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Synthesis and characterization of novel intermetallic catalysts for hydrogenation of carbon dioxide to methanol

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Novel Ni₅Ga₃ and Pd₂Ga catalysts for CO₂ hydrogenation to methanol are prepared by impregnation of aqueous Ni-Ga or Pd-Ga solutions of metal nitrates into high surface area SiO₂, followed by drying, calcinations and reduction of the precursor in a H₂ flow. Steady state experiments are performed in a reactor at atmospheric pressure and stoichiometric CO₂/H₂ mixture, while reaction products are analyzed by gas chromatography. The results are compared to the highly optimized Cu/ZnO/Al₂O₃. The activity and selectivity of the novel catalysts is close to that of Cu/ZnO/Al₂O₃ and the equilibrium conversion to CH₃OH is found to be higher. XRD and XRF are used to investigate the phase and composition of the supported catalysts at the 5 stages of testing, i.e. after drying, calcination, reduction, CO₂ hydrogenation, rapid ageing. SEM and TEM images of the exact same locations are acquired after each of the 5 stages to monitor particle formation and investigate particle size and distribution. In the same way, model systems consisting of Ni₅Ga₃ and Pd₂Ga nanoparticles supported directly on a silica membrane of an Au TEM grid are also characterized by SEM and TEM after each of the 5 stages. Nanoparticles formation is observed after calcination. Size distribution analysis reveals that the Pd₂Ga nanoparticles have a diameter of 5-10 nm which does not change after reduction, methanol synthesis and rapid ageing. Furthermore, *in situ* ETEM is used to monitor the development of the materials system during synthesis and reaction.