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ABSTRACT:

Temperature-Programmed Reduction (TPR): a Powerful Tool of Investigation in Heterogeneous Catalysis. Trick or Reality?

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Since its introduction around the mid-1970s, it is now well established that Temperature-Programmed Reduction (TPR) is a powerful investigation tool in heterogeneous catalysis [1]. TPR allows for the exploration of complex phenomena in catalysis, such as: i) metal-support interactions, ii) the influence of specific phases on the reducibility of multi-component catalysts, iii) the reduction mechanism of the catalyst and iv) when coupled with an oxidation step, the catalyst's redox properties. However, if TPR experiments are performed under inappropriate experimental conditions, artifacts may appear in the TPR profiles, leading to erroneous conclusions [1]. To avoid such artifacts, it is essential to carefully select and appropriately combine the experimental parameters, including the total amount of reducible species in the sample (S_0), the total flow rate of the reducing stream (V_0), and the initial concentration of H_2 in the feed (c_0). To this end, a parameter K (or P, which also accounts for the heating rate) has been introduced [1].

Experimental evidence demonstrates that artifacts significantly perturb the TPR profiles of CuO, Cu_2O, and CuO-ZnO catalysts when inappropriate K values are used, thereby undermining the reliability of the results. Conversely, when proper experimental conditions are applied, TPR proves to be an exceptionally powerful tool. This is exemplified in a TPR study of the interaction between CuO and ZnO species in CuO-ZnO catalysts which also highlights the impact of the preparation method on their nature and reducibility. Finally, the TPR investigation unveiled previously unrecognized redox properties which, as it is largely accepted in literature, play a crucial role in CuO-ZnO catalysts, explaining why this system remains widely employed in methanol synthesis.

[1] G. Fierro, M. Lo Jacono, M. Inversi, P. Porta, R. Lavecchia, F. Cioci, J. Cat., 148, 709, 1994.