

# Gas-phase polymerization of propylene at low reaction rates: a precise look at catalyst fragmentation

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**Abstract** In this work, we reported the development of a mini-reactor experimental setup for synthesizing of polypropylene with heterogeneous Ziegler–Natta catalysts in gas-phase. Use of pro-activated 4th generation of Ziegler–Natta catalyst and preheated monomer feed enabled the polymerization reaction to be carried out at constant temperature. Evaluation of monomer consumption with high precision (0.01 bar pressure drop) allowed the detection of polymerization yield at low reaction rates. In this regard, polymerization yield, particle morphology and catalyst fragmentation were studied, as well. The results of melt microscopy showed that catalyst fragmentation was developed during the reaction, and was not restricted to the initial rupture of catalyst particles. The rate determination showed a peak during the polymerization (not necessarily at the initial stage). The results showed that depending on the reaction condition, this peak could be either a consequence of a major catalyst fragmentation or overheating. Low reaction yield, large fragments of catalyst and agglomeration of particles were considered as evidence of particle overheating and polymer local melting. As we imposed the results of melt microscopy for the polymerization conditions, a layer-by-layer fragmentation of the catalyst was found to be the main fragmentation process, at least at the beginning of the polymerization reaction.

**Keywords** Mini-reactor · Propylene · Polymerization · Gas-phase · Fragmentation

## Introduction

Understanding of the mechanism of polymer growth has been, and still is, one of the most important fields of heterogeneous olefin polymerization studies. Many efforts have been made by research groups to give insight into the polymerization kinetics, catalyst morphology, polymer properties and mass transfer interdependency [1–3].

Catalyst fragmentation during polymerization affects polymerization activity of the catalyst as well as properties of the produced polymer (particle density, porosity, etc.) [4–6]. The morphology of the final polymer particles depends on the mechanical and structural properties of both the catalyst support and the polymer formed at the critical very early stage of the polymerization [7–9].

The first observation relevant to the mechanism of growth of polyolefins on heterogeneous catalysts dates back to the 1970s. Bulls and Higgins with the aim of microscopic images showed fragments of the catalyst particle dispersed within the expanding polymer [10]. Beside model predictions [11–13], many experimental investigations were carried out by researchers on catalyst fragmentation.

Microreactors have been recently employed for studying catalytic olefin polymerization reactions [14]. The use of microreactor devices makes it possible for researchers to perform reactions with good control over mixing, pressure, flow rate, residence time, mass and heat transfer, which resulted in enhanced reproducibility [15]. Different reactor and configurations have been designed to study very specific aspects of the reactions process.

Based on the use of microreactors coupled with visual and/or infrared (IR) microscopy, many in situ or online study methods have been developed to investigate particle growth. With the aim of online microscopy, particle growth kinetics during polymerization had been followed and profitable

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