Nanospring™-Enhanced Continuous Flow Reactors

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INTRODUCTION

A continuous flow reactor (CFR) containing a film with immobilized catalyst particles emerges as a viable alternative not only to facilitate the scale-up process but also to overcome the difficulties involved in the separation of catalyst particles from reaction products.

In the present work, we report the results obtained in a CFR using palladium nanoparticles immobilized on a silica-nanospring substrate for the reduction of 4-nitrophenol with sodium borohydride.

CATALYST CHARACTERIZATION

Loosely intertwinned Nanosprings create an inverse porous material with 100% open porosity

Individual Nanospring comprised of multiple silica (SiO_2) nanowires ~5nm in diameter wrapped around each other in a helical configuration

300m^2/g

1500cm^2 per cm^2 of footprint

The average particle size for palladium is 1-3nm.

The amount of palladium on silica Nanosprings used in the present work was 0.8%w.

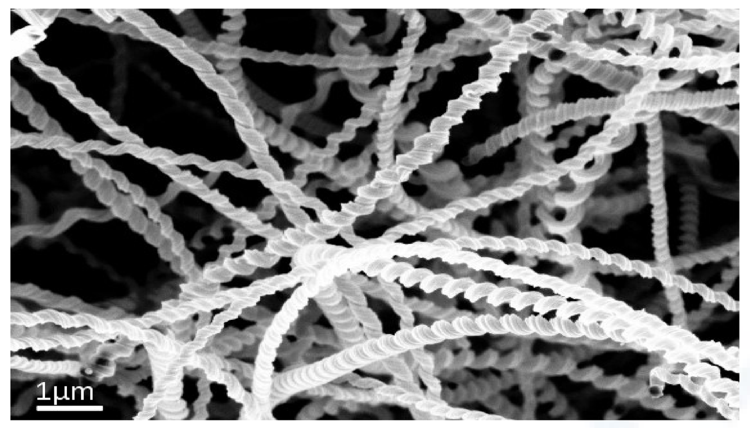


Figure 1.
SEM image of a silica Nanospring mat.

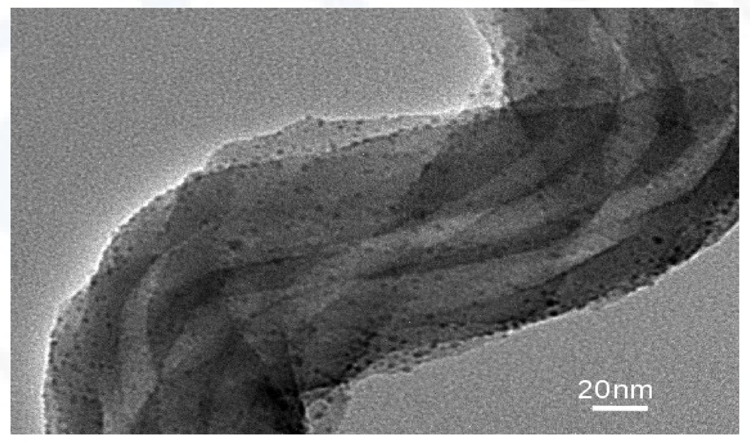


Figure 2
TEM image of a single silica Nanospring coated with palladium nanoparticles.

