Semarak International Journal of Chemical Process Engineering

Journal homepage:
https://semarakilmu.my/index.php/sijcpe/index
ISSN: 3036-020X

Bimetallic Supported on Bivalve Calcium Catalyst for Catalytic Transfer Hydrogenation of Pyrolysis Tire Oil: Effect of Composition

Fatin Amirah Razmi[1], Mohamed Hizam Mohamed Noor[1], Norzita Ngadi[1,*]

[1] Faculty of Chemical and Energy Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia

ARTICLE INFO

Article history:
Received 28 June 2025
Received in revised form 23 July 2025
Accepted 30 July 2025
Available online 4 August 2025

ABSTRACT

Scrap tire pyrolysis oil (TPO) is a promising source of valuable chemicals, notably terpenes for various industrial applications. However, its complex composition and high sulfur content necessitate effective upgrading methods. This study explores the catalytic transfer hydrogenation of TPO using bimetallic nickel-cerium (Ni-Ce) catalysts supported on bivalve shell waste (BVS), a sustainable and low-cost catalyst support. Catalysts were synthesized via a conventional wet impregnation and subsequently evaluated under identical catalytic transfer hydrogenation conditions at varied Ni-Ce ratios (1:5, 3:3, 5:1) to determine their influence on product distribution and sulphur removal efficiency. Among these tested formulations, the 3:3 Ni-Ce/BVS catalyst achieved superior performance, achieving the highest liquid yield, producing 25.67% D-limonene- a valuable terpene compound while simultaneously achieving complete removal of sulphur- containing compounds from the pyrolysis oil. Detailed catalyst characterization confirmed key structural and functional features responsible for enhanced performance. XRD patterns confirmed the formation and stabilization of well-defined crystalline nickel and cerium oxide phases within the catalyst structure with the calcination process notably enhancing the overall crystallinity and phase purity. Furthermore, FTIR analysis detected significant modifications in the surface functional groups of the catalysts, including shifts and intensity changes in characteristic peaks associated with hydroxyl, carbonate and metal-oxygen bonds. These modifications are indicative of successful metal incorporation and interactions with the BVS support which are essential in promoting effective hydrogen transfer and hydrocarbon conversion reactions. This work advances sustainable TPO upgrading techniques and underscores the potential of waste-derived materials for eco-friendly catalytic applications.

Keywords:

Tire pyrolysis oil; catalytic transfer hydrogenation; bimetallic catalyst; D-limonene; sulfur removal

1. Introduction

Scrap tires represent a significant challenge in solid waste management due to their complex composite structures, variable raw material compositions and the inherent properties of the rubber from which they are manufactured [1]. Among various waste management strategies, pyrolysis has

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[*] *Corresponding author.*
E-mail address: norzita@utm.my

https://doi.org/10.37934/sijcpe.4.1.2232

emerged as a promising thermochemical process that converts organic waste materials including agricultural residues [1], wood biomass [2], plastic waste [3], livestock manure [4] and waste engine oils [5] into valuable energy resources. Specifically, the pyrolysis of scrap tires not only mitigates environmental issues associated with landfill accumulation but also facilitates waste valorization by producing tire pyrolysis oil (TPO), a liquid rich in hydrocarbons and valuable chemicals.

TPO predominantly consists of complex hydrocarbon mixtures ranging from C6 to C22. Its high gross calorific value (41-44 MJ kg^{-1}) surpasses that of biomass-derived pyrolysis oils (17-36 MJ kg^{-1}), making it an appealing alternative energy source [6]. However, raw TPO contains elevated sulfur (approximately 60–40%) and high concentrations of aromatics and unsaturated compounds [7], limiting its direct application as fuel. Notably, TPO presents a promising source of terpenes and terpene derivatives, which originate from the breakdown of polyisoprene in tires during pyrolysis. These compounds hold high industrial value across sectors including pharmaceuticals, perfumery, cosmetics and increasingly as renewable biofuel additives [8].

