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Development of High-Surface-Area Alumina-Supported Catalysts for the Generation of Hydrogen from NaBH₄

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NaBH4'ten Hidrojen Üretimi için Yüksek Yüzey Alanlı Alümina Destekli Katalizörlerin Geliştirilmesi

Meltem KARAİSMAİLOĞLU ELİBOL*, 🕕

Turkish-German University, Faculty of Natural Sciences, Department of Energy Science and Technology, Istanbul, Türkiye



Abstract Hydrogen as a v

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Hydrogen as a valuable energy carrier plays a significant role in renewable energy technologies to reduce the greenhouse gas emission into the atmosphere. However, natural hydrogen gas does not exist in the universe and should be gained from hydrogen-containing compounds. In this regard, metal hydrides are excellent candidates for producing hydrogen gas. Among complex metal hydrides, sodium borohydride (NaBH₄) possesses its advantages due to its enhanced hydrogen storage capacity and low cost. In the present study, hydrogen gas was generated through the catalytic hydrolysis of NaBH₄. In this regard, highsurface-area alumina-supported Ni/Al₂O₃, NiCo/Al₂O₃, and Ru-NiCo/Al₂O₃ catalysts have been prepared via wash coating method and tested in a continuous flow reactor. The results indicate that the Ru-NiCo/Al₂O₃ catalyst with a specific surface area of 154.40 m²·g⁻¹ showed the highest initial catalytic activity of 0.031 mmol·s^-1·g^-1 but with a rapid loss in its activity. Compared to that, despite a lower initial catalytic activity in the presence of the NiCo/Al $_2O_3$ catalyst, the hydrogen generation kept rising during the reaction, and 225 mL H₂ was produced after 100 min. Therefore, the NiCo/Al $_2O_3$ catalyst with a surface area of 165.84 $m^2 \cdot g^{-1}$ can be proposed as a promising alternative.

Keywords: Sodium borohydride; Hydrolysis; Hydrogen; Alumina; Catalyst

1. Introduction

Air pollution with the following issue "climate change" is one of the most important issues for societies. Using fossil fuels as energy sources harms air quality. Through the combustion of fossil fuels, the amount of atmospheric carbon dioxide (CO_2) is increased. The increase in the CO_2 amount causes the formation of CO_2 layer around the earth resulting in global warming (Baykara et al. 2022). Hence, the utilization of alternative energy sources is essential to reduce CO_2 emissions. In this respect, hydrogen is a valuable energy carrier, and its reaction with the air produces just water. On the one hand, the usage of hydrogen as an energy carrier can mostly cover the energy demand, on the other hand, it reduces CO_2

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Öz

Değerli bir enerji taşıyıcısı olan hidrojen, yenilenebilir enerji teknolojilerinde atmosfere sera gazı emisyonunun azaltılmasında önemli bir rol oynamaktadır. Ancak hidrojen gazı doğada mevcut olmayıp hidrojen içeren bileşiklerden elde edilmesi gerekmektedir. Metal hidrürler hidrojen gazı üretimi için mükemmel adaylardır. Karmaşık metal hidrürler arasında sodyum borhidrür (NaBH₄), gelişmiş hidrojen depolama kapasitesi ve düşük maliyeti gibi çeşitli avantajlara sahiptir. Bu çalışmada katalitik hidroliz yoluyla NaBH4'ten hidrojen gazı üretilmektedir. Bu amaçla yıkayarak kaplama yöntemi ile yüksek yüzey alanına sahip alümina destekli Ni/Al₂O₃, NiCo/Al₂O₃ ve Ru-NiCo/Al₂O₃ yapılı katalizörler hazırlanmış olup bu katalizörler sürekli akışlı reaktörde test edilmiştir. Elde edilen sonuçlara göre yüzey alanı 154.40 m²·g⁻¹ olan Ru-NiCo/Al₂O₃ yapılı katalizör başlangıçta 0.031 mmol·s⁻¹·g⁻¹ değerinde en yüksek katalitik aktiviteye sahip olup reaksiyon boyunca aktivitesinde hızlı bir azalma gözlemlenmiştir. NiCo/Al2O3 yapılı katalizörün varlığında ise başlangıçta daha düşük bir katalitik aktivite görülmesine rağmen reaksiyon boyunca hidrojen üretimi hızla artmaya devam etmiş olup 100 dk. sonra 225 ml H₂ gazı üretilmiştir. Bu nedenle 165.84 m²·g⁻¹ yüzey alanına sahip NiCo/Al₂O₃ yapılı katalizör alternatif olarak önerilebilir.

