

PERFORMANCE EVALUATION OF A NIOBIUM-BASED PELLET CATALYST: HYDRODEOXYGENATION OF BIO-OIL IN A TRICKLE BED REACTOR

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ABSTRACT: HDO of fast pyrolysis bio-oil shows potential in generating renewable and low-carbon liquid fuels. Catalytic HDO encompasses a series of reactions involving oxygenated organic molecules under pressurized hydrogen and the presence of an active catalyst, resulting in the reduction of the degree of oxygenation. The choice of catalyst support shape, whether pelletized or powdered, can significantly influence catalytic activity and stability. This work compares the performance of powdered and pelletized niobium oxide (Nb_2O_5)-based catalysts during batch-HDO, and evaluates the performance of pelletized catalysts in a continuous trickle reactor. Guaiacol is used as a representative of bio-oil. The study aims to provide an understanding of the variations in catalytic activity, selectivity, and stability between the two forms of catalysts, thereby offering valuable insights into their suitability for large-scale continuous HDO applications. By comprehending the unique characteristics and performance qualities of pelletized and powdered catalysts, the advancement of efficient and scalable catalyst systems for bio-oil refining can be facilitated.

Keywords: Catalytic conversion, pellet, characterization, oxygenated compounds, upgrading

1 INTRODUCTION

The hydrodeoxygenation (HDO) of bio-oil derived from fast pyrolysis is a promising approach for producing renewable and low-carbon liquid fuels. Solid catalysts, particularly those based on niobium (Nb_2O_5), have shown considerable potential in facilitating the HDO process by effectively removing oxygen-containing functional groups [1]. However, the choice of catalyst support shape, whether powder or pellet, can significantly influence the catalytic activity and stability.

In industrial settings, fixed-bed and trickle-bed reactors are commonly employed to evaluate catalysts under typical operating conditions [2]. However, these reactor configurations necessitate the use of catalysts in the form of pellets, tablets, or grains. In contrast, reactions conducted in stirred autoclaves can utilize catalyst powders. The choice between pelletized and powdered catalysts can significantly impact the performance of the HDO process.

Pelletized catalysts offer distinct advantages over powdered catalysts in large-scale continuous reactors. The use of pellets ensures improved catalyst packing and bed stability, leading to enhanced mass transfer and reduced pressure drops along the reactor [3]. The larger surface area and controlled particle size distribution of pellets facilitate efficient reactant diffusion, resulting in enhanced catalytic activity. Furthermore, the pelletized form provides better control over reactant flow and residence time distribution, leading to more uniform reaction conditions and improved selectivity [4].

In contrast, powdered catalysts, typically used in batch reactors, offer certain advantages in terms of surface area and accessibility of active sites. The former permits increased contact between the catalyst and reactants, promoting higher catalytic activity. Additionally, powdered catalysts often exhibit faster reaction kinetics due to the absence of mass transfer limitations associated with pelletized catalysts.

The present work aims to compare the performance of powdered and pelletized niobium oxide (Nb_2O_5)-based catalysts for the HDO of a fast pyrolysis bio-oil (FPBO) surrogate in a batch reactor. Subsequently, the performance of the pelletized catalysts will be evaluated

in a continuous trickle-bed reactor. This comparative study will provide insights into the differences in catalytic activity, selectivity, and stability between the two catalyst forms, shedding light on their suitability for large-scale continuous HDO applications.

The choice of using guaiacol as a model molecule in this study instead of the real fast pyrolysis bio-oil is based on several considerations. Guaiacol, a key component of lignin-derived compounds, can be used as a model compound since it captures several key features and functional groups found in the complex mixture of bio-oil [5]. By using guaiacol as a model molecule, we can focus on understanding the fundamental catalytic mechanisms and reaction pathways involved in the hydrodeoxygenation (HDO) process without the added complexity of the bio-oil matrix. Guaiacol allows for controlled experiments and more precise analysis of the catalytic activity, selectivity, and stability of the niobium-based pellet catalyst. Moreover, guaiacol is readily available, well-characterized, and its reactivity has been extensively studied in the literature, providing a solid foundation for comparison and benchmarking. By utilizing guaiacol as a model compound, we can gain valuable insights into the performance of the niobium-based pellet catalyst and establish a basis for further optimization and scale-up in the context of real fast pyrolysis bio-oil conversion.