To enhance terpene yield and improve TPO quality, catalytic transfer hydrogenation (CTH) offers a safer, efficient alternative to conventional hydrogenation by using organic hydrogen donors and avoiding pressurized hydrogen gas [9]. Alkaline earth-based catalysts, particularly calcium-based materials, have shown remarkable efficacy in CTH under mild conditions, promoting desulfurization and hydrocarbon conversion while minimizing coke formation [10]. Bivalve shell waste (BVS) derived from *Anadara granosa* offers a sustainable, polymorphic calcium-based support with superior surface properties and adaptability for catalyst modification [11]. Parallelly, bimetallic catalysts, particularly nickel-cerium (Ni-Ce) systems, have demonstrated enhanced catalytic activity and selectivity through synergistic effects, improved oxygen storage capacity and stronger metal-support interactions [12]. Coupling Ni-Ce bimetallic catalysts with BVS supports addresses surface area limitations and optimizes the hydrogenation environment for TPO upgrading.

Despite the promising potential of TPO valorization and advancements in catalyst technology, limited studies have investigated the integration of bimetallic Ni-Ce catalysts supported on natural, sustainable calcium-based materials like BVS for TPO upgrading via CTH. Furthermore, the effect of varying Ni and Ce metal ratios on the physicochemical properties, catalytic behavior and terpene yield from TPO remains insufficiently explored. Therefore, this research aims to investigate the influence of varying nickel and cerium ratios in bimetallic catalysts supported on BVS, which are synthesized via the wet impregnation method. The study will assess how different Ni-Ce ratios affect the catalyst structure and physicochemical properties such as surface area, porosity and metal dispersion. Additionally, the research will evaluate the catalytic performance of these Ni-Ce/BVS catalysts in promoting CTH of TPO with a particular focus on enhancing hydrocarbon conversion, achieving effective desulfurization and maximizing terpene yield. Finally, the study will analyze the composition and activity of terpenes and their derivatives present in the upgraded pyrolysis oil to determine its suitability and potential for industrial applications as biofuel additives and raw materials for the chemical and pharmaceutical industries.

2. Methodology

2.1 Materials

Cerium (III) nitrate hexahydrate ($Ce(NO_3)_3.6H_2O$) (99.5 %) and nickel nitrate hexahydrate ($Ni(NO_3)_2.6H_2O$) (99.5 %), were purchased from Sigma Aldrich (USA), whilst, isopropanol (99.5 %) and ethanol (98 %) were purchased from QreC (Asia). All chemicals were met analytical grade standard and utilized without additional purification. Distilled water and deionized water were also employed in the study. The raw tire pyrolysis oil (TPO) sourced from QCS Biotechnology Sdn. Bhd., located in

Seelong, Johor, Malaysia. Furthermore, bivalve shells (BVS) waste, sourced from local restaurant waste in the Skudai area of Johor, Malaysia were employed in the study.

2.2 Pre-Treatment of BVS

The bivalve shells (BVS) waste was used for preparation of the calcium carbonate powder that collected from restaurants waste at area Skudai, Johor, Malaysia. The samples of BVS were boiled for 15-20 minutes to remove any impurities that attached on the seashells and cooled at room temperature. The BVS samples were immersed sodium hypochlorite (for 4 hours) to prevent the unwanted microorganism contamination on the surface BVS followed by washed thoroughly with tap water and scrubbed with brush then dried in an oven with temperature 105 °C for 4 hours [13]. Subsequently, the sample was pulverized using mortar pestle and sieved with particle size of < 100 µm to form fine powder depicted in Figure 1.