Anahtar Kelimeler: Sodyum borhidrür; Hidroliz; Hidrojen; Alümina; Katalizör

emissions because of its carbon-free nature (Baykara et al. 2018). Compared to the storage of fossil fuels, the storage of hydrogen gas is challenging and still needs to be improved (Zhu et al. 2023). In this regard, metal hydrides are good candidates to store hydrogen, conveniently. They are classified as binary, intermetallic, and complex hydrides. This type of material has the potential to desorb the pre-absorbed hydrogen atoms forming the hydrogen gas. Binary metal hydrides in a nominal chemical formula of MH_x consist of a main group or transition metal and hydrogen (Luo et al. 2020). AB_xY_x represents intermetallic metal hydrides, herein, A and B are for hydrating and non-hydrating metals, respectively (Schneemann et al. 2018). Complex hydrides shown as MEMx include alkaline metal cations and hydrogen-

containing anions such as alanates (AlH₄⁻) and borohydrates (BH₄⁻) and possess the highest hydrogen storage capacity (Schneemann et al. 2018). Hydrolysis of complex metal hydrides is a convenient method to release hydrogen at ambient temperature. LiBH₄, NaBH₄ and Mg(BH₄)₂ possess high hydrogen storage capacity and are suitable for obtaining hydrogen via the hydrolysis process (Zhu et al. 2023, Laversenne et al. 2008)). In the presence of these metal hydrides, hydrogen obtained has a high purity required for real-time proton membrane fuel cell applications. LiBH₄ possesses the highest hydrogen storage capacity with 13.9 wt%, however, due to its enhanced cost, it is hard to use for large-scale applications. Mg(BH₄)₂ with its hydrogen capacity of 12.8 wt% is another candidate for hydrolysis processes, but a large amount of heat is released during the reaction. In addition, toxic B_2H_6 is produced as a byproduct that makes the process unfavorable (Solovev et al. 2018).

Regarding its lower price and non-toxic byproduct during hydrolysis, NaBH₄ gained more attraction to release hydrogen at ambient temperature. Several studies have been conducted to enhance the hydrogen evolution rate through catalytic hydrolysis (Balkanli and Figen 2019, Özkar and Zahmakiran 2005, Xu et al. 2024, Zhu et al. 2023). In the presence of noble metals, high catalytic activity has been reported (Patel and Miotello 2015), however, the higher cost of noble metals is a significant reason for developing of alternative catalysts with transition metals. Additionally, unsupported catalysts suffer from the agglomeration of its particles which leads to a decrease in catalytic activity. Hence, the utilization of support materials is inevitable (Fernandes et al. 2009). Several studies have been focused on the implementation of noble metals such as Ru, Pt, Pd, and Rh on the carbon nanotubes (CNTs) or cobalt oxide (Co₃O₄) supports (Bozkurt et al. 2019, Uzundurukan and Devrim 2019, Zhang et al. 2021). Catalysts which include non-noble metals such as Co, Ni, Fe, and Cu exhibit relatively good performance in hydrolysis of NaBH₄ (Filiz and Figen 2019, Wang et al. 2021). Furthermore, their lower cost compared to the noble-metal-included catalysts widens their application in this process.

