# Mastering Particle Design: Advanced Catalyst Shaping with Top and Bottom Spray Fluidized Bed Methods

<u>Ali M. Alkadhem\*</u><sup>1</sup>, Hend Omar Mohamed<sup>1</sup>, Shekhar R Kulkarni<sup>1</sup>, Torsten Hoffmann<sup>2</sup>, Diego Zapater<sup>1</sup>, Valentina E. Musteata<sup>3</sup>, Evangelos Tsotsas<sup>2</sup>, Pedro Castaño<sup>1,4</sup>

<sup>1</sup> Multiscale Reaction Engineering, KAUST Catalysis Center (KCC), King Abdullah University of Science and Technology (KAUST), Thuwal 23955-6900, Saudi Arabia; <sup>2</sup> Thermal Process Engineering, Otto von Guericke University, Universitätsplatz 2, 39106 Magdeburg, Germany; <sup>3</sup> Imaging and Characterization Core Lab, King Abdullah University of Science and Technology (KAUST), Thuwal 23955-6900, Saudi Arabia; <sup>4</sup> Chemical Engineering Program, Physical Science and Engineering (PSE) Division, King Abdullah University of Science and Technology

\*Corresponding author: ali.alkadhem@kaust.edu.sa

#### Highlights

- Bottom and top spray fluidized bed process to produce technical catalysts.
- Elucidation of the particle stage growth.
- Utilization of dimensionless numbers to assess the different growth stages
- Principal component analysis identifies key parameters in the growth stage.

### 1. Introduction

Developing an advanced method for manufacturing technical catalysts using cutting-edge technology is strongly encouraged. This approach should enable a catalyst preparation process with high capacity while also allowing precise control over particle parameters like size distribution and morphology [1]. Various shaping techniques like extrusion [2], and spray drying [3] are employed based on the specific requirements of the catalyst and the process involved. This work aims to gain insights into the particle growth stages in a spray-fluidized bed using different spray configurations, namely top and bottom. Through a series of experiments, we investigated the effects of critical operating parameters of the granulation process, including atomization gas velocity, temperature, and liquid flow rate. Additionally, we examined the influence of feed properties such as solid concentration and viscosity.

### 2. Methods

Catalyst particles were generated in a bottom and top spray fluidized bed; Figure 1 depicts the interior configuration of the employed reactor. In the bottom spray arrangement, catalysts were created by creating a slurry in a dry base containing 50 wt% zeolite ZSM-5 (Si/Al = 25, Zeolyst International), 20 wt% bentonite (Sigma-Aldrich) as the binder, and 30 wt% alumina (Sigma-Aldrich) as filler. The catalysts were made using zeolite ZSM-5 as the initial mass bed for the top spray design. Bentonite and alumina were sprayed from the top over the fluidized tiny zeolite particles. Catalysts were examined with static light scattering, scanning electron, and optical microscopy. Dimensionless numbers and PCA analysis were employed to understand better the prevailing forces during the agglomeration phases and the effects of operating conditions and catalyst parameters.



Figure 1. Reactor configuration a) bottom b) top.

## 3. Results and discussion

Figure 2 shows the particle-size distributions as violin plots, where the three quartiles, namely,  $d_{25}$ ,  $d_{50}$ , and  $d_{75}$ , correspond to 25%, 50%, and 75% of the size distribution, respectively. To analyze the impact viscosity, we used the plots of V1 (0.1P), V2 (0.3P), and V3 (0.5P). The letter A stands for the effect of atomization at 3 and 7 Lmin<sup>-1</sup>, respectively, the F letter stands for the fluidization effect at 8, 6, and 5 Lmin<sup>-1</sup> for the bottom experiment and at 6 and 8 Lmin<sup>-1</sup> for the top experiment, the T letter stands for the temperature at 250 and 150 °C, L stands for the liquid flow rate at 4 and 6 mLmin<sup>-1</sup>, and G stands for total mass in the bottom spray experiment (8 and 4 g) and initial mass bed (1 and 5 g) for top spray configuration. Viscosity played a significant role in determining the particle-size distribution, as higher viscosities led to smaller average particle sizes and narrower distributions. Increasing the atomizing airflow resulted in smaller particle sizes and narrower distributions. The fluidizing airflow slightly affected the particle-size distribution in the case of the bottom spray but had a more significant effect in the top spray configuration. Temperature had an inverse relationship with the particle-size distribution. The liquid flow rate directly influenced the average particle size.



Figure 2. Catalyst particle-size distribution under the different operating conditions.

### 4. Conclusions

The batch fluidized bed process with both configurations successfully formed the homogenous spherical morphology of zeolite at different size distribution ranges. The study identified the effect of reactor configurations on the orders of growth stages: dust formation, seed formation, seed agglomeration, and a combination of seed agglomeration and dust layering. This finding is approved with the dimensionless numbers analysis. The microscopic images revealed the effect of process conditions on the stages and particle shapes.

#### References

- [1] A.B.D. Nandiyanto, K. Okuyama, , Adv. Powder Technol. 22 (2011) 1–19.
- [2] A.M. Alkadhem, F. Tavares, N. Realpe, G. Lezcano, A. Yudhanto, M. Subah, V. Manaças, J. Osinski, G. Lubineau, P. Castaño, A, Fuel. 349 (2023) 128717.
- [3] T. Shoinkhorova, A. Dikhtiarenko, A. Ramirez, A. Dutta Chowdhury, M. Caglayan, J. Vittenet, A. Bendjeriou-Sedjerari, O.S. Ali, I. Morales-Osorio, W. Xu, J. Gascon, ACS Appl. Mater. \& Interfaces. 11 (2019) 44133–44143.

## Keywords

Technical catalyst, spray fluidized bed, particle growth, dimensionless number.