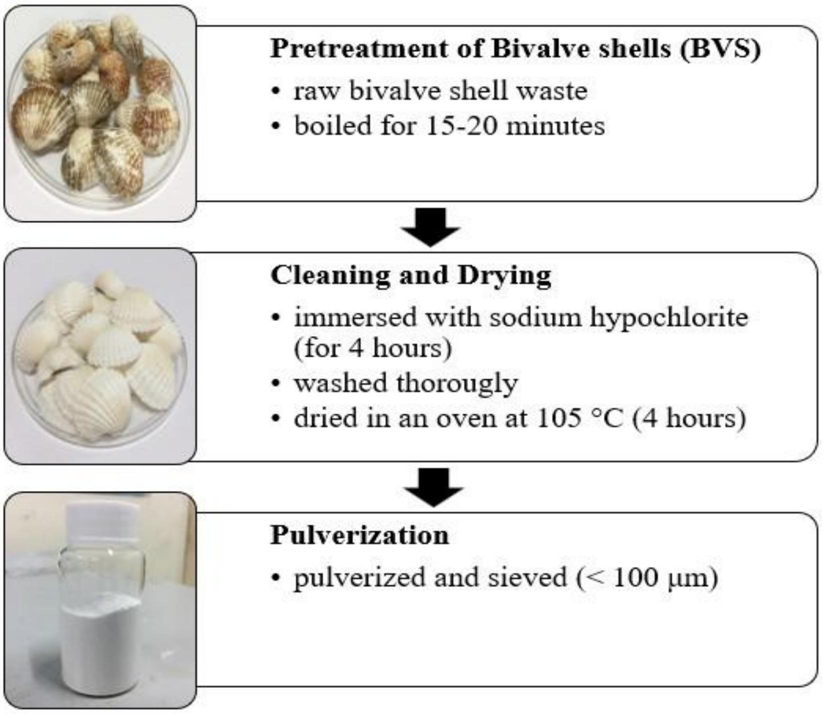

Fig. 1. Flowchart of pre-treatment process of BVS

2.3 Catalyst Preparation

The technique used to synthesis of nickel-cerium biogenic calcium catalyst wet impregnation method [14], whereby the precursor used namely are nickel (II) nitrate hexahydrate ($Ni(NO_3)_2.6H_2O$), cerium(III) nitrate hexahydrate ($Ce(NO_3)_3.6H_2O$) and bivalve shells (BVS) waste powdered. Initially, the nickel salts (Ni) and cerium salts (Ce) were mixed physically with an equivalent amount of weight percentage, wt% (3:3 = Ni/Ce). For dispersion medium, the ethanol (C_2H_6O, 98% purity) dissolve with distilled water in separate beaker. Subsequently, the salts mixture was added into dispersant solution. Then, the BVS powdered was added into the mixture solution and placed on water bath with stirring speed at 350 rpm and temperature mixing of 40°C. The solution was stirred for 3 hours which allows the reaction reach to completion. Subsequently, the solution was filtered and washed, then the sample was further dried the lump of powder was pulverized using mortar material in fine-powder form with particle size smaller than 100 µm and pestle to ensure form fine powder. Then, the BVS powdery were calcined at 550 °C (muffle furnace, Carbolite-Gero Ltd, model ELF 11/14B, England) for 4 hours. The final product is nickel-cerium supported biogenic BVS catalyst denoted as Ni-Ce -BVS catalyst throughout of this study. Herein, the synthesis procedure for Ni-Ce -BVS catalysts were repeated with a different of three variable parameters which are temperature of ageing (40, 60, 80 °C), temperature of calcination (550, 800, 1000 °C) and ratio compositions of Ni-Ce (1:5, 3:3, 5:1 wt.%, BVS = 94 wt%). All modified catalyst were denoted as *n*Ni-Ce/BVS (*n* = 1:5, 3:3, 5:1) and stored in the desiccator before applied on the following catalytic upgrading reaction.

2.4 Catalytic Transfer Hydrogenation (CTH)

In Figure 2, the batch reactor was filled with approximately 123–127 mL of TPO and isopropanol as a hydrogen donor solvent and the Ni-Ce/BVS catalyst (approximately 300 mg). For each catalytic transfer hydrogenation reaction, the heating process requires approximately 50–55 minutes to elevate the temperature from 25°C to the desired reaction temperature (375 ± 1°C), with a heating rate of approximately 7°C/min at stirring speed of 400 rpm. The stirring speed and temperature were maintained for 2 hours. Throughout the reaction, temperature measurements were recorded every minute using a temperature detector. Then, the reactor was cooled to room temperature over a period of time. Subsequently, the final reaction mixture, along with other oil samples, were extracted from the reactor via the port and subjected to volatile terpene compound analysis.

