

# DESIGN OF NANOPOROUS CARBON MATERIALS VIA THE CARBIDE-DERIVED CARBON METHOD IN A FLUIDIZED BED REACTOR

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## *Summary*

For the first time nanoporous carbon synthesized via the carbide-derived carbon (CDC) method was obtained in a fluidized bed reactor. The kinetics of the reaction was investigated and compared to the kinetics gained during reactions in a horizontal hot wall tubular reactor. The usage of a fluidized bed reactor in the course of the CDC process seems to be very useful with the objective to create nanoporous carbon material with high accuracy.

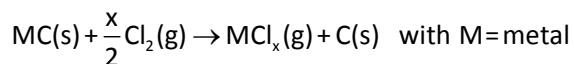
## *Keywords*

Novel functional materials; novel reactor technologies; rational design of catalysts

## **Introduction**

During the last decades nanoporous carbon materials have attracted a high content of interest because of their wide range of different possible applications [1]. These materials show for example interesting properties in their use in the field of separation technology as purification or separation media, electrode materials in electrochemical applications, carrier materials in the field of catalysis and as gas storage materials [2].

Gogotsi et al. presented studies concerning the synthesis and the design of nanoporous carbon materials via gas phase reactions. They applied the carbide-derived carbon (CDC) method to achieve highly porous carbon materials with a narrow pore size distribution with pores in Ångström accuracy [3]. In the course of this method metal carbides are chlorinated at high temperatures to remove the metal selectively:



Recent research is applied in the improvement of the process by varying for example the chlorination temperature. An alternative approach to achieve process intensification is to use a more suitable reactor type in the course of the CDC process. Normally the reaction takes place in a hot wall tubular reactor, where due to the chlorine concentration depletion material from the beginning of the reactor is etched more than material at the outlet. A fluidized bed reactor seems to be useful to

achieve mixing of the carbide particles and an isothermal temperature profile inside the reaction zone. This can lead to better energy utilization and a higher space time yield.

In this work a novel approach to synthesize nanoporous carbon materials is presented, the CDC method performed in a hot wall fluidized bed reactor. The achieved carbon structures were analyzed and compared with the materials that were synthesized in a conventional horizontal hot wall tubular reactor.

## **Methods**

Different metal carbide bulk materials were chlorinated both in a conventional horizontal hot wall tubular reactor and in a fluidized bed reactor. As demonstrator material SiC powder ( $d = 40 \mu\text{m}$ ) and TiC powder ( $d = 4 \mu\text{m}$ ) were etched by chlorine gas treatment. The etching rate was investigated in dependence of the process temperature, the total gas volume flow, the reaction time, the chlorine gas concentration, the initial carbide material and the particle size.

The etching kinetics of different demonstrator materials of two different carbides, SiC and TiC, with two different particles sizes was obtained for the CDC process carried out in a horizontal hot wall tubular reactor. Furthermore CO<sub>2</sub>- and N<sub>2</sub> sorption measurements were performed to determine the dependency of different reaction conditions on the pore size distribution. Mercury porosimetry measurements were made to exclude the formation of

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additional macropores and consequently to proof the formation of a highly pure microporous carbon material. Laser supported particle size measurements were carried out to evaluate the change in size of the particles during the CDC process. Macrokinetic investigations concerning the mass transport during the chlorination reaction were also conducted.

## Results

The proof of concept for employing a fluidized bed for the production of carbide-derived carbons was successfully. Parallel tests in a hot wall tubular reactor showed different to the fluidized bed reactor an inhomogeneous etching and no rise in the etching rate if the ceramic crucible is loaded with higher amount of carbide.

The dependency of the etching rate was studied for different gas volume flows, different reaction temperatures and different chlorine contents for the horizontal reactor. Thereby nearly no influence of the temperature on the reaction rate was observed. Contrary to this the resulting micropore structure differs strongly for different temperatures, showing the importance of isothermal processing. Thereby mesopores are created at higher temperatures and the specific surface area gets lost. The chlorine concentration influence can be described fine with a Langmuir-Hinshelwood approach. Detailed kinetic studies are being performed at the moment also within the fluidized bed reactor.

## Conclusion

For the first time the carbide-derived carbon method was carried out successfully in a fluidized bed reactor. The application of a fluidized bed reactor for the CDC method offers a great potential in the field of process intensification. The reactor seems to be a promising alternative for the industrial production of CDC materials, compared to the horizontal tubular reactor which is employed in research. More homogeneous etching could be achieved within the reactor. Consequently the application of fluidized bed reactors in the field of gas phase reactions seems to be useful.

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