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Development of Advanced Catalytic Systems for Upgrading Biocrude Oil and Biodiesel into High-Quality Renewable Transportation Fuels

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Abstract: The passage discusses research on improving biofuel production through efficient catalysts for oxyorganic hydrodeoxygenation (HDO) processes. Key findings include Ni–Cu catalysts outperform single Ni catalysts under mild conditions for HDO.Copper aids in reducing nickel oxide at temperatures below 300°C.Copper also inhibits methanation of oxy-organic compounds between 280–350°C.Catalyst supports are crucial; CeO2 and ZrO2 are most effective due to their ability to further activate oxy-compounds on their surface.The catalysts developed are non-sulfided, making them suitable for upgrading bioliquids with low sulfur content.

Keyword: Biomass, Catalyst, Hydrode oxygenation(HDO), Hydrotreatment, Bio-oil, Bioliquid

1.INTRODUCTOIN:

Oxygen and increase hydrogen content to improve fuel characteristics. Recent research on The 20th century saw a surge in petroleum hydrodeoxygenation (HDO) of fatty acid esters consumption, driven largely by the automobile (biodiesel) aims to upgrade biodiesel and bio-oil for industry. Currently, fossil fuels like coal, oil, and use as spark engine fuels. Most studies use natural gas supply over threequarters of global conventional hydrodesulfurization (HDS) energy needs. Biomass, as a renewable resource from catalysts—sulfided Co–Mo and Ni–Mo supported on plants, offers a sustainable alternative for energy and alumina—but these require sulfur-containing motor fuels. Biodiesel and bioethanol are existing additives (e.g., H2S or thiophene) to keep the substitutes for conventional fuels, while bio-oil— catalysts active. However, bio-crude-oil and produced via biomass flash pyrolysis— represents a biodiesel have low sulfur content, causing these promising feedstock for engine fuels. However, catalysts to reduce to metal, leading to coke crude bio-oil cannot be directly used in spark ignition formation and deactivation. Adding sulfur donors engines due to its poor quality, primarily from high prevents this but introduces complexity, as sulfur is oxygen content. Unlike biodiesel and bioethanol, converted to H2S and removed from the products. bio-oil requires catalytic pretreatment to remove The surge in petroleum consumption during the 20th century is largely attributed to the growth of the automobile industry. Currently, fossil fuels like coal, oil, and natural gas account for over three-quarters of global energy use. However, renewable biomass from plants offers a sustainable alternative as a source of energy and motor fuels, making it the only sustainable option for both industry and automobile sectors. Biodiesel and bioethanol are already viable substitutes for conventional fuels. Another promising source is bio-oil, produced through biomass flash pyrolysis. Yet, crude bio-oil cannot be used directly in spark engines due to poor performance, primarily caused by its high oxygen content. Unlike biodiesel and bioethanol, which can be blended with traditional fuels, bio-oil requires catalytic

