

Single Particle Assays to Determine Heterogeneities within Fluid Catalytic Cracking Catalysts



Anne-Eva Nieuwelink



Marjolein E. Z. Velthoen



Kristel L. Jagtenberg



Yoni C. M. Nederstigt



Florian Meirer



Bert M. Weckhuysen



Utrecht University



MCEC

Netherlands Center for
Multiscale Catalytic Energy Conversion

ALBEMARLE®

Invited for the cover of this issue is the group of Bert M. Weckhuysen at Utrecht University. The image depicts an artistic impression of catalyst particles in full color: by combining multiple characterization techniques per single Fluid Catalytic Cracking (FCC) particle, insights are provided into the heterogeneity of an industrially used catalyst. Read the full text of the article at [10.1002/chem.201905880](https://doi.org/10.1002/chem.201905880).

What are the key findings?

The work shows that, by correlating information from multiple characterization techniques per FCC catalyst particle, valuable insights are provided into the heterogeneity of density-separated FCC particles. Furthermore, the possibility to measure multiple catalyst particles adds statistical value to the data obtained. Where bulk characterization measurements—i.e., chemical analysis by inductively coupled plasma-optical emission spectroscopy (ICP-OES), gas sorption and ammonia-temperature programmed desorption (TPD)—can give insight into the trends between the six, density-separated, fractions of FCC catalysts under study, the diagnostics of single catalyst particles, as provided by catalyst staining with dye molecules in combination with fluorescence microscopy, can show acidity and accessibility trends even within each fraction. Furthermore, the large intrinsic inter-particle heterogeneity of each density separated fraction was visualized by overlaying micro-X-ray fluorescence (μ XRF) maps of Fe and Ni with the fluorescence microscopy maps, obtained after catalyst particle staining. It was found that with increasing catalyst density and metal content, the acidity and accessibility of the catalyst particles decrease, while their distribution narrows with catalyst age. For example, particles containing high Ni levels possess very low acidity and are hardly accessible by a Nile Blue A dye. Single catalyst particle mapping identifies minority species, like the presence of a phosphated zeolite ZSM-5-containing FCC additive for selective propylene formation, catalyst particles without any zeolite phase, and catalyst particles which act as a trap for SO_x .

Why did you investigate this topic?

We looked for a laboratory-based approach that could bridge the gap between bulk- and single particle characterization, to overcome the lack of detail found with bulk characterization methods on the one hand, and the lack of statistical relevance for single particle based techniques on the other hand. Inspired by essays that are common practice in biochemical enzyme and cell research, we

looked for an approach that could screen many single particles in a similar way. The spent FCC catalyst is known for its large inter-particle heterogeneities and is therefore used in this showcase study.

What was the inspiration for this cover design?

The cover gives an artistic impression of the three analytical techniques used to characterize industrially used FCC particles. By overlaying/superimposing the three primary colored red, green and blue lasers, a full color image is obtained that reveals in full detail the properties of the (catalyst) particles on several important levels, namely the shape, structure and color of the particles in the image. The three lasers represent the characterization techniques that have been used to investigate the accessibility, acidity and metal accumulation in industrially used FCC particles. By combining the RGB colors, a white light is created that reveals all details of the particles under study, shown in the front of the image. The two particles that first appeared similar in the light of the three colored lasers, now show large differences. This idea represents the correlated chemical and physical properties of the catalyst particles, as revealed by the spectroscopic and microscopic techniques that were combined to reveal the heterogeneity of the FCC sample under study. These details only come to light by using multiple methods and overlaying their results.

