**Al2O3-supported Co3O4 nano-catalysts for the preferential oxidation of carbon monoxide: A synchrotron-based *in situ* XAS study**

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**1. Introduction**

Cobalt(II, III) oxide (Co3O4) is a promising catalyst for the preferential oxidation of carbon monoxide (CO-PrOx) in hydrogen-rich gases, however, Co3O4 suffers from bulk reduction at elevated temperatures (> 200 °C), resulting in the formation of metallic Co (Co0). The formation of Co0 changes the conversion pathway of CO from oxidation to hydrogenation, forming methane (CH4) instead of carbon dioxide (CO2), which is an undesired outcome [1]. We attempted to stabilise Co3O4 nanoparticles on an alumina (Al2O3) support using two different anchoring methods, which resulted in two Al2O3-supported Co3O4 catalysts, *i.e.*, CAT 1 and CAT 2, where the strength of the Co3O4-Al2O3 interaction was different between the two prepared catalysts. The phase stability of the catalysts was monitored *in situ*, *i.e.*, during a CO-PrOx reaction, using synchrotron-based X-ray absorption spectroscopy (XAS) in Diamond Light Source, United Kingdom (session number: SP16006-1). This was later consolidated with kinetic data obtained *via* gas chromatography at the University of Cape Town, South Africa [1].

**2. Results**

Our results show that the Co3O4 nanoparticles in CAT 1 reduce to CoO and finally to Co0 to a greater extent than in CAT 2 (Figure 1), which can be attributed to the weaker interactions existing between Co3O4 and Al2O3 in CAT 1. The weak interactions also allow for high CO2 formation (*i.e.*, high CO2 outlet flow rates) over CAT 1, however, this catalyst also produces high amounts of undesired CH4 at relatively low temperatures than in the case of CAT 2 (Figure 2) due to the high amounts of unwanted Co0 formed in CAT 1 (Figure 1). In conclusion, *in situ* XAS was utilised successfully to confirm the phase changes occurring in each catalyst, and to help explain the differences between the catalysts in terms of their catalytic performance during CO-PrOx.

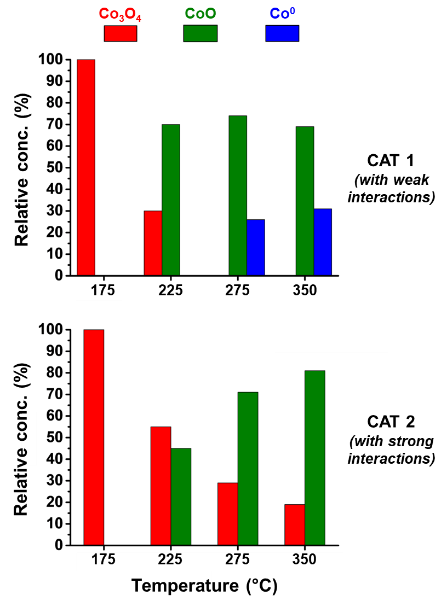
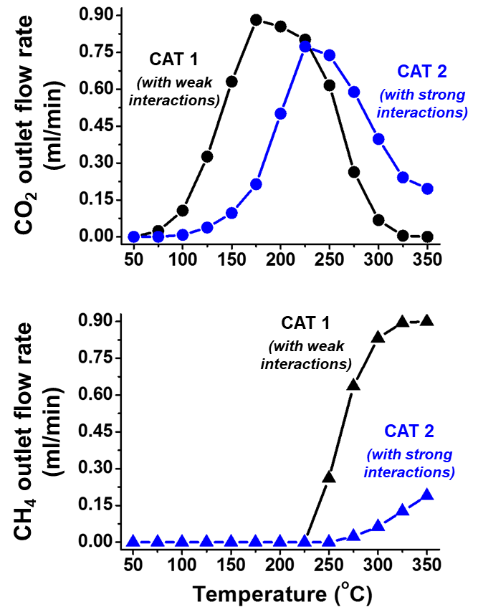
 

Fig. 1: Relative phase concentrations as a function of temperature as detected using *in situ* XAS during CO-PrOx.

Fig. 2: Outlet flow rates of CO2 and CH4 as a function of temperature during CO-PrOx.

**3. References**

[1] T.M. Nyathi, N. Fischer, A.P.E. York, D.J. Morgan, G.J. Hutchings, E.K. Gibson, P.P. Wells, C.R.A. Catlow and M. Claeys. *ACS Catal*. **9** (2019) 7166.