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Supported catalyst for autothermal reforming of methane

WHEC 16 / 13-16 June 2006 – Lyon France 4/6 The composition of the product gas (dry gases) is shown in Table 1. Hydrogen concentration (63 - 65 %) is higher compared to data published by Lee et al. (42 % H₂) [15], as nitrogen dilution was avoided. It should be noted that the CO concentration of 5 % is comparable to the low. This can be attributed to the high steam-carbon ratio (H₂O/CH₄) of 3, which increases the activity of changing water gas and the controllable low temperature. Low carbon monoxide is beneficial with regard to the fuel gas refinery for PE fuel cell applications [17]. Table 1: Composition of dry gas. Input power/(kWth) WHSV (h⁻¹) GHP (h⁻¹) CH₄/CO₂(%) CO (%)H₂(%) 1.09 119 14095 10.616 5.2 65.40 97.105 12474 11.118 65. 2 65.00 84 91 133 49 11 91.94 9.4 64. 20 73 90 9474 12.919 14.6 63.3 Conditions of action: pabs = 0.21 MPa, O/C = 0.89, S/C = 2.9. The results obtained for different reaction conditions are summarized in Table 2. The efficiency of the autothermal reform (RE) can be defined as the ratio between the lower heating value (LHV) of the hydrogen produced and the LHV of the fuel used [9]. Because RE depends on methane conversion, a specific reform efficiency (RE* = RE/XCH₄) has also been calculated. Table 2: Results of autothermal catalytic partial oxidation on a monolith catalyst coated with Ru. Input power/(kWth) WHSV (h⁻¹) GHSV(h⁻¹) O/C S/C CH₄(%/YCO(%) YCO₂(%/YH₂(%) RE(%) RE*(%) 0.84 94 1.116 0.893.0063 3.10 6 46 34.8 9 42.273 70.84 92 11031 0.793.0257 9.9 44.5 48.5 43.975 80.84 91 10995 0.693.0551 8.6 68 41.1 4.41 39.977 70.84 112 13349 1.003.8476 6 11.9 54 59.1 53.475 60.84 111 13243 0.893.8656 9 6.50 57.4 40.475 70.84 108 13007 0.793.8259 7 8.0 46 95.0 74.095 80.84 107 12949 0.693.8351 9 5.8 42 0.40 4.0 39.176 71.09 119 14095 0.892.9070 3.1 4.6 51.7 60.8 55.078 20.9 107 12474 0.892.9667 14.2 50.7 53.476 60.84 91 10824 0.892.8967 7 13 25.1 58.2 52.677 70.73 108 9474 0.892.9165 3 12.3 51.7 57.0 51.578.9XCH₄: Methane conversion, YCO, YCO₂, YH₂: Yields CO, CO₂, H₂, S/C = H₂O/CH₄, O/C = 2°O₂/CH₄RE: Reform efficiency (RE = LHV of H₂ product / LHV methane supplied) x 100, RE*: Specific reform efficiency (RE* = RE / XCH₄) x 100. All experiments were conducted at a total pressure of 0.21 MPa. C, H, O balances: 0.96 - 1.02. The production of methane synthesis gas is an important process of conversion of natural gas, one of the most abundant and cleanest fossil fuels, into high-quality value-added liquid products (Gas-to-liquid technology, GTL). Steam reforming (SRM) was the preferred technology for industrial production of methane synthesis gas to produce ammonia or methanol (Rostrup-Nielsen et al., 2002; Navarro et al., 2007a,b). However, SRM is a highly energy-intensive process [Equation and more energy-efficient alternatives are sought to produce synthetic gas. CH₄ + H₂O → CO + 3H₂ ΔH_{298K}° = +206 kJ/mol] (1) Among alternatives, alternatives, for the production of methane synthesis gas (CPOM) is more energy efficient because it has rapid kinetics and is exothermic, thus avoiding the need for large reactors and large amounts of overheated steam (Bharadwaj and Schmidt, 1995). In addition, OPC stoichiometry [Equation 2] produces a synthesis gas with a H₂/CO ratio of 2:1, which allows its direct use for the synthesis of methanol or Fischer-Tropsch without further adjustments. Partial catalytic oxidation of methane in singas is a challenge due to the difficulty of controlling the selectivity of the reaction to total combustion. Several studies have been carried out in the literature to describe the reaction mechanism involved in CPOM and two reaction mechanisms have been proposed (Dissanayake et al., 1991; Hickman and Schmidt, 1992): the direct mechanism, in which CH₄ and O₂ react on the surface of the catalyst to produce CO and H₂, and the combustion reform mechanism, in which CH₄ and O₂ first react to the H₂O and CO₂ form and then, with excess methane, the dry reforming of CO₂ [Equation 3] and the steam reforming [Eq.4] produce final CO and H₂. Due to excessive temperature gradients at a high conversion rate, the exothermic nature of the reaction and rapid deactivation due to carbon deposition on the catalyst surface, the development of effective catalysts for CPOM was a challenge. NPs of noble metals (Pt (Ji et al., 2001), Rh (Ruckenstein and Wang, 1999; Puolakka and Krause, 2007), Ru (Ashcroft et al., 1990), Pd (Veron et al., 1990) and non noble metals (mainly Ni and Co (Wang and Ruckenstein, 2001)) supported by various oxide substrates were studied in the CPOM reaction. Nickel