holes" that oxidize surface species. Decreasing the Schottky barrier height (SBH) with increasing Pt content (from 0.42 eV at 0.5 wt.% Pt to 0.16 eV at 2 wt.% Pt) accelerates electron transfer and favours $ABTS^{\bullet+}$ reduction/oxygen activation, while higher SBH retains electrons in $TiO_2$ and supports the formation of $^{\bullet}OH$ radicals. BPA degradation was performed in a slurry batch reactor ($c(BPA)_0$=10 mg $L^{-1}$; $c_{cat}$=125 mg $L^{-1}$; $V_{reactor}$=250 mL; purged with air @ 45 L $h^{-1}$; $T_{reactor}$=20 °C; lamp=150 W halogen; UV cut off filter $\lambda$>410 nm). None of the materials showed adsorption of BPA in the 30-minute dark phase. When the light was switched on, we found that the addition of Pt significantly increased the catalytic activity compared to pure $TiO_2$, with the sample TP+2% Pt showing the fastest BPA degradation rate among the analyzed materials. True mineralization (determined by TOC and CHNS analyses) increased with Pt content in the TP+Pt samples, consistent with greater BPA turnover. The best performing catalyst TP+2% Pt maintained its performance over five cycles with ~1% loss, highlighting its robustness.

Overall, the results show that the Schottky barrier engineering controls the speciation of ROS, thereby governing the photocatalytic efficiency of $TiO_2$+Pt catalysts in visible-light-driven BPA degradation.

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# INDIRECT TECHNIQUES OF EPR SPECTROSCOPY AS AN EFFECTIVE TOOL IN DETECTION OF TRANSIENT PARAMAGNETIC SPECIES

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Various strategies have been developed to improve the optical properties of wide-bandgap metal oxides such $TiO_2$ or ZnO, and a relatively new approach involves preparation of systems with oxygen vacancies as defects. Oxygen vacancies show significantly enhance electrical, optical and magnetic properties and their presence improves the utilization of ultraviolet light and extends the absorption edge into the visible-light spectrum. This enhancement increases the efficiency of various photocatalytic and photoelectrochemical processes. Oxygen vacancies can *i*) introduce additional energy levels in the material, *ii*) act as specific reaction sites for certain molecules in (photo)catalysis (*e.g.*, they can serve as electron scavengers, facilitating the conversion of oxygen gas into superoxide radical anions), *iii*) alter chemical reaction rates by enabling charge transfer from electrons or holes, and *iv*) enhance the electrical conductivity of materials. So, the oxygen vacancies may influence the properties of synthetized metal oxides, and one of the goals within the research is to understand the dynamics of photogenerated charge carriers and intermediate radical species in metal oxides and to evaluate their photocatalytic performance in the degradation of various organic pollutants [1,2].

Due to paramagnetic character of oxygen vacancy defects and presence of transient species upon light exposure (UVA or visible) in such materials, the electron paramagnetic resonance (EPR) spectroscopy represents effective tool for their detection and characterization. The lecture will be oriented to indirect techniques of EPR spectroscopy which may be involved, their characteristics and applications [3-5]. More in detail will be explain the spin trapping, spin scavenging, oxidation of sterically hindered amines and photoreduction (photoinduced electron transfer between diamagnetic/paramagnetic species, *e.g.*, nitroxides, and methylviologens) in aqueous and non-aqueous systems.

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# PHASE CHANGES IN $WO_3$ FILMS UPON HEATING AND COOLING.

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$WO_3$ is a promising photocatalyst for drinking water and wastewater treatment. A reproducible crystal structure would be necessary to investigate and optimise the catalytic properties, as amorphous $WO_3$ layers are less suitable for photocatalysis. Temperatures between 300 and 700 °C are often used to produce crystalline $WO_3$ layers. During heating, several phase transformations can occur, which are more or less reversible. Studies on powders have shown that at least seven modifications of $WO_3$ can exist in a temperature range from room temperature to 1000 °C [1, 2]. At room temperature, $WO_3$ can exist in monoclinic, orthorhombic and triclinic structures. However, determining the crystal structure of films is considerably more difficult than for powders, as transfor-mations can take place over larger temperature ranges which can depend primarily on the layer thick-ness, the temperature treatment, the substrate, the manufacturing method and the starting products.

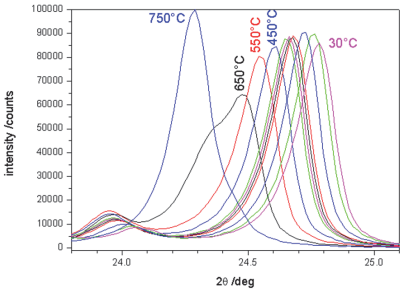


Figure 1: Phase change in a $WO_3$ layer in the range of the (200) peak on fused silica during cooling from 750 °C to 30 °C via XRD-measurements.

