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Hydrogen Production in Institute of Fluid Machinery of Polish Academy of Science in Gdańsk – theory and practice

Abstract

The hydrogen production technologies developed in the Institute of Fluid-Flow Machinery, Polish Academy of Sciences in Gdańsk are discussed here. They include the following methods: dark fermentation, photoelectrochemical water oxidation and hydrocarbons (or alcohols) reforming by microwave plasma. The potential of hydrogen production by using dark fermentation of different popular wastes such as: agricultural wastes, textile or wood waste, was determined using suitable models. Also, the influence of microaeration during dark fermentation of some substrates, e.g. sour cabbage, was tested. Photochemical oxidation is a water-splitting process driven by radiation at the surface of a titanium-oxide anode. The Si microrods covered by titania films were verified as a photoanode material. The hydrogen production from methane, ethanol, isopropanol and kerosene was driven by a microwave plasma. The results obtained confirm that microwave plasma sources have a high potential for hydrogen production via gaseous and liquid fuels reforming.

Keywords: hydrogen, dark fermentation, plasmolysis, photochemical water oxidation.

1. Introduction

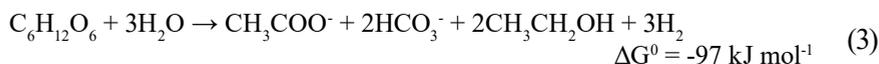
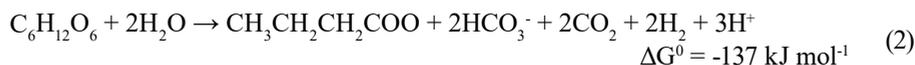
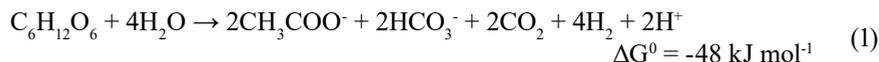
Hydrogen is considered as the fuel of the future due to its high heat of combustion and its abundance (Biedron, 2015; Das, 2001; Sołowski, 2016a). Growing costs of energy and its demand in Poland brings with it increased involvement of research centers (among them Institute of Fluid-Flow Machinery, Polish Academy of Science in Gdansk — IMP PAN) in the development of renewable hydrogen-production technologies. Three methods of hydrogen production are developed in IMP PAN, including dark fermentation, microwave plasma assisted reforming and photochemical water oxidation. The dark fermentation research included theoretical analyses of recent data and modeling of hydrogen-source potential as well as experimental investigation of yields for various substrates (e.g. cotton or sour cabbage) and processes (e.g. the role of microaeration). The microwave plasma research (started in 2012) focused on efficiency of hydrogen production from ethanol, kerosene and isopropanol.

The research related to the photoelectrochemical water oxidation was established in 2016 in collaboration with the research group from Nanobiomedical Centre of Adam Mickiewicz University in Poznań. The undertaken efforts cover electrochemical and

photoelectrochemical characterization of catalytic nanomaterials i.e. silicon nano- and micro-rods covered with different metal oxide films.

2. Dark Fermentation

Dark fermentation is a type of anaerobic digestion, which transforms simple organic compounds like glucose or glycerol into hydrogen, carbon dioxide, and organic acids. It proceeds using three possible pathways: acetate, butyrate or acetate-ethanol process (Bartacek et al., 2007; Woodward et al., 2000):



The hydrogen production by dark fermentation requires less energy than other methods and depends “only” on two variables: raw materials and bacteria (Sołowski, 2016a; 2018). The optimal condition for dark fermentation is widespread availability of raw material rich in carbohydrates (Toledo-Alarcón et al., 2017). According to Ren et al. (Ren et al., 2011; Wang et al., 2013; Liu et al., 2015) pure hexoses and pentoses are the most efficient raw materials for hydrogen production; high yields 124 g of H₂/g of glucose from wastes like used chewing gum (and 2l H₂ per litre of wastes) were confirmed by Seifert et al. (2018). Unfortunately substrates rich in simple sugars seldom occur in wastes, so it is necessary to use more widely available substrates such as lignocellulosic ones: e.g. corn wastes, wood wastes and herbivorous manure (Sołowski, 2016; Xing et al., 2010; Zhu et al., 2009).

