Direct synthesis of hydrogen peroxide with CO₂ as solvent in a membrane micro reactor

Aneta Pashkova, Roland Dittmeyer*
Karlsruhe Institute of Technology, Institute for Micro Process Engineering
e-mail: pashkova@dechema.de

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Motivation

- Environmental advantages: Non-toxic, non-flammable, high activity and selectivity compared to conventional oxygen.
- Economically viable: Suitable for large scale production units (>40 kt/a).
- Environmentally harmless: The by-product is water.

Objectives

Aim of the project is to develop a compact and efficient continuous process for "on site" production of aqueous hydrogen peroxide solutions, based on the direct oxidation of hydrogen with oxygen in liquid or supercritical carbon dioxide over Pd supported catalysts in a special microstructured double membrane reactor.

Advantages of the microreaction technology
- Improved process safety
- Enhanced transport properties
- Non-toxic, non-flammable
- Easy separable from reaction mixture

Advantages of CO₂ as a reaction medium
- Solid CO₂ sublimation line at -78.5°C, 1 bar
- CO₂ liquid at 0°C, 10,000 bar
- CO₂ critical point at 304°C, 73.8 bar
- CO₂ triple point at 216°C, 5.1 bar

Experimental set-up

The experimental set-up is schematically represented below. In the first project phase it was put into operation without the implementation of the CO₂ loop, which is planned for a later stage after extensive investigation of the gas phase composition at the exit of the rotameter, in order to meet the exact requirements for the CO₂ compressor.

Operating conditions and principle

- Pressure: -10 to 60 °C
- Flow: 0.3 – 1.2 L h⁻¹
- Pd amount: ca. 100 mg
- Prototype reactor – upscale factor 10 compared to the laboratory microreactor

Catalyst screening

Aim of these experiments is to identify the most suitable catalyst for the direct synthesis reaction in an earlier project phase. A number of test micro reactors were prepared, each of them representing a separate catalyst in the form of wall coating of the microchannels. 1%Pd/Al₂O₃ and 1%Pd/TiO₂ were the first chosen catalysts in the form of wall coating and other additives.

Test reactors:
- Channel geometry (l, w, h): 150 x 0.5 x 0.6 mm
- Number of channels per plate: 20

Catalyst coating method – washcoating:

Step 1: Preparation of the catalyst powder by impregnation of the support material (TiO₂, Al₂O₃) with the active component (Pd: 0.5; 1 or 2 % w/w) and calcination

Step 2: Preparation of a suspension of the catalyst powder with addition of a binder and other additives

Step 3: Coating of the micro channels

Step 4: Drying and calcination of the coatings either in air or nitrogen atmosphere

The thickness of one coating layer is 20 - 25μm and can be varied through multiple coatings.

Project partners
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