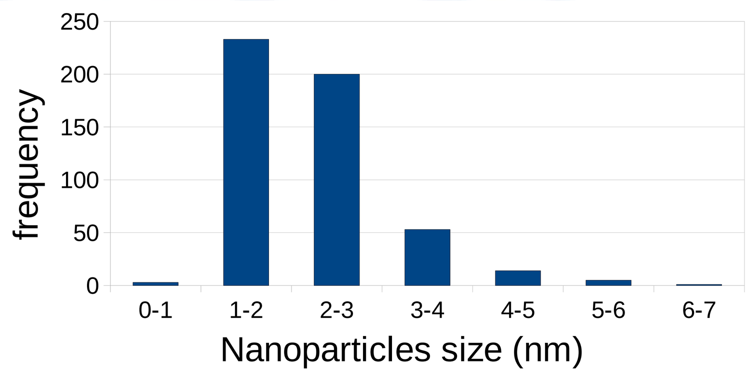


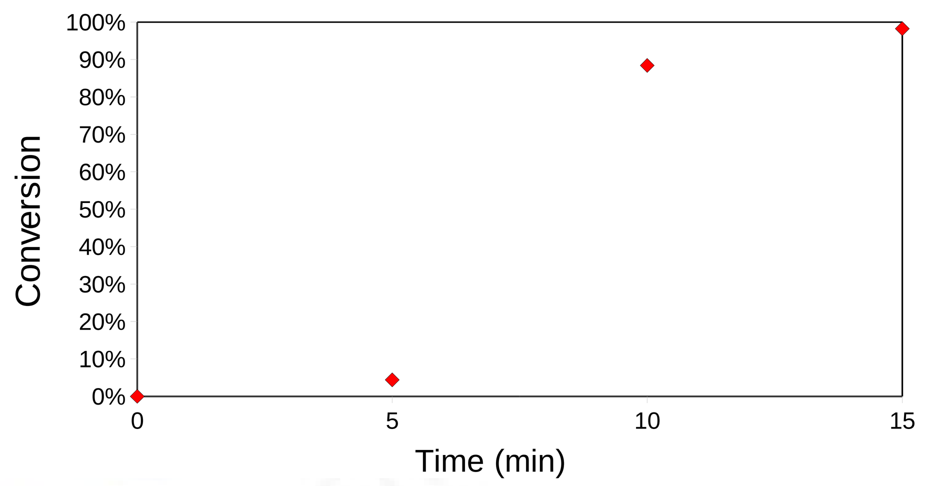
Figure 3
Pd nanoparticle size distribution

EXPERIMENTAL RESULTS

CATALYTIC ACTIVITY TEST

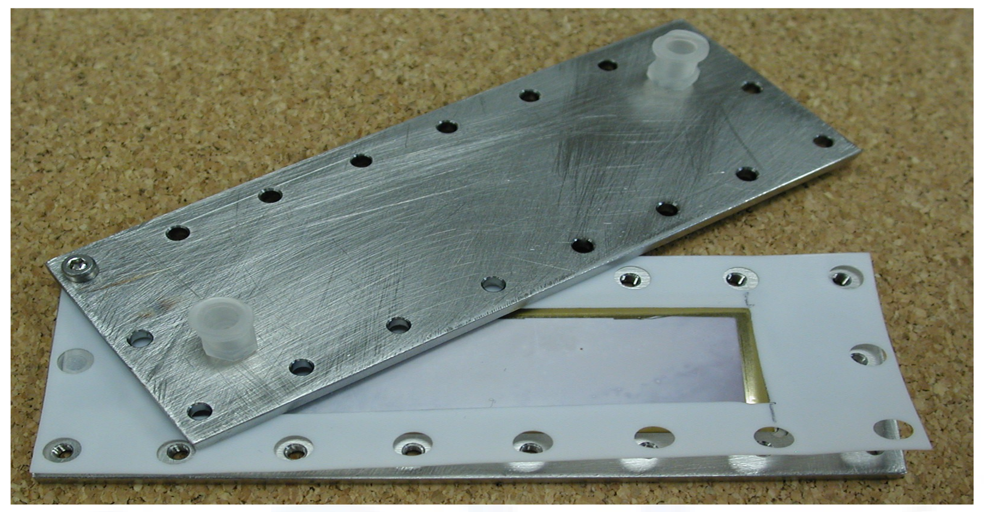
To assess the performance of the catalytic material, a sample was attached to a microscope slide and immersed in a beaker with 120 mL of a solution of 4-nitrophenol 1 mM. Next, 50 mg of solid sodium borohydride were added to the beaker under constant agitation and the absorbance (400 nm) of the solution was monitored over time to calculate the conversion.

