diamond

Oil

CASE STUDY

Understanding lubricant oil additives using X-ray absorption spectroscopy

Lubricant oil additives play a key role in the automotive industry by providing an increased control over tribological conditions in the engine which subsequently have a positive impact on the environment through reduced emissions achieved by improved energy efficiency. Overbased detergents – $CaCO_3$ products are widely used as corrosion prevention additives in combustion engine lubricants. The properties of $CaCO_3$ have been studied extensively but surprisingly, due to many polymorphs available, it is not yet fully understood.

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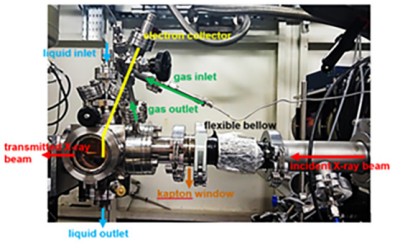
The Challenge

The $CaCO_3$ polymorphs formed, and their physicochemical properties, are greatly dependent on the reaction conditions such as temperature, pH, mixing dynamics, reaction time and the composition of the gas phase. Therefore, it is critical to study this material under *operando* conditions to follow the stages of lubricant oil additive formation and subsequently tune the conditions to obtain a desired form of $CaCO_3$. However, that's not an easy task considering the complex experimental set up, multiple phases of the system and the relatively light elements of $CaCO_3$ to be studied.

The Solution

Researchers from the University of Leeds, Infineum and Diamond have developed a liquid-jet environmental cell, dedicated to *operando* studies of reactions in liquid dispersions and solutions. This versatile set up with continuous flow was first used on B18 to study the early stages of $CaCO_3$ nucleation by Ca K-edge X-ray Absorption Spectroscopy (XAS) under *operando* conditions. Following this successful application, the team were able to combine the liquid-jet cell and a conventional lab-scale agitated reactor with XAS measurements to use as a process analytical technology (PAT) tool to monitor the synthesis of sulfonate-stabilized $CaCO_3$ particles.

The Benefits



This collaborative work has shown that the windowless liquid-jet configuration can accommodate a multiple gas-liquid-solid system and its dynamic processes in liquid dispersions. Solutions were successfully monitored by XAS in the tender X-ray range above 4 keV. The combination of a continuous-flow liquid-jet setup and *operando* XAS studies applied as a PAT tool allowed for a range of studies. These included the monitoring of chemical changes, the exploration of reagent-product correlations, and the investigation of a scaled-down industrial process to produce sulfonate-stabilised $CaCO_3$ particles.

"Understanding the polymorphic forms of crystalline materials, how they form and how different forms can be manipulated is vital across many industries, for ensuring both product performance and quality. Through our ongoing collaborations with the University of Leeds and Diamond Light Source it has enabled Infineum to gain a greater in-depth understanding of our products and the science underpinning them and is a vital element of our innovation and commercial developments."

Prof. Peter Dowding, Infineum

For further information

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