

A new measurement technique for the process monitoring of the catalytic activity of nanoparticles during their production process

N. Neubauer, M. Seipenbusch and G. Kasper

Institute for Mechanical Process Engineering and Mechanics,
Karlsruhe Institute of Technology (KIT), 76131, Karlsruhe, Germany

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Presenting author email: nicole.neubauer@kit.edu

Introduction

For process monitoring and quality assurance integrated gas phase processes require online methods for the determination of the catalytic behaviour of the produced catalyst nanoparticles. In addition, an online technique capable of a substance-specific detection of catalytically active nanoparticles against a background of particles in workplace air is also still needed (Kuhlbusch *et al.*, 2009; Murashov *et al.*, 2009). For very active nanoparticles, such as platinum or nickel, the method of aerosol catalysis enables an online measurement of the catalytic activity of the particles (Weber *et al.*, 1999; Seipenbusch *et al.*, 2001). Less catalytically active nanoparticles, e.g. iron oxide, require an accumulation of nanoparticle material prior to catalytic investigations (Neubauer *et al.*, 2011). Therefore we studied a new measurement technique combining particle sampling on a substrate and the catalytic reaction into one instrument. In the following work the method capable of detecting the catalytic activity of nanoparticles and the achieved results are demonstrated.

Experimental

Catalytically active palladium nanoparticles generated by spark discharge were used as catalysts. The hydrogenation of ethene was chosen as a suitable catalytic test reaction which already runs at ambient air temperature.

In a first step the palladium particles are deposited on a substrate using either filtration or an impaction based process. After the particle deposition over a fixed time interval on a glass fiber filter or a glass impaction plate, the aerosol flow is cut. Both substrates can be heated so that – if the chosen catalytic reaction requires it – the temperature can be increased.

In a second step the deposited palladium particles are exposed over a defined time to the gaseous educts ethene and hydrogen (each 3.0, 50 vol-%) so that the catalytic reaction is initiated. The conversion of ethene to the gaseous reaction product ethane is detected by infrared spectroscopy (Bruker Vector 22).

Results

The concentration of the converted ethene was related to the mass of the accumulated palladium nanoparticles which was determined by gravimetry. For both deposition processes observation gives that the concentration of the converted ethene increases proportionally to the mass of the used palladium nanoparticles (cf. Figure 1 for deposition by filtration).

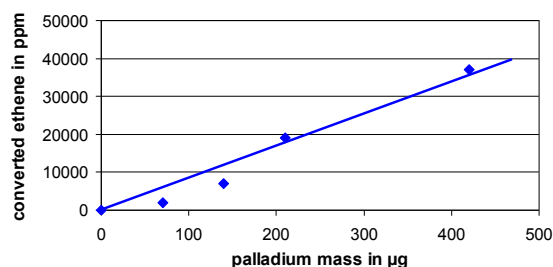


Figure 1. Calibration curve for the concentration of converted ethene vs. the mass of palladium nanoparticles deposited by filtration.

A lower detection limit of 120 ng can be extrapolated based on the curve linearity and assuming a measurement limit of 10 ppm of the ethene decrease by infrared spectroscopy. At aerosol concentrations of 1 mg/m³ and a flow rate of 1 l/min a sampling time of only 8 seconds is sufficient to deposit a mass of 120 ng of palladium particles.

Conclusion

Small amounts of a palladium aerosol can be very rapidly detected on the basis of their catalytic activity. According to the estimated sensitivity of the used technique in the range of nanograms as well as the required sampling time, the technique can be considered a true real-time method for the specific determination of palladium nanoparticles.

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