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Research Article

Effect of Nickel on CO Conversion Over Cordierite-Supported Bi-Metallic Pd-Ni Catalysts

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ABSTRACT

This study focused on the preparation and characterization of Pd-Ni bimetallic catalysts supported on cerium-coated cordierite for carbon monoxide oxidation in a batch system. Using techniques such as XRD, FTIR, SEM/EDS, and BET, we confirmed successful metal loading and strong interactions between the metals and the support. The BET results showed that catalysts with balanced Pd and Ni ratios, especially Ni50, had larger surface areas, likely due to synergistic effects. Catalytic tests carried out at 300 °C demonstrated that these bimetallic catalysts performed nearly as well as the pure Pd catalyst in converting CO, while also benefiting from the inherent activity of the cerium-coated support. Overall, this study suggests that combining Pd with Ni can reduce the amount of precious metal needed without sacrificing efficiency, pointing to a more cost-effective solution for emission control applications.

1. Introduction

Nowadays, air pollution caused by motor vehicles has become a serious environmental problem due to the release of toxic gases such as carbon monoxide (CO), hydrocarbons (HC) and nitrogen oxides (NO_x) into the atmosphere. Catalytic converters are widely used to convert these harmful gases into less harmful compounds before they are released into the atmosphere [1]. Catalytic converter technology plays a critical role in converting exhaust gases into harmless compounds. The success of this conversion depends largely on the type and nature of the catalyst used. The most commonly preferred catalysts in conventional converters were precious metals such as platinum (Pt), palladium (Pd) and rhodium (Rh) [2]. Although precious metals such as palladium (Pd) exhibit high catalytic activity in traditional catalytic converters, alternative systems need to be developed due to their high costs and limited availability [2].

In this context, bimetallic catalysts, especially the use of Pd together with the transition metal were promising in terms of both economics and catalytic performance [3, 4]. The metal that can potentially increase Pd activity was nickel (Ni). Recent studies on three-way catalyst (TWC) systems have revealed the role of transition metals, especially nickel, in enhancing catalytic activity. The strong interactions formed by nickel with ceria–zirconia-based oxide structures improve the redox properties at low temperatures, allowing lower light-off temperatures to be achieved during the cold start-up phase. In addition, the effect of nickel on catalytic behavior appears to be largely dependent on its interaction with the support material. Therefore, nickel is considered as an important supporting element in Pd-based systems [5, 6]. In bimetallic systems, the temperature at which the system is studied plays a critical role in determining the extent of metal-support and metal-metal interactions. For instance, one study demonstrated that the interaction between nickel and an alumina support is significantly enhanced at elevated

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temperatures [7]. The synergistic interaction between palladium (Pd) and nickel (Ni) in bimetallic catalysts has been widely associated with enhanced catalytic activity, particularly when compared to their monometallic counterparts [8]. This enhancement becomes especially pronounced when the total metal loading exceeds 7 wt%, a condition under which direct metal–metal interaction has been reported [9].

The choice of support material for metal loading has a significant effect on catalyst performance as well. Cordierite stands out as an ideal support material for automotive applications thanks to its low thermal expansion coefficient and high temperature resistance [10, 11]. In this direction, cordierite was widely used as the preferred ceramic support material, especially for automotive applications. Thanks to its high mechanical stability and thermal resistance, the active phases coated on cordierite show long-term stability. The combined use of Pd and Ni on cordierite support material can offer significant advantages in reactions such as oxidation of CO and hydrocarbons due to their synergistic interactions. Therefore, understanding the nature of the Pd-Ni interaction and determining the key factors affecting it is important for the development of more effective Pd-Ni based catalytic systems.

It was seen that the studies examining Pd-Ni bimetallic systems on cordierite were limited in the literature. In this study, cordierite supported Pd-Ni bimetallic catalysts were prepared at different ratios and their effects on carbon monoxide conversion were systematically investigated in batch reactor. The study aims to evaluate the effect of Pd-Ni ratios on catalytic activity and to contribute to the lack of knowledge in this area.

2. Materials Methods

2.1. Catalyst preparation

A commercial cordierite honeycomb cordierite ($MgO–Al_2O_3–SiO_2$), measuring 20 mm × 25 mm × 25 mm with a cell density of 200 cpsi, was used as the catalyst support. Prior to metal deposition, the support was activated in 1 M HCl at 90 °C for 8 h. Metal catalysts were prepared via the impregnation method after cerium coating. The details on cerium coating were given elsewhere [12]. Metal salts ($PdCl_2$ and $NiSO_4.6H_2O$) corresponding to a total metal loading of 1 wt.% were dissolved in a mixture of 0.8 mL 0.5 M HNO_3 and 50 mL deionized water to enhance solubility and ensure uniform distribution. The cordierite supports were then introduced into this solution, and the mixture was stirred for 1 h at room temperature.