Regarding the necessity of support materials in catalyst development for the hydrolysis of NaBH₄, high-surfacearea alumina (Al₂O₃) pellets were used in this study. Alumina pellets were coated with Ni-Co alloy and doped with Ru to be tested in hydrolysis of NaBH₄. The asprepared catalysts have been characterized by BET, ICP-MS, and XRD analyses, and their catalytic performances were tested in a continuous reactor under the flow of NaBH₄ solution.

2. Materials and Methods

2.1 Synthesis of catalysts

In this study, three different Al₂O₃-supported catalysts were prepared via the wash coating method listed in Table 1. The synthesis procedure consists of three steps. For the preparation of 1M precursor solutions, the appropriate amount of metal salts (NiN₂O₆·6H₂O, Alfa Aeser, $Co(NO_3)_2 \cdot 6H_2O$, Carlo Erba and $RuCl_3 \cdot xH_2O$), Merck) were dissolved in deionized water, and citric acid was added as chelate agent (n_{nitric acid}:n_{metal cation}=1.5). The pH of each solution was adjusted to 7 with ammonia solution. In the second step, Al₂O₃ pellets (Alfa Aesar, high surface catalyst support) were washed in the precursor solutions several times and then oven-dried at 225 °C. In the last step, they have been calcined at 700 °C for 2h. For the synthesis of Ru-NiCo/Al₂O₃ catalysts, Al₂O₃ pellets were firstly washed in 1M precursor solution including nickeland cobalt salts and calcined at 700 °C for 2 h. NiCo/Al₂O₃ pellets obtained after the first calcination step were washed in 0.05 M precursor solution consisting of Ru salt. After this step, pellets were recalcined at 500 °C for 2h.

2.2 Characterization of catalysts

The crystalline phases of all samples were determined by XRD analyses. XRD measurements were conducted by a Philips Panalytical EMPYREAN X-ray diffractometer (XRD) in a diffraction angle range of 10° to 90° with $CuK\alpha$ radiation. Brunauer-Emmett-Teller (BET) isotherm technique was used to determine the specific surface area of all samples. BET measurements were carried out by nitrogen adsorption using Micromeritcs Gemini VII BET Instrument. Before BET measurements, each sample was pretreated at 300 °C for 12h to remove the humidity from the sample. The metal contents in each sample were determined by Agilent Technologies 7700x inductively coupled plasma mass spectrometry (ICP-MS). Prior to measurements, samples were prepared for analysis in ETHOS EASY MILESTONE microwave acid digestion system at 210 °C.

2.3 Catalytic hydrolysis of NaBH₄

The catalytic hydrolysis of NaBH₄ was carried out in an integrated continuous system as shown in Figure 1. To prevent self-hydrolysis of NaBH₄ at ambient temperature (Schlesinger et al. 1953), firstly, an 0.5 M alkaline solution of NaBH₄ (c_{NaBH_4} : c_{NaOH} =1:1) was prepared, and the solution was fed to the reactor (filled with 1.7 g catalyst) by a peristaltic pump with the flow rate of 3 ml·min⁻¹. The amount of generated hydrogen gas was determined by the water displacement method. All reactions were carried out at room temperature. Before testing the

catalytic performance of the catalysts, they were reduced in 0.075 M NaBH $_4$ -solution.



Figure 1. The scheme of the catalytic performance system (1: inlet solution, 2: peristaltic pump, 3: fix-bed reactor, 4: gas collection (water displacement method))

3. Results and Discussions

3.1. Characterization of catalysts

Pristine alumina pellets possess a high surface area of 212.48 m²·g⁻¹. In Table 1, the BET surface areas of Ni/Al₂O₃, NiCo/Al₂O₃, and Ru-NiCo/Al₂O₃ catalysts were listed, and a decrease in the specific surface area has been noticed. These results revealed that the alumina pellets have been successfully coated with the relevant metal oxide structure. In addition, the NiCo/Al₂O₃ catalyst has