By examining the distinct characteristics and performance attributes of pelletized and powdered catalysts, a comprehensive understanding of their impact on the HDO process can be gained. This knowledge will contribute to the development of efficient and scalable catalyst systems for bio-oil refining, ultimately advancing the utilization of renewable energy sources

1.1 Niobium and Ruthenium Catalysts

Niobium and ruthenium catalysts have garnered significant attention due to their importance and wide-ranging applications in various catalytic reactions. These catalysts exhibit unique properties and offer distinct advantages that make them highly desirable in different catalytic processes.

Niobium catalysts have been extensively studied and utilized in several reactions, including

dehydration, hydration, etherification, hydrolysis, condensation, dehydrogenation, alkylation, photochemical and electrochemical polymerization, and oxidation [6]–[12]. Their water-resistant acid sites and high stability make them promising catalyst supports, particularly for the hydrotreatment of biomass-derived model compounds. Studies have demonstrated that Nb₂O₅-supported catalysts exhibit superior performance in hydrodeoxygenation (HDO) reactions compared to other solid acid catalysts, primarily due to the ability of NbO_x species to promote C-O bond cleavage. The oxophilicity of Nb⁴⁺ and Nb⁵⁺ ions induces polarization of the C-O bond, facilitating bond scission and enabling efficient HDO [13].

Similarly, ruthenium catalysts have shown remarkable versatility and efficacy in various catalytic processes. They have been employed in hydrogenation, oxidation, hydrogen transfer, and various other transformations. Ruthenium catalysts have been explored in the upgrading of biomass-derived compounds, including the conversion of lignin-derived model compounds and pyrolysis vapors [5], [12], [14]. These catalysts exhibit high selectivity and activity, enabling effective deoxygenation and hydrogenation reactions. Additionally, the utilization of ruthenium catalysts in the HDO of real feedstocks, such as pine pyrolysis vapors and raw birch lignin, has demonstrated significant reductions in oxygenated compounds and an increased energy content of the upgraded oils [15].

Recent research papers have delved into the synthesis methods, characterization techniques, and catalytic performance of niobium catalysts. These studies have primarily focused on investigating the behavior of Nb₂O₅-supported catalysts in the hydrotreatment of biomass-derived model compounds, shedding light on reaction pathways and clarifying the catalyst's behavior [9], [16]. Notably, these investigations have revealed the superior performance of Nb₂O₅ catalysts in hydrodeoxygenation (HDO) reactions compared to other solid acid catalysts. This advantage is attributed to the presence of water-resistant acid sites and the oxophilicity of Nb⁴⁺ and Nb⁵⁺ species. As a result, Nb₂O₅ catalysts exhibit enhanced hydrogenolysis and hydrogenation reactions, leading to reduced viscosity and higher aliphatic proton content in the upgraded oils [9].

As support, Nb₂O₅ exhibits distinct physical and chemical characteristics that influence the spillover mechanism. In the presence of Nb₂O₅, dissociated active hydrogen species resulting from the interaction between the metal catalyst and hydrogen molecules can more easily diffuse to the surface of the support [17]. This is attributed to the specific properties of Nb₂O₅, such as its surface chemistry and structure, which promote the migration of active hydrogen species.

Furthermore, the strong interaction between the metal catalyst and Nb₂O₅ support enhances the stability and durability of the catalyst system during the HDO process. This interaction helps prevent the loss of active surface area due to metal leaching or pore sintering, ensuring efficient catalytic activity over extended periods.

Overall, Nb₂O₅ support facilitates the occurrence of hydrogen spillover by providing suitable conditions for the migration of active hydrogen species and maintaining the stability of the catalyst system. A thorough understanding of niobium's role as a support material is crucial for optimizing the HDO process and developing efficient catalyst systems for biomass conversion.

2 METHODOLOGY

A 5% Ru/Nb₂O₅ catalyst was prepared using wet impregnation method. Commercially available Nb₂O₅ (Sigma Aldrich, No. 14898-67-0; St. Louis, MO, USA) was used as the catalyst support, and ruthenium (5 wt. %) was employed as the active metal. The support material used for catalyst preparation included Nb₂O₅ milled pellets or niobic acid, Nb₂O₅·nH₂O (HY340) kindly provided by CBMM, Brazil.

X-ray diffraction (XRD) analysis was performed to determine the crystal structure and phase composition of the catalyst. N₂ physisorption (BET) analysis was conducted to measure the specific surface area of the catalyst. Inductively coupled plasma (ICP) was employed to analyze metal loading. Scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS) was used to examine the morphology and elemental composition of the catalyst.