is one of the most used active phases for CPOM (Miao et al., 1997; Ostrowski et al., 1998; Takehira et al., 2004; Wang et al., 2004). Compared to noble metals, nickel is cheap, but suffers from deactivating on the flow as a consequence of several processes, such as sintering, carbon deposition (Claridge et al., 1993) or solid solutions of nickel with substrate. From the large working body developed on the reaction of the CPOM, it is clear that the activity and stability of nickel catalysts depend on both the active phase and the support. The size of the metal particles has been shown to be an important factor for the initial intrinsic activity and the deactivation rate, both decreasing with the increase in the size of the active metal particles (Barber and Marecot, 1996; Barber, 1997). The influence of support on the performance of Ni-based catalysts has been widely studied in literature (Tspounaris et al., 1998). Non-reducible Al₂O₃ is one of the most studied oxides as support for Ni catalysts due to its thermal stability and high ability to disperse Ni nanoparticles (NP) (Hu and Ruckenstein, 1998; Ostrowski et al., 1998; Zhang et al., 2000). However CPOM reaction is limited due to the relatively high deactivation of PNMs formation of coke deposits (Lu et al., 1998). Magnesium oxide is another widely studied non-reducible medium to disperse stable Ni particles (Choudhary et al., 1998a; Ruckenstein and Hu, 1999; Nishimoto et al., 2004). In this case, the formation of a solid solution between nickel and magnesia (Mg_{1-x}Ni_xO) allows only the reduction of a small fraction of nickel that remains in close interaction with the basic MgO substrate, favouring this structure for the production of syngas by high-activity CPOM (Requies et al., 2005). Lanthan oxide was also used as a support for Ni catalysts (Tspounaris et al., 1998; Nishimoto et al., 2004). For Ni/La₂O₃ catalysts, good stability has been reported and an enhanced metal support interface has been assigned, as nickel NPs are decorated with La₂O₃/CO₂ species that promote coke gasification. Reducible supports (CeO₂, ZrO₂) have also been studied as active and stable nickel particle dispersion systems for CPOM. CeO₂ is known for its ability to improve the dispersion and stabilisation of small nickel NPNs and for its high oxygen storage/transport capacity, which allows the continuous removal of carbonaceous deposits from active sites (Choudhary et al., 1993; Diskin et al., 1998). In addition, under reduced conditions, the effect of SMSI (Interaction with the support of strong metals) could be observed on the wax, which in turn affects the stability and activity of nickel-dispersed particles (Trovarelli, 1996). Zirconia is another medium that shows interesting properties for the dispersion of active and stable NPs in NPs. However, the application of ZrO₂ for the CPOM reaction is questionable, as this support decreases the availability of oxygen participating in CPOM directly to the synthesis gas, leading to a decrease in activity (Pompeo et al., 2005). Incorporating a second metal into Ni-based catalysts is a common practice designed to improve catalytic stability. The beneficial effect of adding small amounts of precious metals, such as Ru, Pt, Pd, Ir and Rh, to a Ni catalyst, has previously been demonstrated (Tomishige et al., 2002). Rh is one of the most promising metals (Tanaka et al., 2010a). The improvement was explained in terms of H-spillover from noble metal to non-noble metal, helping the non-noble metal surface to remain metallic (Chen et al., 1997). After mentioned above, Ni effective catalysts for the CPOM reaction require control over the electronic and structural properties of np nickel, which could be achieved by carefully selecting the support and synthesis procedure. In this context, the main objective of this paper is to study the influence of different media (Al₂O₃, CeO₂, La₂O₃, MgO, ZrO₂) with different textural and surface properties (basic, reducibility) on the activity, and stability of Ni-based catalysts for CPOM reaction at atmospheric pressure. Two different synthesis methods were used: (I) impregnation on different different (II) solid reaction. The influence of a small amount of Rh on the reducibility and reactivity of catalysts was addressed. Using different characterization techniques, we will establish structure-activity relationships that indicate the properties of the catalyst that determines its reactivity and thus give us a way to improve the catalytic performance of systems. Ni catalysts were characterized by X-ray diffraction, nitrogen adsorption-desorption, programmed temperature reduction, X-ray photoelectron