Besides that, the overall mass balance was calculated using Eq. (1). Whilst, the percentage of pyrolysis product yields was calculated according to Eqs. (2)-(5) as follow:

$$\text{Total mass(g)} = \text{Mass of distilled oil (g)} + \text{Mass of semi-solid (g)} \\ + \text{Mass of non-condensable gas (g)} \tag{1}$$

$$\text{Total mass of feedstock (g)} = \text{Mass of STPO (g)} + \text{Mass of H-donor (g)} \tag{2}$$

$$\text{Distilled oil yield (wt\%)} = \frac{\text{Mass of distilled oil (g)}}{\text{Total mass of feedstock (g)}} \times 100\% \tag{3}$$

$$\text{Semi-solid yield (wt\%)} = \frac{\text{Mass of semi-solid (g)}}{\text{Total mass of feedstock (g)}} \times 100\% \tag{4}$$

$$\text{Non-condensable gas yield (wt\%)} = 100\% - \text{distilled oil yield (wt\%)} - \text{semi-solid yield (wt\%)} \tag{5}$$

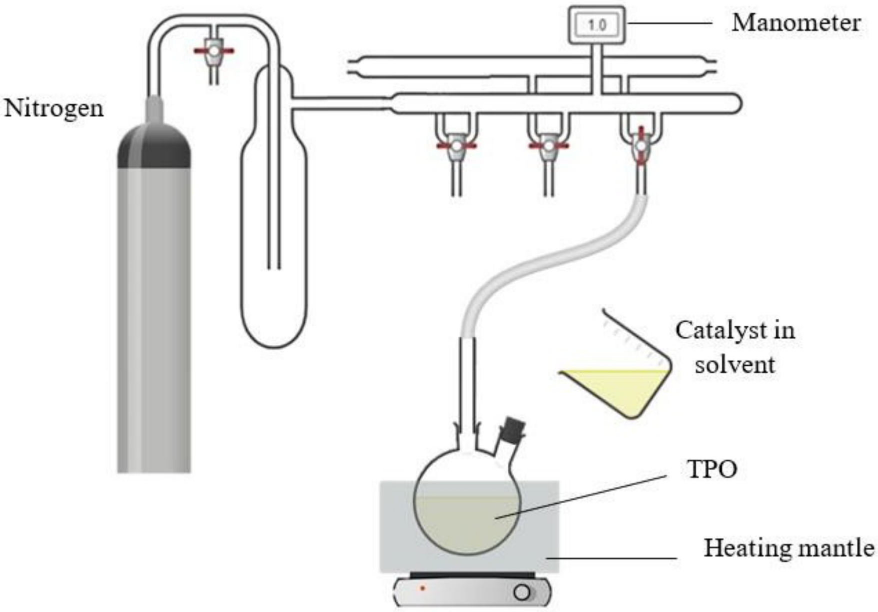

Fig. 2. CTH experimental setup

2.5 Characterization of Catalysts

The modified mesoporous Ni-Ce/BVS catalysts were characterized using several analytical techniques. X-ray diffraction (XRD) was employed to determine the crystallinity and phase identification of the catalysts, utilizing a PANAlytical X'Pert diffractometer with Cu Kα radiation, scanning from 10° to 80° (2θ) at a 0.02° step size and 10 s scanning speed. Fourier Transform Infrared Spectroscopy (FTIR) also was performed to identify functional groups in the organic materials by analyzing the infrared absorption spectra. Data were collected using an IRTracer-100 spectrometer in the range of 4000 to 500 cm^{-1} to observe changes in functional groups between the precursors and catalysts.

3. Results and Discussion

3.1 Catalytic Transfer Hydrogenation

This section discusses the results obtained from the effect of ageing temperature, calcination temperature and Ni-Ce ratio composition.

3.1.1 The effect of ageing temperature

The Figure 3 illustrates the catalytic upgrading yield of a modified Ni-Ce/BVS catalyst synthesized at different ageing temperatures (40°C, 60°C, and 80°C). The trend in yield varies across liquid, semi-solid, and gas phases, with the ageing temperature playing a significant role in determining the overall distribution of products. At 60°C, the semi-solid yield is notably lower compared to the other temperatures, while the liquid yield remains relatively high and the gas yield shows minimal fluctuation.