The bacteria active in dark fermentation are divided into 2 groups: strict and facultative (Chaganti et al., 2012; Sołowski et al., 2018; Tommasi et al., 2008). Strict bacteria or obligate anaerobes (mostly *Clostridium*) are oxygen and substrate sensitive but produce hydrogen with high efficiency. Facultative bacteria like *Enterobacteria*, *Bacillus* or *Citrobacter* are less sensitive to oxygen or feed quality but at least 12% less efficient in hydrogen production than *Clostridium* (Batista et al., 2018). In order to produce hydrogen with high efficiency, the bacteria should be pretreated using thermal (heat shock), physical or chemical processes. These inhibit methanogenic processes that are consecutive to the hydrogen production stage of anaerobic digestion (Wang & Wan, 2008; Chaganti et al., 2012; Chasnyk et al., 2015). The stable continuous hydrogen production is hardly possible, so study results are related to batch type reactors or different types of CSTR (continuous stir tank reactors) (Ferraz et al., 2015; Keskin et al., 2018; Sołowski et al., 2018; Spasiano, 2018). The process can proceed under psychrophilic (below 30°C), mesophilic (33–38°C) or thermophilic conditions (above 55°C). The optimal pH value is in the range between 5 and 6 (Ghimire et al., 2015).

2.1. Model for calculation of hydrogen potential of the raw material

There are two lignocellulose-waste components desired for efficient dark fermentation, i.e. hemicellulose and cellulose, while lignin is considered as inhibiting of dark fermentation (Kongjan et al., 2010; Monlau et al., 2013; Toledo-Alarcón et al., 2017). The mass ratio of cellulose, hemicellulose and lignin vary for different kinds of plant, part of plant, age and area of occurrence. Thus for calculating of hydrogen potential a model was formed considering these lignocellulosic wastes and used to calculate the potential of some textile waste (cotton, flax) (Sołowski, 2016b), wood wastes (beech conifer, spruce, oak, pine) (Sołowski, 2016c) and cereal waste (for corn, wheat and barley) (Sołowski et al., 2019). From 1 kg of these wastes one can obtain potentially:

- 21 g of hydrogen from cotton waste,
- 19 g of hydrogen from linen waste,
- 16.9g of hydrogen for pine wood 1–20 years old,
- 17.4 g of hydrogen for pine wood 21–40 years old,
- 15.9g of hydrogen for pine wood 41–60 years old,
- 16.1g of hydrogen for pine wood 61–80 years old,
- 13.2 g of hydrogen for pine wood 81–100 years old,
- 16.6 g of hydrogen for spruce wood 41–60 years old,
- 11.8 g of hydrogen for fir wood 1–20 years old,
- 11.9 g of hydrogen for fir wood 21–40 years old,
- 12 g of hydrogen for fir wood 41–60 years old,
- 12.1g of hydrogen for fir wood 61–80 years old,
- 12.2 g of hydrogen for fir wood 81–100 years old,
- 17.5 g of hydrogen for beech wood 41–60 years old,
- 14.7 g of hydrogen for oak wood 1–20 years old,
- 14.8g of hydrogen for oak wood 21–40 years old,
- 15.6 g of hydrogen for oak wood 41–60 years old,
- 15.7 g of hydrogen for oak wood 61–80 years old,
- 16 g of hydrogen for oak wood 81–100 years old,
- 14.39 g of hydrogen from wheat straw,
- 13.86 g of hydrogen from wheat bran,
- 11.18 g of hydrogen from barley straw,
- 14.03 g of hydrogen from corn straw.

