Evaluation of an in situ spatial resolution instrument for fixed beds through the assessment of the invasiveness of probes and a comparison with a micro kinetic model

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Introduction
Determining the spatial characteristics within a catalyst bed under reaction conditions would provide a formidable new tool to deliver the basic information required to allow the rational design and engineering of advanced catalysts. Recent research has led to the development of apparatus capable of providing temporal and spatial (axial and radial) analytical information [1, 2]. The results showed the importance of obtaining this sort of information in order to model the process and design better systems.

Presented here is a method [3, 4] to access concentration profiles of reactants, potential intermediates and products and temperature within a packed catalyst bed, under true reaction conditions. Additionally, the invasiveness of the probes is assessed using CFD, while results are compared with a micro-kinetic model [5] to gauge the strength and relevance of this technique.

Experimental
The principle of the technique is based on the combination of a stationary drilled capillary and a reactor connected to an automated linear stage (Figure 1). To monitor the temperature during the reaction a stationary thermocouple has been inserted in the capillary with its tip placed next to the sampling holes. The analysis of the sampled gas is achieved by a mass spectrometer (MS). By moving the reactor stepwise with an automatic stage the full scan of the packed catalyst bed is obtained, with gas concentration and temperature profiles recorded. The reaction used to validate the apparatus was the CO oxidation over a 1wt% Pt/Al2O3. The invasiveness of the probes within the catalyst bed has been assessed using CFD calculations for the experimental conditions reported above. The resultant gas concentration profiles have also been compared with a micro-kinetic model for CO oxidation, based on the kinetic expressions reported previously [5].

Results and Discussion
CFD calculations have shown that, under the conditions reported herein, the presence of the probe was minimally invasive and as such had no undue influence on the reported results. Figure 2 reports the comparison of typical experimental results with the micro-kinetic model. The kinetic model simulated the concentration at set positions along the length of the catalyst corresponding to the positions set during the experiment, and an excellent agreement between experiment and simulation was observed.

Significance
A new in situ method has been developed to analyze the gas composition and temperature within a packed bed of catalyst for gas phase heterogeneously catalysed reactions. CFD calculations have determined the probe to be minimally invasive and an excellent fit was obtained between the experimental profiles and a micro-kinetic model. Hence, this technique has the ability to study catalysts under true reaction conditions. Furthermore, the innovative heating system employed will allow future coupling with spectroscopic techniques meaning that spatially resolved gas concentration profiles, temperature profiles and spectroscopic characterization can be obtained simultaneously.

References