spectroscopy, oxygen-pulse chemisorption, electronic transmission microscopy and Raman spectroscopy. The evolution of the structure and morphology of catalysts is reported and correlated with its catalytic performance. Catalyst preparation materials and methods Prepared by impregnation of Ni commercial media (5% weight) catalysts supported on commercial Al₂O₃ (Johnson Matthey, 99.97%), CeO₂ (Johnson Matthey, 96%), La₂O₃ (Fluka Chemie, 99.98%), MgO (Fluka Chemie, 99.98%) and ZrO₂ (Johnson Matthey, 99.5%) prepared by wet impregnation. First, the supports were thermally stabilized by calcination at 850°C over 3 hours. They were then sited between 212 and 425 μm. The corresponding amount of Ni(NO₃)₂·6 H₂O (Aldrich Chemie) was dissolved in distilled water. Each holder has been impregnated with this solution by rotary evaporation (70°C, 1 h). Then the obtained solid was dried at 110°C for 2 hours and charred at 500°C for 3 h. Catalysts prepared by the solid reaction RhNi catalysts (0.1% Rh, 10% Ni, weight), supported by Al₂O₃, CeO₂, La₂O₃, MgO, and ZrO₂, were prepared by mixing and grounding in a mortar and adding the corresponding quantities of Ni(NO₃)₂·6H₂O (test 98.5%, Aldrich) and support oxides (Mg(NO₃)₂·6H₂O (Schuau, 99%), Ni(NO₃)₂·6H₂O (Johnson Matthey, 99.9%), Ni(NO₃)₂·6H₂O (Sigma Aldrich), Ce(NO₃)₃·6H₂O (Matthey, 99.9%) and Zr(NO₃)₂·6H₂O (Matthey, 99.9%) respectively). Since the amount of Rh is varying small, the reduction of this element was based on the dissolution of the corresponding amount of dicarbonyl (acetylacetonate) Rh(I) (Rh(CO)₂zaccac) (Sigma Aldrich, purum) in acetone and on the addition of this solution in a dropwise way to the mixture of nickel nitrate powder and suitable support carbon nitrate, eliminating it well with a spatula. Then each mixture was dried at 110°C for 2 hours and charred at 500°C for 3 hours. Finally, the samples were ground and sifted between 212 and 425 μm. Characterization Techniques The X-Ray Diffraction XRD models were recorded on a Seifert 3000 powder diffractometer using Cu radiation (λ = 0.15418 nm) generated at 40 kV and 40 mA. Scans were recorded at a rate of 0.02°/s for diffraction angles from 10 and 90°. The properties of adsorption-desorption isotherms were assessed by adsorption-desorption N₂ of samples recorded at liquid nitrogen temperature with a Micromeritics ASAP2000 apparatus. The samples were degassed at 150°C below Overnight. Certain areas were calculated by applying the BET method. Prior to the reduction experiments, the samples (approximately 30 mg) were heat-treated under air to 300°C to remove moisture. TPR profiles were obtained by heating samples at a flow rate of 10% H₂/Ar (50 mL/min) from 25 to 800°C with a linear rate of 10°C/min. Oxygen chemisorption capacity The chemisorption capacity was determined by chemisorption with o₂-pulse. First, the catalyst sample (60–70 mg) was inserted into a U-pick reactor and heated to 500°C in argon, maintaining this temperature for 15 minutes. The temperature was then lowered to room temperature and the sample was reduced under a H₂/Ar (10% H₂) to 750°C in 60 minutes. Eventually, the carrier gas was changed to helium, and once the baseline was stabilized, the O₂ pulses were injected until the peaks detected by O₂ showed the same area. X-Ray Photoelectron Spectroscopy To extract information about the chemical state and composition of Ni-supported samples, XPS measurements were purchased using a monochromated X-ray source (Al Kα, 1486.6 eV) operating at 200 W and a hemispheric electron analyzer (Phoibos 100, SPECS GmbH). The high-resolution data were obtained with a passing energy of 18 eV. CasaXPS software was used to analyze the data. The Photoelectron X-ray spectra of rhni samples were recorded on a VG Escalab 200R spectrometer equipped with a hemispheric analyzer and Mg Kα (1253.6 eV) X-ray source (12 kV and 10 mA). The powder samples were degassed overnight before being transferred to the analysis chamber. The samples were connected to the analyzer by attaching the samples using conductive adhesive tape. The data were recorded with XPS peak software. The spectra were decomposed with the smallest squares mounting routine using the Gaussian/Lorentzian functions after decreasing Shirley's background. The Raman spectra of the samples were recorded in the air under ambient conditions (being samples hydrated by air humidity), using a single renshaw monochromator inVia 100 system equipped with a thermoelectric cooled CCD detector and Super-Nohz holographic filter. The samples were excited at 535 nm (1800 lines/mm). Catalytic activity tests The catalytic behaviour of the various catalyst precursors for