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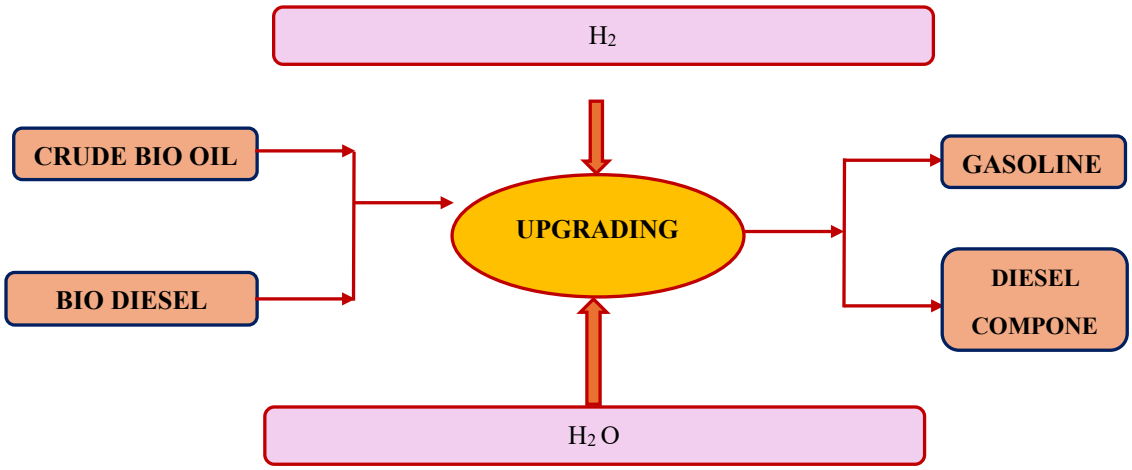
pretreatment to remove oxygen and increase hydrogen content to improve its fuel quality. Recent publications have focused on the hydrodeoxygenation (HDO) of fatty acid esters (biodiesel) to improve these fuels for spark engine use. Most researchers employ conventional hydrodesulfurization (HDS) catalysts, such as sulfided Co–Mo and Ni–Mo supported on alumina, for the HDO reaction. However, these catalysts require sulfur-containing additives like H2S or thiophene in the reaction environment to maintain their active sulfided state. Comprehensive reviews by Bridgwater et al. and Elliott highlight that HDO of pyrolysis bio-oil typically uses the same sulfided Co–Mo and Ni–Mo catalysts as biodiesel upgrading. Yet, conventional HDS catalysts are not ideal for bio-crude-oil or biodiesel hydrotreating because the initial bio-feedstocks have low sulfur content. This lack of sulfur causes the reduction of sulfided Co or Ni catalysts to their metallic forms, leading to coke formation and catalyst deactivation. To prevent catalyst desulfurization, sulfur donor compounds can be added to the feedstock, generating H2S that is subsequently removed from the hydrogenated products. Alternatively, using non-sulfided catalysts can eliminate the need for this sulfurization step altogether, simplifying the process. The high stability of anisole in hydrogenolysis reactions makes it a suitable model compound for hydrodeoxygenation (HDO) tests of bio-crude-oil components.A schematic of the upgraded bio-fuels production process is presented in Scheme 1.The development of new catalysts for reductive upgrading of bioliquids is based on the concept that effective catalysts should be bifunctional. Specifically, one function requires an oxide form of a transition metal with variable valence to activate the oxygen-containing groups in bio-crude-oil compounds. The other function requires a reduced form of a transition metal to activate dihydrogen (H_2). Catalyst deactivation due to coke formation is a concern, so reaction conditions should be controlled with temperatures not exceeding 350–400 °C and hydrogen pressures around 8.0–10.0 MPa. Oxides of metals such as Mo, W, Co, Mn, Zr, Ce, Y, Sr, and La have mobile oxygen at these conditions, which aids in activating oxygen-containing compounds. Noble metals like Pt, Pd, and Rh are commonly used for hydrogen activation. However, because large-scale HDO of bioliquids would be costly with noble-metal catalysts, nickel-based catalysts are considered more practical since they can also activate hydrogen effectively under the specified conditions.



SCHEME 1. The developing pathways of the uprraded bio-fueis production from bio-liquids.

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2.EXPERIMENTAL:

2.1. Oxy-organic substrates: The study used anisole ($C_6H_5OCH_3$) sourced from Sigma Co. and biodiesel derived from rapeseed oil (Czech Republic) as the oxygen containing substrates for hydrodeoxygenation (HDO) process experiments. The biodiesel composition included oleic acid methyl ester (59%), linoleic acid methyl ester (20%), stearic acid methyl ester (10%), linolenic acid methyl ester (8%), and erucic acid methyl ester (3%).

2.2 Catalysts and Their Preparation Method: This study tested Rh-, Rh–Co-, Ni-, and Ni– Cu containing catalysts for hydrodeoxygenation (HDO). Catalyst supports included SiO_2, Al_2O_3, ZrO_2, CeO_2, and CeO_2–ZrO_2. Commercial SiO_2 and Al_2O_3 were sourced from Sasol Company, while ZrO_2, CeO_2, and CeO_2–ZrO_2 supports were prepared at the Boreskov Institute of Catalysis (Russia).Catalysts were synthesized either by wet impregnation of the supports with aqueous . Gasoline components diesel components crude bio - oil bio - diesel upgrading h 2 h2o metal salt solutions or by co-precipitation. For impregnated catalysts, spherical Al_2O_3 (1.8 mm diameter) was used, calcined at 1000°C to achieve a BET surface area of about 100 m^2/g. The catalysts were then crushed and sieved to a particle size of 0.25– 0.5 mm. In coprecipitation, 1N NaOH solution was added with vigorous stirring to a metal salt solution at 70°C.