The hydrodeoxygenation (HDO) reactions were carried out in a batch reactor under a hydrogen atmosphere (80 bar at room temperature). A 10% guaiacol solution in decalin was used as the reactant, and the reactions were performed at 250 °C for 2 hours. After the reaction, the products were analyzed using gas chromatography-flame ionization detection (GC-FID) and gas chromatography-mass spectrometry (GC-MS) techniques to determine the product distribution and identify the converted compounds.

In addition to the batch reactor experiments, the performance of the pelletized catalysts was also evaluated in a continuous trickle bed reactor. The continuous reactor setup consisted of a stainless-steel column packed with the pelletized Nb₂O₅-supported catalyst.

The trickle-bed reactor was operated under similar HDO conditions as the batch reactor, with a hydrogen pressure of 300 bar at room temperature. A feed solution containing 10% guaiacol in decalin was continuously pumped into the reactor at a flow rate of 300 ml/h or 0.2101 mol/h of guaiacol and a hydrogen flow rate of 0.4 mol/h. The reactant mixture was heated and circulated through the system. Inside the reactor, liquid-phase HDO reactions took place as the feed solution trickled down through the catalyst bed while being simultaneously contacted with a continuous flow of hydrogen gas.

3 RESULTS AND DISCUSSION

3.1 Catalyst Characterization

The catalyst properties are presented in **Table I** and show the impregnation of Ruthenium (Ru) onto the powder and pellet support. Furthermore, a significant disparity is observed between the metal loading measurements obtained by ICP-OES (bulk catalyst) and EDS (catalyst surface), with EDS measurements reflecting higher metal concentration on the catalyst surface due to the wet impregnation technique. ICP analysis reveals that the powder catalyst has approximately 2.4 times higher Ru content compared to the pellet catalyst, while REM/EDS surface composition analysis shows a slightly higher amount of Ruthenium (8.14) on the powder catalyst compared to the pellet catalyst (7.44). This discrepancy between bulk and surface composition can be attributed to the wet impregnation method used in catalyst preparation

The application of the Scherrer equation reveals that

the average crystallite size of Ru particles is larger on the pellet support compared to the powder support, as determined to be 22.41 nm and 16.1 nm, respectively. This suggests that the pellet support promotes a slightly larger crystallite size for Ru. Moreover, the BET surface area measurements indicate that the pellet catalyst exhibits a slightly higher surface area (115.27 m²/g) compared to the powder catalyst (105 m²/g), indicating greater availability of catalytic sites on the pellet catalyst.

Although both the powder and pellet catalysts underwent the same calcination temperature, the observed differences in crystallite size and surface area cannot be solely attributed to the calcination temperature, emphasizing the influence of other factors.

Table I: Properties of the supports and fresh catalysts

	Composition by ICP (wt%) ^a		Composition by REM/EDS (wt %) ^a			
	Ru	Nb	Ru	Nb	O	C
Powder	3.17	60.2	8.14	65.36	21.62	4.35
Pellet	1.32	63.05	7.44	61.8	19.55	11.75

Scanning electron microscopy (SEM) images, as shown in **Fig. 1**, were employed to verify the successful impregnation of Ru onto the catalyst's surface. The images provide visual confirmation of Ru distribution on both the powder and pellet catalysts.

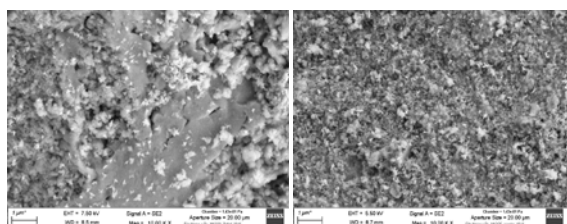


Figure 1 SEM images of fresh 5% Ru/Nb₂O₅ powder (left side) and fresh 5% Ru/Nb₂O₅ pellet (right side).

X-ray diffraction (XRD) analysis, depicted in **Fig. 2**, was conducted to examine the crystalline structures of the catalysts. The XRD patterns exhibit similarities between the two catalysts, indicating comparable crystallographic properties. However, a distinctive peak at 2θ of 26.51° is observed in the XRD pattern of the pellet catalyst, which corresponds to the presence of C graphite. This peak can be attributed to the use of C graphite as a binder in the pellet formulation.