2.2. Role of microaerobic conditions

Influence of microaerobic conditions on dark fermentation of sour cabbage under neutral pH conditions, for VSS (volatile suspended solids) 5 g/l and 10 g/l was investigated (Sołowski et al., 2018a; Sołowski et al., 2018b). One of the known problems, which must be solved before viable process industrialization, is methane production inhibition (the process that consumes hydrogen). It is already known that hydrogen sulphur formation during anaerobic digestion can be prevented by the addition of small amounts of oxygen (Duangmanee 2009; Jenicek et al., 2010; Ramos et al., 2014). It was checked which process: methanogenesis or hydrogenesis, is more sensitive to small oxygen presence.

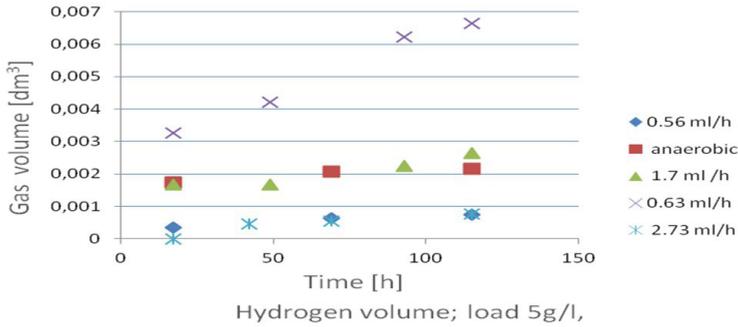


Fig. 1. Time evolution of total hydrogen production under strict anaerobic conditions (red squares) and with small oxygen flow rates: 0.56 ml/h (dark blue diamonds), 0.63 ml/h (violet crosses), 1.7 ml/h (green triangles), 2.73 ml/h (light-blue stars) (Sołowski et al., 2018; Sołowski et al., 2018b)

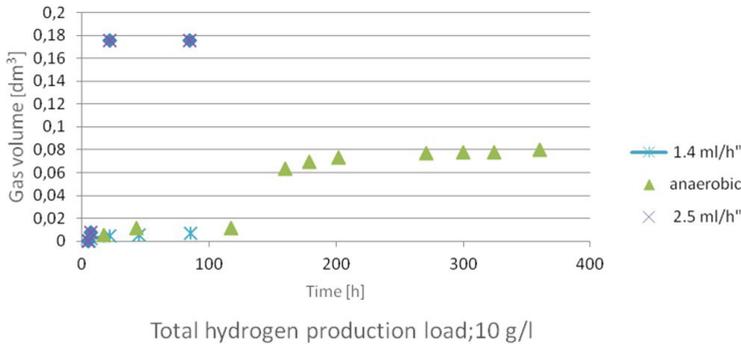


Fig. 2. Time evolution of total hydrogen production for load 10 g/l of sour cabbage under strict anaerobic conditions (green triangles) and with small oxygen flow rates: 2.5 ml/h (violet crosses), 1.4 ml/h (light-blue stars) (Sołowski et al., 2018; Sołowski et al., 2018a)

After 115 hours of dark fermentation, under strict anaerobic conditions and substrate load 5 g/l, methane production is twice as high and hydrogen production is 3 times as low compared with a system with oxygen flow rate 0.63 ml/h — see Fig. 1. The proper oxygen flow rate that can improve hydrogen production in the case of 5 g/l feed rate must be higher than 0.56 ml/h and lower than 1.7 ml/h.

For feed VSS 10 g/l the oxygen flow rate should be a minimum of 2.5 ml/h in order to increase hydrogen production. The highest hydrogen production was observed with an oxygen flow rate of 4.5 ml/h — see Fig. 2 (Sołowski et al., 2018a; 2018b).