2.3 Catalysts Characterization: The specific surface area of catalysts was measured by the BET method using nitrogen adsorption at 77 K with an ASAP 2400 volumetric device. X-ray diffraction (XRD) analysis was conducted on a Siemens D500 diffractometer with Cu Kα radiation. Diffraction patterns were recorded by scanning from per point. In situ high-temperature XRD experiments 30° to 80° 2θ with a 0.058° step and 5 s accumulation were performed in a hydrogen atmosphere (600 cm^3/min, 0.1 MPa) with a heating rate of 25°C/min up to 300°C. Patterns were recorded between 30° and

55° 2θ until equilibrium was achieved. Crystallite sizes were calculated using the Scherrer equation.

2.3HDO Reaction Conditions:

Hydrodeoxygenation (HDO) of oxy-organic compounds was carried out isothermally at 250– 400°C and total pressure of 0.5–2.0 MPa in a fixedbed flow reactor (5 mm internal diameter). The reactor was packed with 0.5 mL of catalyst diluted with 1 mL of quartz sand (0.25–0.5 mm fraction). The feed gas was a 50:50 mixture of H_2 and Ar at 20 L/h. Liquid hourly space velocity (LHSV) ranged from 1 to 6 h^{-1}.

2.3 Product Analysis:

Liquid products (organics and water) were collected in an ice-cooled trap every 30 minutes for anisole HDO and every 60 minutes for biodiesel HDO. Analysis was performed using a gas chromatograph (Hromos GH-1000, Russia) equipped with a flame ionization detector (FID, 300°C) and a Zebron ZB-1 capillary column (100% dimethylpolysiloxane, 0.25 mm × 0.32 mm × 30 m). Gaseous products were analyzed by gas chromatography using FID and thermal conductivity detectors (TCD) with packed columns containing Silohrom and activated carbon stationary phases. The HDO degree was calculated

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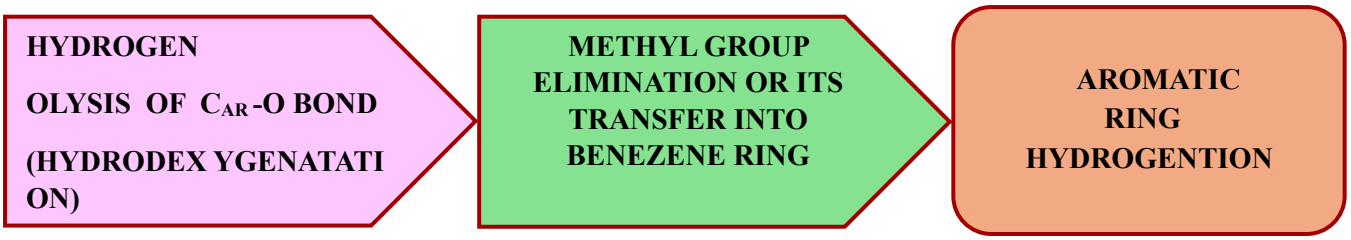

As the selectivity of oxygen-free product formation: HDO (%) $(\Sigma C^0_i / \Sigma C_i) \times 100$ where C^0_i is the concentration of oxygenfree product i, and C_i is the concentration of any product i. Specific catalytic activity was defined as the rate of oxygen-free product formation (mol/h) per gram of Ni in the Ni-based catalyst.

3.1. HDO of Anisole: Initial tests on anisole hydrodeoxygenation (HDO) using commercial

selectivity (~15%) for oxygen-free products, alongside rapid catalyst deactivation due to coke formation. Oxidized Co–Mo or Ni–Mo catalysts also performed poorly, mainly producing phenol (~40%) and methylphenol isomers (~60%). Due to these limitations, the study shifted to non-sulfided heterogeneous catalysts, starting with Rh-based catalysts (0.5 wt.% Rh), known for efficient dihydrogen activation. Screening results (Table 1) showed bimetallic catalysts outperform monometallic ones, supporting the idea that HDO requires two were calcined at 1000°C for 2 hours to form δAl2O3, which had a BET surface area of approximately 100 m²/g,

