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Doping SBA-15 with Nickel Oxide by Freeze-Drying Impregnation

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Immobilization of transition metal oxides on a solid support by impregnation with solutions of metal salts is a common route in catalyst design. An inherent difficulty of such methods is that precipitation by solvent evaporation leads to undesirably large catalyst crystallites forming at the outer surface of the porous support. In this study we explore the potential of an alternative route, based on eutectic crystallization of the aqueous metal salts in the pores of ordered mesoporous silica [1] and removal of ice by freeze-drying. The so-called two-solvents method was used to selectively load the pore space [2], i.e., to avoid an excess layer of salt solution coating the outside of the silica particles. The resulting samples were frozen with liquid nitrogen and lyophilized, then slowly heated to 500℃ and calcined. A morphological and structural analysis of the formed nickel oxide species in the pores was performed and compared to samples prepared by conventional solvent evaporation.
The immobilized nickel oxide prepared by the new technique appeared in two morphologies: (a) crystallites of size somewhat smaller than the pore diameter of SBA-15 (7 nm); (b) material causing no XRD reflections but still detectable by energy-dispersive X-ray spectroscopy (EDX). Nitrogen sorption isotherms provided further information about the distribution of NiO in the pores: For samples containing NiO crystallites the pore-size distribution (psd) of the primary pores was strongly broadened towards lower diameters. For samples showing no crystallites the psd remained narrow but was shifted, indicating that NiO was forming a thin layer at the pore wall.

References

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Primary authors: Mr PRAUSE, Albert (Technical University Berlin); Dr MEISSNER, Jens (Technical University Berlin); Prof. SCHOMÄCKER, Reinhard (Technical University Berlin); Prof. FINDENEGG, Gerhard (Technical University Berlin)

Presenter: Prof. FINDENEGG, Gerhard (Technical University Berlin)

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