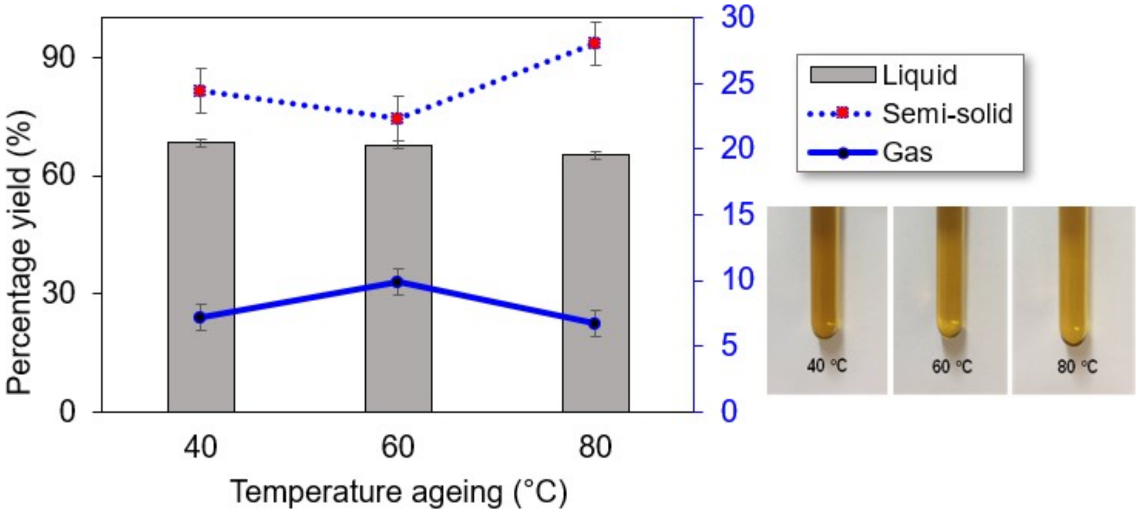

Fig. 3. Percentage yields of effect of ageing temperature for catalytic upgrading reaction
*(Reaction condition: H-donor = isopropanol (IPA); TPO/IPA v/v= 1:1, catalyst = 10%, temperature = 375 °C; time = 1 hrs; heating rate = 7 °C/min; stirring speed = 400 rpm; condenser temperature = 18 °C)

The role of nickel and cerium in this catalytic activity is key to understanding these trends. Nickel, known for its strong hydrogenation capabilities, promotes the conversion of unsaturated hydrocarbons into more saturated forms, which generally enhances liquid product yield [15]. Cerium, with its redox properties (Ce^{3+}/Ce^{4+}), provides oxygen mobility and stabilizes the nickel sites, thus balancing oxidative and reductive pathways [16]. At 60°C, the balance between these two metals appears optimal, resulting in a reduction of semi-solid products. The lower semi-solid yield suggests more efficient cracking of larger molecules into liquid hydrocarbons, which aligns with the catalytic role of nickel in hydrogenation and cerium in enhancing oxidative processes [17].

Furthermore, the slight increase in gas yield at 60°C, compared to 40°C and 80°C, suggests that this ageing temperature supports partial oxidation of smaller hydrocarbons, likely influenced by the oxygen storage and release properties of cerium. The lower semi-solid production at 60°C could also be linked to better dispersion of the active nickel species on the BVS support, preventing the formation of heavier hydrocarbons that typically lead to semi-solid residues.

The catalytic upgrading results indicate that 60°C provides the most favourable balance in product yield, with the lowest semi-solid formation and relatively high liquid yields. This trend can be attributed to the optimized interaction between hydrogenation function of nickel and redox behaviour of cerium at this ageing temperature [18]. The lower semi-solid yield at 60°C highlights the efficient catalytic cracking of larger molecules into lighter hydrocarbons, supporting the hypothesis that this temperature allows for a better dispersion of active sites and more selective conversion. Thus, 60°C emerges as the optimal ageing temperature for synthesizing the Ni-Ce/BVS catalyst, striking a balance between liquid and gas production while minimizing undesired semi-solid products.