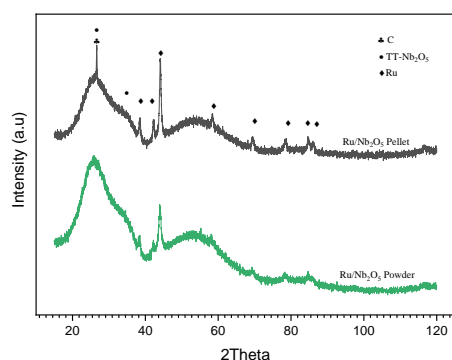


Figure 2 XRD Patterns of 5% Ru/Nb₂O₅ pellet and

powdered catalyst

2.1 Catalyst Performance - Batch Reactor

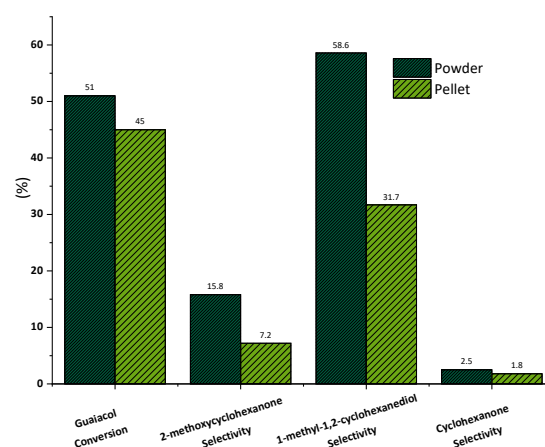


Figure 3: Conversion of Guaiacol and products selectivity (%) of HDO in the batch reactor with powder and pellet catalyst

The catalytic performance of the 5% Ru/Nb₂O₅ powder catalyst was evaluated, resulting in a guaiacol conversion of 51% and a rate of 0.18 mol/h. The selectivity of the powder catalyst was observed to be 15.7% for 2-Methoxycyclohexanone, 58.6% for 1-methyl-1,2-cyclohexanediol, and 11.7% for Cyclohexanone (**Fig. 3**). In comparison, the 5% Ru/Nb₂O₅ pellet catalyst exhibited a guaiacol conversion of 45% and a rate of 0.27 mol/h. The selectivity of the pellet catalyst was found to be 7.2% for 2-methoxycyclohexanone, 31.7% for 1-methyl-1,2-cyclohexanediol, and 1.8% for Cyclohexanone.

The formation of 2-methoxycyclohexanone and 1-methyl-1,2-cyclohexanediol can be explained by a reaction pathway involving the direct hydrogenation of the aromatic ring in guaiacol. This initial hydrogenation step leads to the formation of an intermediate product, 2-methoxycyclohexanone, also detected. Subsequently, this intermediate undergoes isomerization and further hydrogenation, resulting in the formation of 1-methyl-1,2-cyclohexanediol [18].

The presence of the saturated ketone product, 2-methoxycyclohexanone, as an important reaction intermediate suggests its role in facilitating the subsequent transformations [19]. Under acid-free conditions, the transformation of methyl-1,2-cyclohexanediol to cyclohexanol can occur, where the cyclohexanone intermediate formed by the hydrogenation of guaiacol can be further converted to cyclohexanol via hydrogenolysis. This step involves the hydrogenolysis of the methoxy group and the dehydration of the hydroxyl group.

At higher temperatures (≥250 °C), additional reactions take place, including the transformation of cyclohexanol into cyclohexane. These reactions involve the hydrogenolysis of the methoxy group and the dehydration of the hydroxyl group, leading to the conversion of the phenyl ring with two oxygen-containing functional groups into cyclohexane.

Overall, the main reaction pathway for the conversion of guaiacol involves the transformation of guaiacol into

1-methyl-1,2-cyclohexanediol, which can further undergo reactions to form cyclohexanol and ultimately cyclohexane. The formation of 2-methoxycyclohexanone and 1-methyl-1,2-cyclohexanediol as intermediates highlights their significance in the overall reaction mechanism, contributing to the selectivity towards the desired products.