3. Microwave plasma — based hydrogen production

The atmospheric-pressure microwave-plasma sources (MPS) suitable for hydrogen production from gaseous and liquid fuels offer significant advantages. Microwaves

(MW) supply the energy to create plasma in gases. Properly designed MPS assures high efficiency (almost 100%) of microwave power transfer from generator to the plasma. Thus MPS are considered to be more efficient in energy transfer than other plasma sources (i.e. dielectric barrier discharge, corona discharge or glow discharges). A MW plasma operated by electricity provides a fast response time of electrons, ionised and excited states of atoms, molecules and radicals of highly reactive properties. Due to this the catalyst-free processing of different hydrogen precursors becomes possible, at the same time avoiding the potential problems related to catalyst deactivation and regeneration. Additionally, thanks to the electrodeless operation of the MPS, a high purity plasma can be obtained.

Various atmospheric pressure MPS were designed, which supplied power through waveguides, coaxial lines or micro-strip lines, in IMP PAN (Mizeraczyk et al., 2012). However, for hydrogen production purposes a waveguide-supplied metal cylinder-based MPS (Jasiński et al., 2013) of different configuration and operated at two MW frequencies (915 MHz and 2.45 GHz) appeared to be the most convenient. The applied MPS configuration includes introducing the hydrogen precursor into the microwave plasma region either in the form of a swirl or axial flow, or directly into the MW-plasma flame, behind the MW-plasma generation-region. The decomposition of gaseous (methane, biomethane) and liquid hydrogen precursors (ethanol, isopropanol, kerosene) was performed. Figure 3 presents the idea of the microwave (915 MHz or 2.45 GHz) plasma system for hydrogen production from methane.

The influence of supplied microwave energy, of hydrogen precursor flow rate and of plasma forming gas flow rate on energy efficiency of hydrogen production (hydrogen production rate ($\text{g}[\text{H}_2]/\text{h}$), energy yield of hydrogen production ($\text{g}[\text{H}_2]/\text{kWh}$), conversion degree of the hydrogen precursor (%), hydrogen volume concentration in the outgas (%) and concentrations of other gaseous compounds resulting from the hydrogen precursor processing was investigated.

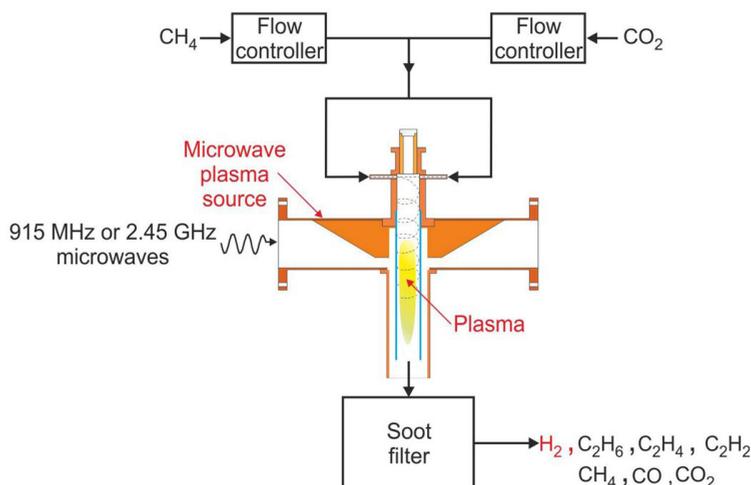


Fig. 3. Scheme of the microwave (915 MHz or 2.45 GHz) plasma system for hydrogen production from methane

Table 1. Comparison of hydrogen production rates and energy yields for selected microwave plasma methods of hydrogen production

Initial gas composition (frequency)	Hydrogen production rate g(H ₂)/h	Energy yield g(H ₂)/kWh	Reference
CO ₂ — 3000 NL/h CH ₄ — 3000 NL/h (915 MHz)	156 108	21 24	Hrycak, Czyłkowski, Jasiński, Dors, & Mizeraczyk, 2019
CO ₂ — 1200 NL/h CH ₄ — 3000 NL/h H ₂ O — 2 kg/h (2.45 GHz)	192	43	Czyłkowski et al., 2016
N ₂ — 3900 NL/h C ₂ H ₅ OH — 1.2 kg/h (915 MHz)	96 67	19 22	Miotk et al., 2016
CO ₂ — 2700 NL/h C ₃ H ₇ OH — 2.4 kg/h (915 MHz)	93	18.5	Miotk et al., 2016
CO ₂ — 2700 NL/h Kerosene — 1.2 kg/h (915 MHz)	37	7.5	Czyłkowski et al., 2018

Table 1 presents a comparison of the best obtained hydrogen production efficiency parameters. It can be seen that the highest hydrogen yield was achieved in the case of combined steam reforming of methane.