the highest, and because of the additional coating procedure, the catalyst Ru-NiCo/Al₂O₃ possess the lowest specific surface area, as expected. ICP-MS analyses have been conducted to determine the metal contents in each catalyst. NiCo/Al₂O₃ and Ru-NiCo/Al₂O₃ catalysts have similar Co and Ni content, however, in Ru-NiCo/Al₂O₃ catalyst, the presence of Ru has been detected which shows that the coating with RuO₂ has been accomplished. The crystal phases of all catalysts were determined by XRD analyses, and XRD patterns are shown in Figure 2. The diffraction pattern of pristine alumina displays significant peaks at 20 of 37.12°, 39.49°, 45.06°, and 67.52°. The Ni/Al₂O₃ catalyst possesses peaks at 2θ of 37.11°, 45.14°, 59.81°, and 65.75°, which are assigned to the crystal phase of NiAl₂O₄. Furthermore, the presence of the NiAl₂O₄ phase indicates that the Ni²⁺ cations were successfully incorporated into the alumina structure. The XRD pattern of the NiCo/Al₂O₃ catalyst shows the characteristic peaks for the Co_2NiO_4 crystal phase at 20 of 37.32°, 60.18° and 66.16°. Herein, these results confirm the deposition of Co_2NiO_4 on the alumina. The Ru-NiCo/Al₂O₃ catalyst differs from the NiCo/Al₂O₃ through RuO coating, and the presence of RuO has been proved at 20 of 28.30° and 54.45°.



Position [°2 Theta] (Copper (Cu))

Figure 2. XRD patterns of all synthesized catalysts.

Table 1. Synthesized	l catalysts with thei	r crystal phases by XRD	, metal contents by ICP-M	S and specific surface areas by BET
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Codo for catalysts	Crystal phases by XRD	Metal contents by ICP-MS				$PET(m^{2}a^{-1})$
	(Reference Code)	Ru (wt %)	Co (wt %)	Co (wt %) Ni (wt %) A		- DEI (III g)
Ni/Al ₂ O ₃	NiAl ₂ O ₄ (01-078-6954)		-	8.20 ± 0.16	31.54 ± 2.1	165.44
	Al ₂ O ₃ (00-056-1186)	-				
NiCo/Al ₂ O ₃	Co ₂ NiO ₄ (04-018-4105)	_	4.16 ± 0.22	2.86 ± 0.05	32.55 ± 2.2	165.84
	Al ₂ O ₃ (00-056-1186)					
Ru-NiCo/Al ₂ O ₃	RuO ₂ (00-040-1290)					
	Co2NiO4 (04-018-4105)	0.02 ± 0.0024	3.96 ± 0.21	2.89 ± 0.06	31.32 ± 2.1	154.40
	Al ₂ O ₃ (00-056-1186)					

3.2. Catalytic hydrolysis of NaBH₄

The hydrogen generation through NaBH₄ hydrolysis takes place through the following reaction (Equation (1)) (Liu and Li 2009). In this process, 0.5 M NaBH₄ solution was fed to the reactor continuously, and hydrogen generation was measured by the water displacement method.

$$NaBH_4(aq) + 2H_2O \rightarrow NaBO_2(aq) + 4H_2(g)$$
(1)

Firstly, the catalytic performance of the pristine alumina was tested to understand whether the alumina pellets have a catalytic effect on the hydrolysis reaction. As a result, in the presence of pristine alumina, no hydrogen generation was observed. The three synthesized different catalysts have been tested in this continuous flow reactor at the same reaction parameters. Among these catalysts, the Ni/Al₂O₃ sample exhibited poor hydrogen generation which shows that the monometallic Ni coating did not improve the catalytic performance of alumina. Additionally, regarding the XRD results, it can be concluded that NiAl₂O₄ species are not active phases to dissociate NaBH₄.