partial oxidation of methane in singas has been studied under atmospheric pressure at 750°C using a fixed stainless steel bed reactor (length = 150 mm, inside = 9 mm) placed inside a hinged furnace. Catalysts (100 mg) were subjected to a pretoration below 50 mL/min of H₂N₂ (10% H₂, molar) for 1 h for Samples supported by RhNi before the reaction. The reactors were then fed to the reactor (22.6 mL/min Ni₂, 6 mL/min O₂ and 12 mL/min CH₄). The reaction stream was analysed online by gas chromatography (Varian 45-GC) with a thermal conductivity detector, equipped with a molecular sieve 5A (P77538) to separate H₂, N₂, CH₄ y CO and a PorAQBOND Q column (P7354) to separate CO₂ and H₂O with a thermal conductivity detector. Argon was used as a carrier gas to increase sensitivity for H₂ detection. Methane conversion, CO selectivity, H₂ yield and H₂/CO ratio were defined as follows: CH₄conversion (%)= molar flow CH₄ (input)-molar flow CH₄ (output)/molar flow CH₄ (input) · Molar yield H₂ (output) · Molar yield H₂ (%)= Molar yield H₂ (output) · Molar flow CH₄ (input) · 100 CO selectivity (%)= molar flow CO (output)/molar flow CH₄ (input) · 100 H₂/CO molar ratio)= H₂molar flow (molar flow CO) exit) · Methyl results and discussions Catalysts were analysed before reaction (section Physico-chemical characterization of calcinated and reduced samples) of XRD, Adsorption-desorption N₂, TPR and XPS and after reaction (section Physico-chemical characterization of catalysts used) of XRD, Raman spectroscopy and XPS. Finally, the results of the activity tests are presented and discussed (activity tests). XRD models of our calcinated and reduced samples Structural properties X-ray diffraction (before the reduction) samples shown in Figure 1 (line 2). As regards the catalysts prepared by impregnation, the diffraction lines are assigned mainly to the corresponding media. In the case of the catalyst supported by lanthana, the formation of the LaNiO₃ phase cannot be eliminated. For the catalyst supported on magnesia, the corresponding MgO diffraction lines and the solid Ni-Mg-O solution cannot be distinguished because of their strong overlap (Arenia et al., 1996). The formation of the solid solution Ni-Mg-O is due to the relatively high calcination temperature used in the preparation of the catalyst. As regards the zirconium-supported catalyst, for this method of preparation, zirconium crystallizes in the tetragonal phase. Rh reflections were not observed, so it was expected from the very small crystalline size. With regard to the presence of ceria/lanthan nickel species, only in the case of the catalyst supported on CeO₂ it was possible to observe a small diffraction line of the plane (2 0 0) of NiO. The crystallinity of the catalysts has changed considerably; higher for the MgO-supported catalyst and lower for the Al₂O₃-supported catalyst. The XRD diagram of this last catalyst indicates its amorphous nature, as the calcination temperature was not high enough to obtain a crystalline structure of the alumina. The temperature range required for the gamma phase of alumina is 600–875°C (Sathayaseelan et al., 2013). The Scherrer equation has been applied to these X-ray diffraction diagrams, and the average dimensions of the different crystalline phases are shown in Table 2. For these calcinated samples, prepared by solid reaction, the average size of the range between the different media did not change as much as that which occurred for the calcined samples prepared by impregnation of the commercial media. In addition, it is worth noting that the crystalline domains of NiO, if observed, were smaller than those for samples prepared by impregnation of commercial media (Table 1). Table 2. Average domain dimensions for calcined samples (before reduction) prepared by impregnation (determined by the Scherrer equation). For Ni/MgO and Ni/Al₂O₃ catalysts, the peaks corresponding to nickel oxide overlap with those corresponding to the respective supports. In the Ni/La₂O₃ catalyst, the built-in NiO reacts to the LaNiO₃ phase, the built-in NiO reacts to the LaNiO₃ phase (00-01-075-1519); • NiO (00-01-01-1258); • CeO₂ - (00-049-1415); □: Table 1. Average domain dimensions for calcined samples (before reduction) prepared by impregnation (determined by the Scherrer equation). For Ni/MgO and Ni/Al₂O₃ catalysts, the peaks corresponding to nickel oxide overlap with those corresponding to the respective supports. In the Ni/La₂O₃ catalyst, the built-in NiO reacts to the LaNiO₃ phase, the built-in NiO reacts to the LaNiO₃ phase (00-01-075-1519); • NiO (00-01-01-1258); • CeO₂ - (00-049-1415); □: Table 1. 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