3.2 Catalyst Performance – Pellets in Continuous Reactor

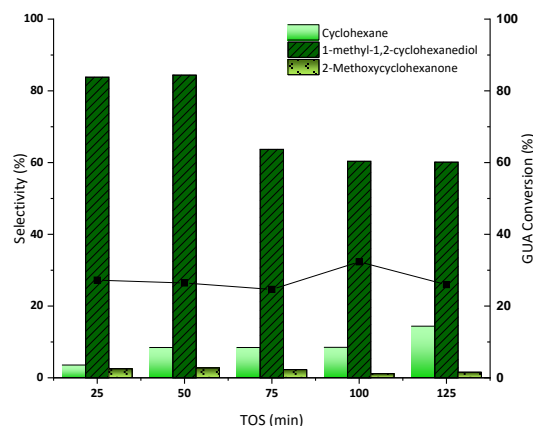


Figure 4: Conversion of Guaiacol and products selectivity (%) of HDO in the continuous reactor with Pellet catalyst

In this study, a continuous reactor system was employed to investigate the catalytic hydrogenation using the same solution of guaiacol in decalin. The reactor operated at a temperature of 250°C, a liquid flow rate of 300 ml/h, and an H₂ flow rate of 0.4 mol/h, under a pressure of 300 bar. The Weight Hourly Space Velocity (WSHV) was set at 3.75 h⁻¹.

The observed conversion of guaiacol in the continuous reactor, ranging between 20% and 30% (Fig. 4), can be attributed to the specific operational conditions employed, particularly the Weight Hourly Space Velocity (WSHV) setting. The chosen WSHV value was selected to avoid overheating and ensure the optimization of the hydrogenation process. Although the conversion is relatively low, it indicates that a significant portion of guaiacol is undergoing conversion into other compounds, while some unreacted guaiacol remains. These results support the effectiveness of the 5%Ru/Nb₂O₅ Pellet catalyst in promoting the hydrogenation reactions under the given operating parameters

Due to its high energy content, the catalytic hydrodeoxygenation (HDO) of guaiacol into cyclohexane represents a valuable model reaction for upgrading bio-oil [20]. In this process, a notable portion of guaiacol is transformed into cyclohexane, a fully deoxygenated product with diverse applications. The selectivity for cyclohexane, which accounts for approximately 15% of the converted guaiacol, highlights the effectiveness of the Ru/Nb₂O₅ Pellet catalyst in facilitating the desired hydrodeoxygenation reactions. This successful conversion demonstrates the catalyst's potential for producing oxygen-free products and emphasizes its role in optimizing bio-oil upgrading processes.

Notably, the selectivity for 1-methyl-1,2-cyclohexanediol was found to be higher than 60%. This suggests that a significant fraction of the converted guaiacol undergoes hydrogenation reactions leading to the formation of 1-methyl-1,2-cyclohexanediol. This

compound is an important intermediate in the reaction pathway and can further participate in subsequent transformations to yield other oxygenate products.

The achieved conversion and selectivity values demonstrate the effectiveness of the 5% Ru/Nb₂O₅ Pellet catalyst for the hydrogenation of guaiacol.

4 CONCLUSIONS

In this study, the performance of powdered and pelletized niobium oxide (Nb₂O₅)-based catalysts for the hydrodeoxygenation (HDO) of guaiacol was compared. The results revealed that both catalyst forms exhibited catalytic activity and selectivity towards the desired HDO products. The powdered catalyst demonstrated higher guaiacol conversion and selectivity towards the target intermediates, 2-methoxycyclohexanone and 1-methyl-1,2-cyclohexanediol. On the other hand, the pelletized catalyst achieved lower guaiacol conversion but maintained selectivity towards the desired products. These findings indicate that the choice between pelletized and powdered catalysts can influence the reaction kinetics and selectivity in the HDO process.

In terms of catalyst stability, the pelletized catalyst exhibited a slightly larger crystallite size and higher surface area, indicating the potential for improved long-term performance. The use of pellets in large-scale continuous reactors offers advantages such as enhanced mass transfer, reduced pressure drops, and better control over reactant flow and residence time distribution. These factors contribute to more uniform reaction conditions and improved selectivity. However, the powdered catalyst demonstrated higher surface area and accessibility of active sites, resulting in faster reaction kinetics.

Overall, this study highlights the importance of catalyst form selection in the HDO process. Pelletized catalysts show promise for large-scale continuous reactors, offering improved stability and selectivity. However, powdered catalysts can provide faster reaction kinetics and higher surface area. The insights gained from this comparative study contribute to the development of efficient and scalable catalyst systems for bio-oil refining, advancing the utilization of renewable energy sources and promoting a low-carbon future.

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