

Highly Efficient Silver Niobium Alumina Catalyst for the Selective Catalytic Reduction of NO by *n*-Decane: Detailed Experimental Part

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5 Catalyst Preparation

Preparation of Ag_{CAL823}

0.0664 g of silver chloride and 6.400 g of aluminium chloride are added, under inert atmosphere, to a solution of dichloromethane (10 cm³) and isopropyl ether (10.2 cm³).
10 Then the reaction mixture was charged into a 130 cm³ stainless steel autoclave equipped with a glass liner. The autoclave is then placed into the drying oven and kept at 383 K for 6 days. After this ageing, the gel was washed with CH₂Cl₂ and dried under vacuum firstly at room temperature
15 and then at 393 K. The sample was calcined at 823 K under air flow for 8 hours.

Preparation of AgNb_{CAL823}

0.0664 g of silver chloride, 0.127 g of niobium chloride (V)
20 and 6.234 g of aluminium chloride are added, under inert atmosphere, to a solution of dichloromethane (10 cm³) and isopropyl ether (10.1 cm³). Then the reaction mixture was charged into a 130 cm³ stainless steel autoclave equipped with a glass liner. The autoclave is then placed into the drying oven
25 and kept at 383 K for 6 days. After this ageing, the gel was washed with CH₂Cl₂ and dried under vacuum firstly at room temperature and then at 393 K. The sample was calcined at 823 K under air flow for 8 hours.

30 Preparation of Ag_{HDT1023} and AgNb_{HDT1023}

1g of AgNb_{CAL823} or 1g of AgNb_{CAL823} is set on a porous fritte of a U tube quartz reactor in which an air flow of 50 cm³/min passes through. Then, the reactor is heated at 1023 K with a ramp of 6 K/min and kept at this temperature for 16 hours and
35 then cooled to room temperature. The injection of H₂O_{liq.} (0.0041 cm³/min), by a syringe-pump, is started during the ramp of temperature when the temperature of the sample is around 373 K. The injection of water is stopped during the cooling of the furnace once the temperature is below 673 K.

40 Catalytic Test Reaction

The SCR of NO by *n*-decane was performed in a flow reactor operating at atmospheric pressure. An aliquot (0.040 g) of the powdered catalyst was placed in a reactor and pretreated in
45 situ at 823 K for 1 h in air. After cooling to 473 K, the reaction was performed using a gas mixture containing 0.04 vol% NO (purity >99.995%), 0.02 vol% *n*-decane (purity >99.5%), 2.5 vol% H₂O (purity >99.5%) and 8 vol% O₂ (purity >99.995%), the balance with helium. The total flow
50 rate was 100 cm³/min (space velocity: 150,000 h⁻¹) and the temperature was varied from 473 to 823 K (ramp: 5 K/min).

The composition of the effluents was monitored continuously by sampling on line to a quadrupole mass spectrometer (Pfeiffer Vacuum) equipped with Faraday and SEM detectors
55 (0–200 amu) and following the masses 28, 30, 44, 46, and 57. The possible N₂O and CO formations were checked by analysing the outlet gas with a two module micro gas chromatograph (CP-4900 Micro-GC Varian), each module being equipped with a thermal conductivity detector (TCD).
60 The first module has a 5 Å molecular sieve column, allowing the analysis of O₂/N₂/CO/CH₄ and a back flush system to send the heavy products and CO₂ towards the second module. The latter has a PORAPLOT Q column in order to separate CO₂ and N₂O. With the CP-4900 μGC, a sample analysis of the gas
65 phase, which injection is performed through a system capillary/micro-pump, can be realised every 20 K