dAl2O3, ZrO2, CeO2, and SiO2 were prepared. Some catalysts were doped with Cu since copper facilitates nickel oxide reduction at lower temperatures than nickel alone. The HDO test results (Table 2) indicate that Ni–Cu catalysts are more active in anisole HDO compared to single Ni catalysts. For Ni/Cr2O3, cyclohexanol (~80%) was the main product, showing predominant aromatic ring hydrogenation, with limited Car–O bond

sulfided Ni–Mo and Co– Mo catalysts under mild conditions (250–350°C, 1.0 MPa H2) showed low HDO efficiency. For example, at 300°C and 1.0 MPa with sulfided CoMo/Al2O3, products included 41 mol.% phenol, 23% benzene, 5% cyclohexane, and about 30% methylphenol isomers. Sulfided NiMo/Al2O3 showed even lowe

active sites: one for dihydrogen activation, and another with variable oxidation state metals (Co, Ce, Zr) for oxy group activation.

Significant coke formation was observed with γAl2O3 supports due to weak Lewis acid sites and high methyl transfer selectivity, attributable to alumina's acidity. Although anisole conversion was high, γ-Al2O3 itself showed no HDO activity, producing mainly phenol and derivatives. To reduce acidity, Al2O3 supports

The goal of this work was to develop nonsulfided heterogeneous catalysts that are both active and stable for hydrodeoxygenation (HDO). A series of Ni catalysts supported on

hydrogenolysis products. The highest HDO degrees were found in bimetallic Ni–Cu systems supported on d-Al2O3 and CeO2. Catalyst activity correlates with reactant conversion, but specific catalytic activity (normalized to Ni content) decreases as Ni content increases, possibly due to reduced nickel dispersion. Some of these catalysts were also tested on real bio-crude-oil HDO at the University of Groningen, demonstrating a

International Journal of Engineering Research & Technology (IJERT)
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Vol. 15 Issue 08 , August - 2026

significant reduction of oxygen content from about 40 wt.% to 5 wt.%.

3.2. Hydrotreatment of Biodiesel:

An additional consideration in selecting catalyst supports is that ceria (CeO2) and zirconia (ZrO2) are
particularly suitable because their metal valence states can change under biodiesel HDO conditions, potentially enabling extra activation of oxygencontaining compounds. The catalysts studied were chosen to evaluate two factors: the effect of copper addition to Ni-based catalysts on activity and selectivity in biodiesel HDO, and the influence of different supports on

and rapid deactivation due to coke formation, with heptadecane (C17H36) as the main product. In contrast, Ni and Ni– Cu catalysts produced liquid linear hydrocarbons ranging from C6 to C19 and methane in the gas phase.

catalyst performance. The reactions were conducted in a fixed-bed flow reactor under consistent conditions, except for temperature, which was varied. Figure 1 illustrates how temperature affects catalyst efficiency. Notably, Figure 1b shows that most catalysts achieve full biodiesel conversion between 280°C and 330°C. However, the activity of the single CeO2–ZrO2 support without metals was significantly lower than that of Ni and Ni–Cu supported catalysts. The CeO2– ZrO2 support alone showed about 25% biodiesel conversion

Heptadecane content in liquid products ranged from 40% to 80%. Increasing temperature led to decreased heptadecane yield and increased formation of lighter hydrocarbons and methane.

CATALYST[a]	**Ni/SiO_2**	**Ni/Cr2O3b**	**Ni/ZrO_2**	**Ni-Cu/Al_2O_3**	**Ni/Al_2O_3**	**Ni-Cu/ZrO_2**	**Ni-Cu/CeO_2[d]**
LHSV(H^{-1})	1.0	6.0	6.0	1.0	1.0	0.75	1.0
ALIPHATIC/ AROMATIC PRODUCTS RATIO	1.44	1.53	0.12	4.81	3.02	0.20	-d
TOTAL CONVERSION OF ANISOLE (%)	92.8	90.2	26.0	99.6	80.0	63.5	100.0
HOD DEGREE (%)	46.0	15.7	99.0	99.2	95.0	60.0	100.0