In this work, the structural changes during heating of amorphous layers to 750 °C were investigated by *in situ* XRD and compared with the changes during cooling of the calcined layers. Both fused silica and FTO-coated borosilicate glass were used as substrates [3]. The coating was applied using spray pyrolysis of peroxotungstic acid with a substrate temperature of 250 °C. In the range from 650 to 730 °C, a phase transformation could be clearly observed both during heating and cooling, which in powders was attributed to the transformation of orthorhombic (Pbcn) to monoclinic ($P2_1/c$). A transformation from the monoclinic to the orthorhombic or the triclinic phase could not be demonstrated. The slight difference in the diffraction peaks did not allow a distinction to be made using this method for polycrystalline layers, in contrast to powders. A clear specification of the crystal structure of $WO_3$ layers is therefore not possible with the X-ray examination described and requires more specific methods.

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# PLASMA DIAGNOSTICS FOR REACTIVE HiPIMS PROCESS CONTROL OF VANADIUM OXIDES

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$VO_2$ thin film as a potential thermochromic and photochromic material with metal-insulator transition (MIT) characteristics, can be obtained via reactive high power impulse magnetron sputtering (r-HiPIMS) [1] due to the clean processing environment and high quality. However, a key challenge in r-HiPIMS of vanadium oxides is to regulate the deposition process in the transition region and obtain stoichiometric films, thus it's necessary to effectively monitor the deposition conditions, especially the deposition conditions as well as the plasma characteristics in real-time.

Based on the RF Sobolewski method [2-3] which measures the plasma properties including *ion flux, plasma sheath impedance, electron density* and *electron temperature* by inserting a probe at the substrate position, [4-5] the plasma properties in the vicinity of target can also be measured. In this way, the target itself is regarded as the "probe", thus no extra probe is required to insert into the chamber. Such non-invasive method has been proposed by D. Ludin et al. [6] to prepare for the plasma diagnostics especially for industrial systems. This work has conducted the RF Sobolewski plasma diagnostics at both the substrate side (with an inserted probe) and the target side (target as the probe) during the r-HiPIMS process of vanadium oxides. The plasma characterization reveals that the imaginary part of sheath impedance is the most promising parameter for future HiPIMS process regulation. Besides, according to the HiPIMS discharge waveforms, the average current per pulse proves to be more helpful than the peak current (density) for the process regulation when constant HiPIMS power is set.

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# HYBRID LAYERS OF POLYMERIC AND GRAPHITIC CARBON NITRIDE FOR ADVANCED PHOTOACTIVE COATINGS

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Carbon nitride represents a promising semiconductor material for photocatalytic and photoelectrochemical applications in the field of sustainable technologies. Among its most important forms are graphitic carbon nitride (g-$C_3N_4$) and poly(heptazine imide) (PHI). While g-$C_3N_4$ is distinguished by high chemical stability and sensitivity to visible light, its practical use is limited by poor conductivity and processability. In contrast, PHI exhibits the ability to form stable aqueous formulations and gel systems, which enables the efficient preparation of homogeneous and mechanically robust layers without the need for binders [1,2].

The aim of this work was to prepare and characterize coatings based on PHI and its blends with g-$C_3N_4$. Three types of formulations were prepared – pure PHI and PHI:g-$C_3N_4$ blends in ratios of 1:1 and 3:1. The coatings were deposited by doctor blade and drop-casting techniques. After deposition, the samples were subjected to three thermal treatment regimes: air drying, heating at 150 °C for 1 hour, and calcination at 450 °C in a nitrogen atmosphere. The selected processing conditions significantly influenced the crystallinity and structural ordering of the prepared layers.

Characterization included profilometry to determine thickness and homogeneity, scanning electron microscopy (SEM) for morphological evaluation, Fourier transform infrared spectroscopy (FTIR) for chemical bonding analysis, and X-ray diffraction (XRD) for crystallinity assessment. Photoelectrochemical properties were investigated using linear sweep voltammetry and chronoamperometry, which provided information on stability and charge separation efficiency within the layers.

The results demonstrated that pure PHI formed compact and stable coatings, while its blends with g-$C_3N_4$ exhibited higher photoelectrochemical response and improved charge transport, highlighting the synergistic effect of combining both materials. These findings confirm the potential of water-soluble PHI as a suitable precursor for the preparation of photoactive layers and indicate their applicability in solar energy conversion and environmental technologies.

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