On the other hand, the NiCo/Al₂O₃ catalyst showed a promising catalytic activity as demonstrated in Figure 3a and 3b. In the presence of this type of catalyst, the initial hydrogen generation rate was slow, but it increased continuously. The behavior of this catalyst can be attributed to the presence of inactivated catalyst species that are activated after self-reduction with the generation of H₂. After a 2h reaction, the H₂ generation rate rises to

0.0014 mmol·s⁻¹·g⁻¹. As a result, it can be said that the Co_2NiO_4 crystal phase acts as an active species for the dissociation of NaBH₄ molecules.

The addition of RuO into the catalyst structure has a significant improvement on the performance of NiCo/Al₂O₃ catalyst as seen in Figure 3c and 3d. In the presence of the Ru-NiCo/Al₂O₃ catalyst, the hydrolysis reaction starts with a reaction rate of 0.031 mmol·s⁻¹·g⁻¹, but the reaction rate decreases rapidly and the hydrogen generation rate sinks to 0.005 mmol·s⁻¹·g⁻¹. The behavior of the catalyst can be assigned to the deactivation of Ru species through the accumulation of Na-based compounds on the catalyst surface (Arzac et al. 2012).

Table 2 gives an insight into the performances of various catalysts from the literature comparable with results from the present study. Here, it can be concluded that the alumina-supported catalysts in powder form exhibited higher H₂ generation due to its higher amount of active metal sites on the catalyst surface (Xu et al. 2007). However, in the presence of a catalyst supported on ceramic foam (Balkanlı and Figen 2019) or cylindrical pellets (Su et al. 2012), H₂ generation is significantly lower than in the presence of the powder catalyst. In this study, despite the lower concentration of NaBH₄ solution and lower amount of noble metal (Ru) in the catalyst structure, a maximum H₂ generation rate of 50.4 ml·min⁻ ¹·g⁻¹ was achieved. In addition, it is expected that with an increase in the concentration of NaBH₄ solution achievement of higher H₂ generation compared to those stated in the literature is possible.



Figure 3. Hydrogen generation from NaBH₄ in the presence of NiCo/Al₂O₃ and Ru-NiCo/Al₂O₃ catalysts at 25 °C.

Catalyst composition	Support form	т (°С)	NaBH₄ solution	Max. H ₂ generation	Ref.
0.05 wt% Pd-4.7 wt% LiCo/SiC-based ceramic foam	Ceramic foam	25	cNaBH ₄ : cNaOH=1:1	4.76 ml∙min ⁻¹	Balkanli and Figen 2019
5 wt% Ru/Al ₂ O ₃	Pellet	25	10 wt% NaBH₄-1 wt% NaOH	65.5 ml∙min ⁻¹ ∙g ⁻¹	Su et al. 2012
2 wt% Pt/Al ₂ O ₃	Powder	30	10 wt% NaBH₄-5 wt% NaOH	8510 ml·min⁻¹·g⁻¹	Xu et al. 2007
7 wt% NiCo/Al ₂ O ₃	Pellet	25	2 wt% NaBH₄-2 wt% NaOH	2.2 ml·min⁻¹·g⁻¹	Present study
0.02 wt% Ru-7 wt % NiCo/Al ₂ O ₃	Pellet	25	2 wt% NaBH ₄ -2 wt% NaOH	50.4 ml·min⁻¹·g⁻¹	Present study

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Table 2. Comparison of the results from the present study with those in the literature

4. Conclusions

In summary, the high-surface area alumina pellets have been proposed as an effective support material for the catalysts in the hydrolysis reaction of NaBH₄. Alumina samples loaded with metal oxides were successfully prepared via the wash coating method. Results revealed that Co_2NiO_4 species form active centers for the dissociation of NaBH₄ molecules. Consequently, the Ru-NiCo/Al₂O₃ catalyst exhibited the highest initial hydrogen generation rate, however, the NiCo/Al₂O₃ catalyst can be proposed as a promising alternative due to its increasing catalytic performance during the reaction.

Declaration of Ethical Standards

The authors declare that they comply with all ethical standards.

Declaration of Competing Interest

The authors have no conflicts of interest to declare regarding the content of this article.

Data Availability

All data generated or analyzed during this study are included in this published article.

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