Catalysts with varying Pd:Ni ratios (100:75:50:25:0) were synthesized and labeled as Ni0, Ni25, Ni50, Ni75, and Ni100, respectively. Reduction of the metal ions was carried out by the dropwise addition of 10 mL of 0.20 M $NaBH_4$. Following reduction, the solvent was evaporated under heating and continuous stirring. The resulting materials were dried at 120 °C for 4 h and calcined at 500 °C for 16 h. Finally, thermal reduction was performed at 350 °C under a argon-hydrogen (95:5) atmosphere in a tubular furnace to ensure complete metal reduction on the support surface.

2.2. Catalyst characterization

The catalysts were subjected to X-ray diffraction examination using a PANalytical Powder Diffractometer operating at 45 KV and equipped with a Cu kα source (l = 1.54 A). We obtained the scans from 2θ = 10–80°. Pd and Ni particles were identified on the carbon support using the scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM/EDX) technique. A PerkinElmer Optima 7000 DV was used for inductively coupled plasma (ICP) analysis in order to quantify Pd and Ni particles on the support. FTIR analyses were carried out to determine the surface functional groups and support-metal interactions of the catalysts. Spectra were recorded in the 4000–400cm^{-1} range in ATR (Attenuated Total Reflectance) mode using a Jasco FTIR spectrometer. The specific surface area of the samples was determined using the Brunauer–Emmett–Teller (BET) method with nitrogen adsorption at 77 K, and the BET surface area was calculated using a Micromeritics TriStar Plus II instrument.

2.3. Catalyst performance

The carbon monoxide conversion experiments were carried out in a batch reactor (Uniterm 100 mL). The 2.5 g catalyst was placed in the reactor and sealed, after that purged the air with Argon. The reactor was pressurized with CO at 1.0 bar. The temperature used for conversion was 300 °C at 250 rpm stirred for 30 min. The details on experiments were given elsewhere [12]. A thermal conductivity detector (TCD)-equipped online gas chromatograph (Agilent GC/TCD 7842) was used to analyze the compositions of the output gases. The Supelco 60/80 Carboxen 1000 columns were used in the instrument. For the GC analysis, the following temperature program was utilized: holding at 35 °C for 5 minutes, heating at a rate of 20 °C per minute to 225 °C, and then holding at this temperature for 10 minutes.

3. Results and Discussion

3.1. Evaluation of catalyst

Cordierite ($Mg_2Al_4Si_5O_{18}$) is a well-defined crystalline material used as a ceramic and catalytic carrier. When cerium (usually in the form of CeO_2) was loaded onto it, some differences in the XRD (X-ray diffraction) pattern can be observed (Fig 1a). The XRD peak intensity of the main phase was decreased by coating the cordierite surface following Ce loading.

XRD analyses were performed to determine the metallic phases and possible alloy formations in the catalysts (Fig 1b). Although the main phase diffraction peaks of cordierite slightly decreased upon cerium loading, no significant changes were observed in the XRD patterns of the resulting catalysts due to the low metal loading (~1%) on the surface. The increase in the intensity of cordierite diffraction peaks with higher Pd loading may be attributed to enhanced X-ray reflectivity due to the presence of Pd species on the surface, improved crystallinity of the cordierite phase induced by Pd deposition, or changes in surface morphology facilitating better diffraction signal collection [13, 14].

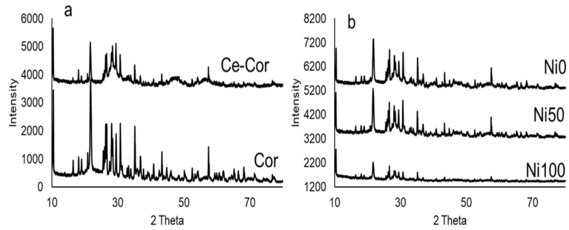

Figure 1. XRD spectras of cordierite (a) and catalysts (b)

Ni and Pd were loaded onto a cerium-coated cordierite support at different ratios, and structural analyses of the resulting catalysts were performed using Fourier Transform Infrared Spectroscopy (FTIR). FTIR measurements were used to determine the interactions between the support material and the metal species and to determine the functional groups on the surface. In the FTIR spectra shown in Figure 2a, the characteristic bands observed particularly in the 700–500 cm^{-1} range correspond to metal–oxygen (M–O) vibrations. The prominent peak observed around 670 cm^{-1} indicates Ce–O bonds and demonstrates the successful application of the cerium oxide coating [15]. Furthermore, the weak bands around 460–480 cm^{-1} can be attributed to the Si–O–Si and Al–O–Si vibration modes in the cordierite support structure.