Figure 4.
Conversion vs time



CONTINUOUS FLOW REACTOR TESTING

Figure 5.
Continuous Flow Reactor used in this work



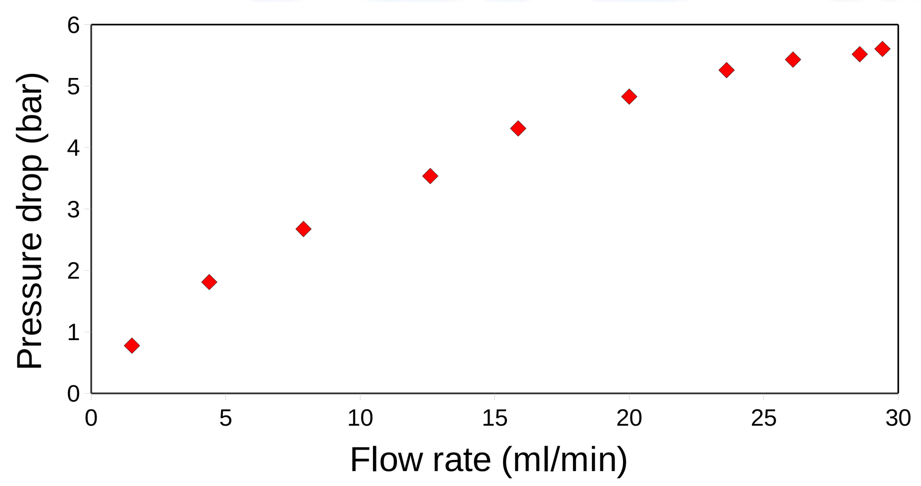


Figure 6.
CFR pressure drop as a function of the flow rate using water as reference fluid.

To test the microreactor, a solution of 4-nitrophenol 0.1mM was introduced into the reactor at different flow rates using a stroke pump. The reducing solution of sodium borohydride (0.3125 g/L) was supplied using a syringe pump at a constant flow rate (25 mL/h). The conversion was estimated by measuring the absorbance of the liquid product at 400 nm. The space velocity was calculated by dividing the total flow rate of reactants by the volume of the reactor (which was measured as equal to 0.1 mL).

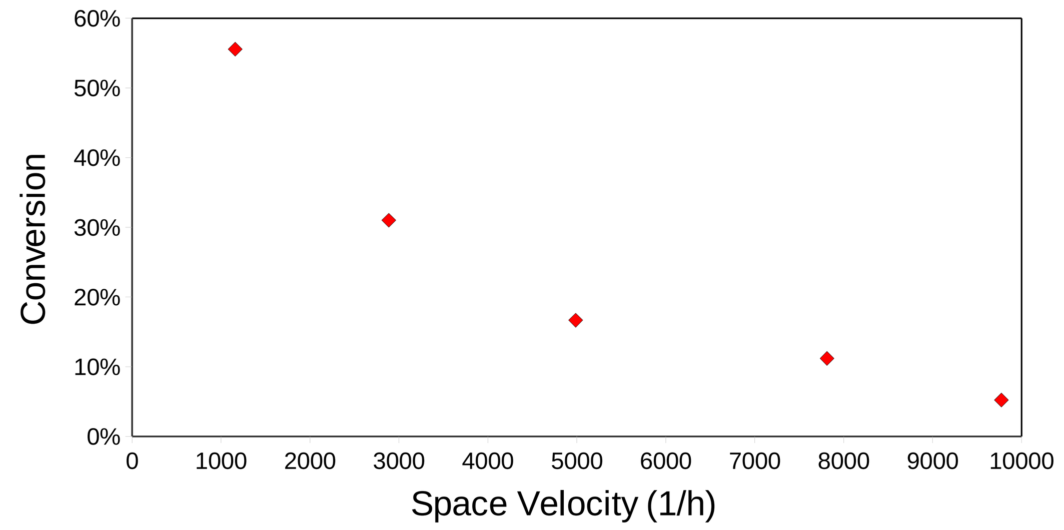


Figure 7.
Conversion vs. space velocity for CFR containing Pd supported on silica nanosprings

CONCLUSIONS

- The immobilization of palladium nanoparticles on silica Nanosprings produces a highly active catalyst sample, which effectively reduces 4-nitrophenol at rates higher than those reported in the literature.
- The CFR operating at single pass seems to enhance the catalyst performance as a result of the forced contact between catalyst film and reactants.
- The pressure drop through the reactor (∼1bar at 2ml/min) can be reduced by lowering the space velocity, which in turn increases the conversion. Large space velocities produce moderate pressure drops, however, to increase the conversion the CFR (58% at 2ml/min) must be operated in recirculating mode.

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