T. Meller, Prof. Dr. H.-U. Moritz, Dr. W. Pauer

University of Hamburg, Institute of Macromolecular and Technical Chemistry

Bundesstraße 45, 20146 Hamburg

Design and characterization of a gas-solid reaction calorimeter

Despite the wide range of applications in the technology of calorimetry, it is extremely challenging to investigate reactions between solids/ highly viscous reagents and gases. In order to address this problem, a gas-solid reaction calorimeter has been designed and realized. Essentially, it is a modified and insulated semi batch rotary pipe reactor (Fig. 1). By means of the design, heat can be measured via heat balance, solids can be continuously mixed and gas can be simultaneously dosed.

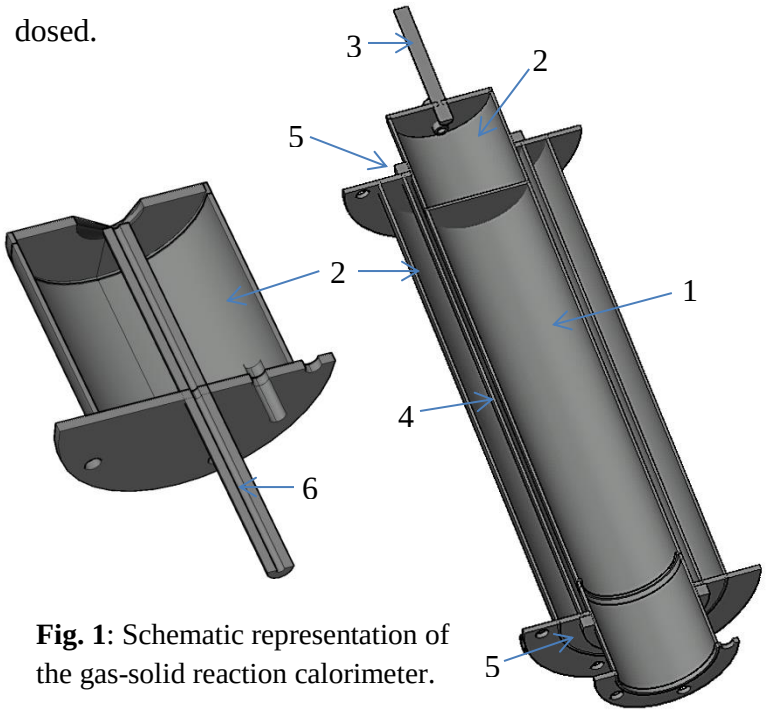

Fig. 1: Schematic representation of the gas-solid reaction calorimeter.

The cylindrical reactor consists of a reaction chamber (1) a cavity (2) and a rotating shaft (3) which allows the reactor to rotate. Further cavities (2) are also implemented in the lid and in the jacket of the reactor. The cavities can be evacuated in order to minimize heat losses to the environment. In combination with the reactor the jacket forms an annular gap (4). During a reaction a tempering medium flows through the gap and transports heat out of the reactor. By measuring the temperature at the inlet/outlet and considering the mass flow, the released heat can be calculated. Furthermore, the rotation of the reactor leads to a specific flow pattern in the gap (Taylor vortices) which facilitates a uniform flow with reduced backmixing. In order to hold the reactor in position and to avoid leaks, the jacket contains two ball bearing and two radial shaft sealings connected to a flange (5). Gas is dosed into the reaction chamber through a pipe in the lid (6). The flow regime in the annular gap as well as the motion behavior of solids inside of the reaction chamber was investigated. The accuracy of the system was quantified and the limits of detection were validated. Furthermore, gas-solid reactions were performed. As a

result the suitability of the reactor for calorimetric measurements was confirmed. In future research, chemical processes, which have not been proper investigated by calorimetric techniques due to a lack of adequate measurement systems, will be focused on.