Small shifts and intensity differences were observed in this region depending on the Ni and Pd loadings [16-18]. This indicates that the metal species interacted with the CeO_2 matrix and caused structural changes on the surface. In CeO_2 supported Ni and Pd based catalysts, the shifts and intensity differences observed in the range of 400–800 cm^{-1} showed that metal species interact with the CEO_2 matrix and cause structural changes on the surface. In our study, cerium nitrate was used as a cerium source. A nitrate N-O stretching centered at 1350 cm^{-1} [19-21]. The absence of bands belonging to residues such as nitrate (NO_3^-) in the spectra indicates that the prepared catalysts had clean surfaces.

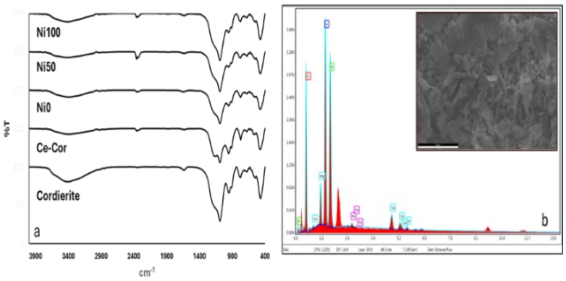

Figure 2. FTIR spectra of samples (a), SEM-EDS of Ni0 catalyst

BET surface area measurements revealed that the metal composition loaded onto the support material has a significant effect on the surface properties of the catalyst. The Ni50 sample had the largest surface area, measuring 13.3754 m²/g, when various Ni-Pd ratios were deposited onto a cerium-coated cordierite support. The sample with only Pd (Ni0) had a surface area of 6.3879 m²/g, whereas the sample with only Ni (Ni100) had the lowest surface area, at 4.2271 m²/g. This suggested that the combined use of Ni and Pd created a synergistic effect, creating a more open and porous structure on the support surface. Similarly, it has been reported in the literature that bimetallic catalysts can provide higher surface areas and more active centers compared to monometallic catalysts [22, 23]. The Ni-Pd combination, in particular, is known to improve catalytic properties through both structural and electronic interactions [24]. The decrease in surface area observed with increasing Ni content can be explained by nickel particles partially blocking the pores on the support surface or agglomeration, reducing the surface area. Indeed, it has been reported in the literature that Ni can form larger particles on the support surface at higher temperatures, limiting the surface area [25].

3.2. Catalyst activity

The catalytic activity of the synthesized catalyst was evaluated through the oxidation of carbon monoxide. Cordierite-supported bimetallic PdNi catalysts were developed in the present study. The Ni metal was taken into consideration when naming the catalysts. Ni100 was the name of the catalyst that had no Pd metal, while Ni0 was the name of the catalyst that contained all Pd. As indicated in Fig 3, the presence of noble metals strongly enhanced carbon monoxide conversion. The cordierite support material used in this study was coated with cerium (Ce) before metal loading in the catalyst preparation (Ce-Cor). A very high CO conversion rate of 87.88% was obtained in CO oxidation experiments performed without any metal (Pd or Ni) loading on the cerium-coated cordierite. This result clearly showed that the support material itself has significant catalytic activity. This high activity was mainly due to the strong oxygen storage and release capacity (OSC) of cerium based on the $Ce^{4+} \rightleftharpoons Ce^{3+}$ redox couple. The CeO_2 phase facilitated the conversion of molecular oxygen into the active form by creating oxygen vacancies on its surface, thus supporting the oxidative transformation of CO [26, 27].

Thus, active oxygen species were continuously provided in the reaction medium, enabling CO molecules to effectively react with oxygen on the surface. The redox capability of the CeO_2 support created the oxidative environment even though, as mentioned in the experimental section, no external oxidant other than CO was added during the reaction. The CeO_2 facilitates oxygen transport to the active metal sites by acting as an oxygen reservoir through its Ce^{4+}/Ce^{3+} redox cycle [28]. Oxidation processes were helped by stabilizing chemical intermediates and providing lattice oxygen. The interaction between CeO_2 and Pd–Ni particles might be an essential point for oxygen overflow, improving catalytic activity. However, despite the high performance of the cerium-coated cordierite support, the presence of transition metals such as Pd and Ni is critical, especially in terms of increasing the reaction rate, lowering the activation energy and providing activity at lower temperatures.

The catalyst with only Pd (Ni0) exhibited the maximum activity with a CO conversion of 99.88%, According to this result, Pd had strong catalytic activity in the CO oxidation reaction, and cordierite-supported systems may successfully sustain this activity. The catalyst Ni25 showed a performance very close to the pure Pd catalyst with a conversion rate of 99.80%. This implied that adding small levels of Ni did not significantly affect the catalytic activity of Pd and, in certain cases, may even have a synergistic effect [29, 30]. It was possible that Ni electron-donating ability enhanced the reaction kinetics by balancing the CO adsorption and desorption on Pd.