The expected theoretical yield of oxygen-free products in biodiesel HDO is about 84 wt.%, based on the reaction of methyl oleate to octadecane and methane. However, the predominance of heptadecane suggests DCO is a main pathway, removing the carboxyl group. Hydrocarbons chain shortening mainly occurs

by removing terminal – CH2– groups, and hydrocracking increases with temperature. Selectivity data show CeO2–ZrO2 supports favor heptadecane formation, while Ni/ZrO2 produces more lighter hydrocarbons (C6– C12). Adding copper to Ni/ZrO2 shifts selectivity toward heptadecane. Ni/CeO2 catalysts show

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high selectivity for C18 and C19 hydrocarbons, suggesting FAME deoxygenation occurs via C–O bond hydrogenolysis. In contrast, Ni–Cu/ZrO2 appears to promote HDO mainly through decarboxylation. HDO should be near 340 8C. As far as the HDO and hydrocracking processes are exothermic, one can expect the catalysts layer overheating, which could result in intensification of hydrocracking. This effect may be caused by an uncontrolled temperature rise and, as a result, full methanization of biodiesel. Thus, among the tested catalysts, most attractive are Ni–Cu/CeO2–ZrO2 and Ni–Cu/CeO2 due to their ability to prevent the methane formation over wide temperature range 280–340 8C. In distinction to these catalysts, Ni/CeO2, Ni/ZrO2 and Ni/CeO2–ZrO2 cannot produce the liquid alkanes in comparable amounts at temperatures above 300 8C. Biodiesel can be almost quantitatively converted to methane over these catalysts even at 320 8C. Fig. 3 shows the methane yields obtained with the above mentioned catalysts. The lowest biodiesel conversion to methane was obtained using Ni–Cu/CeO2. Even at so high temperatures as 400 8C, selectivity of the methane formation was quite low. As seen from Fig. 3, single Ni-based catalysts seem to be unpromising for the biodiesel HDO. In the case of Ni/CeO2, Ni/ZrO2 and Ni/CeO2–ZrO2, at temperatures below 290 8C the biodiesel conversion did not exceed 70%, whereas at 290 8C and higher temperatures the uncontrolled exothermic hydrocracking was observed with full biodiesel conversion to CH4. Note that even at high temperatures the selectivity towards the light hydrocarbons (C6–C14) formation in the FAME hydrocracking process is negligible. XRD analysis showed that the initial Ni–Cu catalysts contain nickel and copper oxides. In situ reduction at 300°C for 3 hours forms a solid solution $Ni_{1-x}Cu_x$ (with x ranging from 0.22 to 0.32 depending on the loading). For the Ni–Cu/CeO2 catalyst with 29.9% Cu, metallic copper is also present. The average crystallite sizes of $Ni_{1-x}Cu_x$ particles are 10–15 nm. Under these conditions, Ni-based catalysts are not fully reduced; raising the temperature to 350°C leads to metallic nickel formation. During reduction, the Ni–Cu/CeO2 catalyst's CeO2 support lattice parameter increased from 5.417(3) Å to 5.446(2) Å.

3. CONCLUSIONS:

The catalyst screening showed that supported Ni based catalysts are effective for hydrodeoxygenation (HDO) of aliphatic and aromatic oxy-organic compounds. Ni–Cu catalysts outperform single Ni catalysts because copper aids nickel oxide reduction below 300 °C and inhibits methanation at higher temperatures. Catalyst supports significantly influence performance, with CeO2 and ZrO2 being the most effective due to their ability to additionally activate oxy compounds on their surfaces. The catalysts are non-sulfided, making them suitable for upgrading bioliquids with low sulfur content.

The passage discusses the performance and characteristics of Ni-based catalysts in biodiesel hydrodeoxygenation (HDO) and related processes:

Single Ni-based catalysts such as Ni/CeO2, Ni/ZrO2, and Ni/CeO2–ZrO2 show limited effectiveness for biodiesel conversion below 290°C, achieving less than 70% conversion.

At temperatures of 290°C and above, these catalysts cause uncontrolled exothermic hydrocracking, converting biodiesel completely to methane (CH4). Even at high temperatures, the selectivity for producing light hydrocarbons (C6–C14) from FAME hydrocracking is very low.

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