3.1.2 The effect of calcination temperature

The Figure 4 presents the catalytic upgrading yields of a modified Ni-Ce/BVS catalyst synthesized at different calcination temperatures (550°C, 800°C, and 1000°C). The yields are distributed among liquid, semi-solid, and gas phases, with the calcination temperature significantly affecting the product composition. At 800°C, the catalyst exhibits the highest liquid yield and the lowest semi-solid yield compared to the other calcination temperatures, making it a point of interest for optimizing the catalytic process.

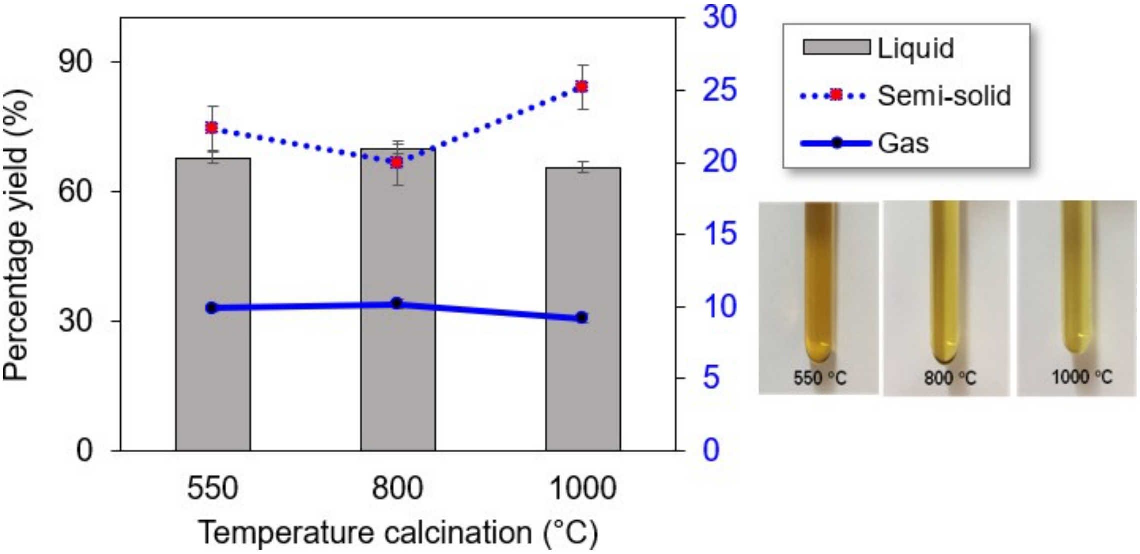

Fig. 4. Percentage yields of effect of calcination temperature for catalytic upgrading reaction
*(Reaction condition: H-donor = isopropanol (IPA); TPO/IPA v/v= 1:1, catalyst = 10%, temperature = 375 °C; time = 1 hrs; heating rate = 7 °C/min; stirring speed = 400 rpm; condenser temperature = 18 °C)

The trend shows that as the calcination temperature increases from 550°C to 800°C, there is an increase in liquid yield, while semi-solid production decreases. This trend can be attributed to the enhanced dispersion of nickel and cerium species on the BVS support at 800°C. Nickel, with its well-established role in hydrogenation, facilitates the breakdown of larger hydrocarbons into lighter liquid fractions [19]. Cerium, known for its oxygen mobility and redox cycling (Ce^{3+}/Ce^{4+}), stabilizes the active nickel sites and enhances oxidative reactions, which further promotes the conversion of heavy hydrocarbons into liquids [20].

At 800°C, the interaction between nickel and cerium seems to reach an optimal state, leading to a more efficient cracking process and suppressing the formation of heavier semi-solid byproducts. The relatively low semi-solid yield at this temperature suggests that the catalyst is effective in promoting hydrogenation and oxidative dehydrogenation reactions that crack larger hydrocarbon chains into smaller liquid molecules. The slight increase in gas production could be associated with partial oxidation of smaller hydrocarbons, likely facilitated by redox properties of cerium [21]. However, at 1000°C, the liquid yield decreases, while the semi-solid yield increases slightly. This may be due to the sintering of nickel and cerium particles at higher temperatures, which can reduce the surface area and catalytic activity, thereby decrease the efficiency of hydrocarbon cracking and lead to the formation of more semi-solid residues.