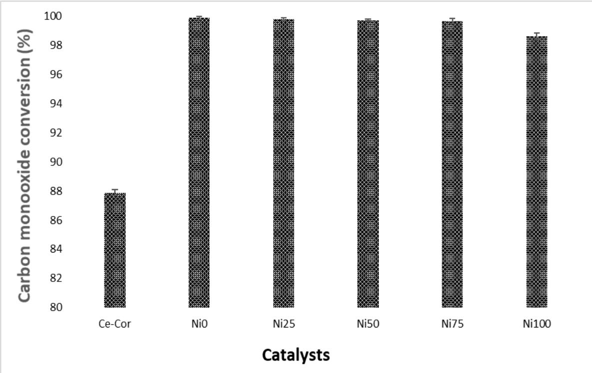

Figure 3. The percentages carbonmonoxide conversion of catalysts

The catalyst containing 50% Ni (Ni50) again exhibited a very high performance with a conversion rate of 99.71%, but a small decrease in catalytic efficiency was observed with the decrease of the Pd content. Although this conversion indicated that Pd and Ni continue to work together, the Pd content shouldn't be lowered below the essential range. According to Liu et al. investigation on the catalytic performance of Pd–Ni nanoalloys in CO oxidation, the composition of 50% Pd–50% Ni exhibited the maximum activity. The strong relationship between this synergistic impact and the alloy phase state, chemical composition, and atomic structure was underlined [31]. There were examples in the literature where Pd–Ni alloys or doping alter energy barriers, creating a balance between concentration and activity-selectivity. Here, it was shown that doping Pd into Ni containing CeO_2 supported systems not only resulted in the formation of a Pd–Ni alloy structure, but also reduced energy barriers for dehydrogenation reactions with the assistance of oxygen species. This provides an example of how electron interactions between Pd–Ni can regulate catalytic activity [32]. Pd-Ni alloys can also exhibit concentration-selective behavior; selectivity and C-C bond breakage are affected by changes in Pd/Ni ratio and concentration.

In the case of Ni75, the conversion rate of catalyst was 99.67%. Despite achieving a high conversion, catalytic performance noticeably decreased as the Pd content decreased. This indicated that in order for Pd to maintain its role as an active center, it must be present at a minimum concentration. The lowest carbon monoxide conversion was shown by Ni100 catalyst (98.64%). This result clearly showed that Ni alone can be active in CO oxidation, but it did not have as high an activity as Pd. The low activity of Ni can be explained by the fact that CO molecules cannot be adsorbed sufficiently effectively on the surface or that oxygen activation was limited. Additionally, the work by Requies et al. shown that the activity of CO oxidation was enhanced by the addition of noble metals [33].

Pd was often necessary for catalytic activity, and alloying it with Ni in specific ratios can be regarded as a cost-cutting method without lowering performance much. These metals can have synergistic effects, as seen by the high conversion rates seen in Pd-Ni bimetallic systems. Furthermore, it was determined that, in terms of cost/performance, Ni25 and Ni50 ratios could be more appropriate for industrial applications, even though the reduction of Pd content results in minor performance losses.

4. Conclusion

The efficiency of the system design solution, which combined catalyst and engine technology, was realized decades after converters were invented. In this study, Pd-Ni bimetallic catalysts supported on cerium-coated cordierite were successfully prepared and characterized through a range of techniques including XRD, FTIR, SEM/EDS, and BET analysis. The characterization results confirmed that both metal dispersion and metal–support interactions were effectively achieved. BET measurements, in particular, showed that bimetallic compositions such as Ni25 and Ni50 provided increased surface areas, suggesting a synergistic structural effect.

Catalytic tests further demonstrated that these compositions maintained high CO conversion rates, nearly matching that of the pure Pd catalyst. These findings highlight the potential of Pd-Ni systems to reduce noble metal usage without compromising performance, offering a promising route toward more cost-effective and efficient catalytic materials for emission control. The significance of cerium oxide coatings in improving oxygen storage and transfer was highlighted in this work, which contributed to the growing body of research exploring noble-metal dilution approaches through bimetallic design. Future studies should concentrate on thermal aging effects, long-term stability testing under actual exhaust settings, and mechanical insights using in situ or operating spectroscopic methods. It would also be crucial for developing up the catalyst composition and include it into monolithic converter systems in order to assess its economic potential. High catalytic efficiency with significantly reduced Pd loading has important economic and environmental advantages from an industrial perspective, especially for use in stationary pollution control systems and automotive exhaust treatment.

Article Information Form

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No conflict of interest or common interest has been declared by authors.

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