4. Photochemical oxidation

The research in IMP PAM focused on verification of photochemical oxidation of different metal oxide layers loaded onto the silicon pillars acting as an ordered substrate. In the paper by Pavlenko et al. (2017), the influence of thickness of TiO₂ layer deposited over the Si pillars on their activity in solar driven water decomposition was studied. The voltage obtained for photolysis equals 1.16 V vs. RHE while usually water splitting process proceeds at 1.30 V (Grimes et al., 2008). The Si/TiO₂ exhibits weaker efficiency by 25% than WO₃, however this material is very much cheaper (Alexander et al., 2008; Bloor et al., 2016). The result is also better than reported by Ni et al. (2007). The highest current registered upon electrode illumination was achieved for material composed of 40 nm-thin titania film obtained via atomic layer deposition over the 5 μm-long Si pillars, see Fig. 4.

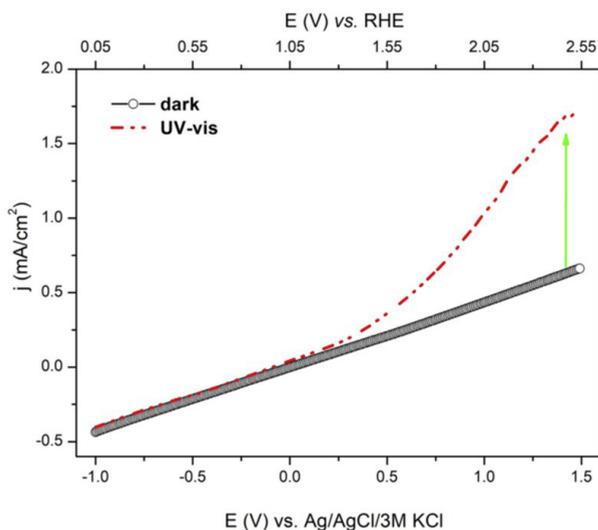


Fig. 4. Linear voltammetry curves of photocurrent density versus potential in TiO_2/SiNP for n-type Si. Length of SiNP equal to $5\ \mu\text{m}$; ALD TiO_2 thickness equals $40\ \text{nm}$; electrolyte solution was $0.5\ \text{M}\ \text{K}_2\text{SO}_4$ (Pavlenko et al., 2017)

5. Conclusions

Three technologies of hydrogen production, based on microwave plasmolysis, photochemical oxidation and dark fermentation, have been investigated in IMP PAN. A theoretical model has been developed to calculate potential of hydrogen production using the dark fermentation method from various substrates such as textile, wood and corn wastes. It was also proven experimentally that microaeration applied to anaerobic digestion of some substrates (like sour cabbage) inhibits methanogenesis and so, it supports hydrogenesis.

The experimental results of microwave-plasma investigation show that the hydrogen production efficiencies in the waveguide-supplied metal cylinder-based MW-plasma reactor of different configuration are acceptable. For example, the methane reforming (in mixture with CO_2 and H_2O) resulted in the energy yield of hydrogen production of $43\ \text{g/kWh}$, which is close to the U.S. Department of Energy (DOE) requirement ($60\ \text{g/kWh}$ by 2020) (see Randolph, 2013). Also, the absence of oxygen as by-products in the off-gas is highly beneficial. So, the proposed microwave plasma system is found to be a low cost and effective means of hydrogen production, and thus promising for industrial implementation.

Finally, it was found that the TiO_2 layer deposited over the Si pillars increase their activity in solar driven water decomposition.

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