The results indicate that 800°C is the optimal calcination temperature for synthesizing the Ni-Ce/BVS catalyst, as it produces the highest liquid yield and the lowest semi-solid yield. This is likely due to the optimal dispersion and interaction of nickel and cerium at this temperature, which enhances the hydrogenation and oxidative dehydrogenation processes [22]. The improved catalytic efficiency at 800°C ensures more effective cracking of larger hydrocarbons into lighter liquid fractions, minimizing the formation of unwanted semi-solid products. In contrast, higher calcination temperatures, such as 1000°C, may lead to sintering, reducing catalytic activity and shifting the product distribution towards less favourable outcomes.

3.1.3 The effect of Ni:Ce (wt/wt) ratio composition

The Figure 5 illustrates the catalytic upgrading yield of a modified Ni-Ce/BVS catalyst synthesized with different weight ratios of Ni to Ce (1:5, 3:3, and 5:1) during catalyst preparation. The results show how the ratio of these two metals impacts the distribution of liquid, semi-solid and gas products during the upgrading process.

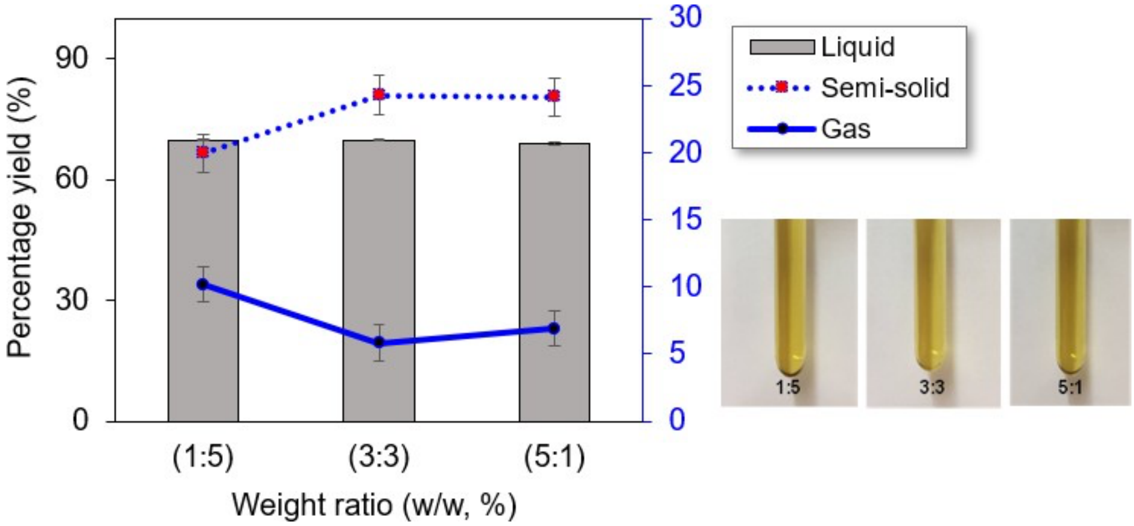

Fig. 5. Percentage yields of effect of ratio composition Ni:Ce (wt/wt) for catalytic upgrading reaction
*(Reaction condition: H-donor = isopropanol (IPA); TPO/IPA v/v= 1:1, catalyst = 10%, temperature = 375 °C; time = 1 hrs; heating rate = 7 °C/min; stirring speed = 400 rpm; condenser temperature = 18 °C)

As the Ni to Ce ratio changes, the yield of liquid hydrocarbons follows a distinct pattern, with the 3:3 ratio exhibiting the highest liquid yield and the lowest semi-solid yield compared to the other ratios. At the 1:5 Ni to Ce ratio, with a higher proportion of semi-solid byproducts, this indicates that cerium plays a role in facilitating oxidative reactions, but at this higher concentration (1:5), the catalytic balance shifts towards excessive oxidation or less effective cracking of heavy hydrocarbons, leading to an increase in semi-solids.

Nickel, known for its hydrogenation capabilities, supports the cracking of heavier hydrocarbons into lighter liquid fractions. In the case of the 5:1 ratio, the higher concentration of nickel promotes more extensive hydrogenation, but this also leads to a slight increase in semi-solid production. This increase could be due to excessive promotion of hydrogenation of nickel, where heavier hydrocarbons are only partially converted, resulting in more semi-solid products [23].

The 3:3 Ni to Ce ratio shows the most balanced performance, with the highest liquid yield and the lowest semi-solid yield. This indicates that at this ratio, there is an optimal synergy between hydrogenation ability of nickel and redox properties of cerium, leading to more efficient hydrocarbon cracking and minimal formation of undesired byproducts [24]. The role of cerium in promoting oxidative dehydrogenation is balanced at this ratio, allowing the catalyst to perform efficiently by converting hydrocarbons into lighter liquids without excessive oxidation that could lead to gas formation or semi-solid residues [25].

The key takeaway from this data is that the ratio of Ni to Ce plays a critical role in determining the efficiency and selectivity of the catalytic upgrading process. A higher nickel ratio (5:1) enhances hydrogenation but can increase the formation of semi-solid byproducts, whereas a higher cerium ratio (1:5) promotes oxidative reactions but may lead to lower liquid yields and more semi-solid residues. The 3:3 ratio represents an optimal balance where the interaction between nickel and cerium is most effective, leading to high liquid yield and minimal byproducts. This finding emphasizes that optimizing the Ni-Ce ratio is essential for achieving the desired catalytic activity. The balance between oxidative and hydrogenation reactions must be fine-tuned to promote efficient conversion of heavy hydrocarbons into lighter, more valuable products, such as liquid fuels, while minimizing undesired gas and semi-solid formation.

In summary, the results demonstrate that the Ni to Ce ratio in the catalyst strongly influences the catalytic upgrading process. The 3:3 and 1:5 ratio achieve the best overall performance, producing the highest liquid yield and minimizing semi-solid byproducts. This balance results from the synergistic effects hydrogenation properties of nickel and redox behaviour of cerium, which together enhance the conversion of hydrocarbons into desirable liquid products [26]. Deviating from this ratio, either towards higher nickel (5:1) or higher cerium (1:5), leads to reduced liquid yields and increased byproducts, highlighting the importance of fine-tuning the metal ratio for optimal catalytic activity.

4. Conclusions

This study demonstrates the effectiveness of bimetallic Ni-Ce/BVS catalysts for the catalytic upgrading of TPO, with a particular focus on terpene production and sulfur removal. The 3:3 Ni-Ce ratio provided the most balanced performance, yielding high concentrations of D-limonene and achieving complete desulfurization, crucial for improving the fuel quality of TPO. The catalytic performance was strongly influenced by the nickel-to-cerium ratio, with higher nickel content favoring overall hydrocarbon production but potentially compromising sulfur removal. Nickel was found to promote hydrogenation, while redox properties of cerium enhanced oxidative dehydrogenation, both essential for selective hydrocarbon conversion. Future research should focus on further optimizing these catalysts by fine-tuning the Ni-Ce ratio and examining the influence of reaction conditions to enhance the yield and selectivity of valuable hydrocarbons. These findings contribute to the development of more efficient and sustainable methods for upgrading scrap tire pyrolysis oil, reducing environmental impact, and enhancing the economic viability of tire recycling.

Acknowledgement

The authors would like to extend their sincere gratitude to the Universiti Teknologi Malaysia for the financial supports received under UTM Fundamental Research Grant (Vot no. Q.J130000.3809.22H62). Co-author, Mohamed Hizam Mohamed Noor also would like to acknowledge the financial support funded by Kurita Water and Environment Foundation through Kurita Overseas Research Grant 2024 (24Pmy104 or Vot no. R.J130000.7709.4J703).

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