

# Hydrogen-free Deoxygenation of Waste Cooking Oil over Unreduced Bimetallic NiCo Catalysts for Biojet Fuel Production

Brandon Han Hoe Goh<sup>a</sup>, Cheng Tung Chong<sup>a,\*</sup>, Jo-Han Ng<sup>b</sup>

<sup>a</sup>China-UK Low Carbon College, Shanghai Jiao Tong University, Lingang, Shanghai 201306, China

<sup>b</sup>Carbon Neutrality Research Group, University of Southampton Malaysia, Iskandar Puteri 79100, Johor, Malaysia  
ctchong@sjtu.edu.cn

The conversion of waste cooking oil (WCO) into renewable fuels has been proposed as a waste utilisation method which can ease global energy demand. The deoxygenation of WCO into fuel range hydrocarbons avoids the use of expensive hydrogen but comes at the cost of poor fuel properties and elevated reaction conditions. The use of bimetallic catalysts for the deoxygenation can improve catalyst performance and selectivity for alkane and alkene chains. The present work attempts to utilise the synergistic combination of nickel and cobalt catalysts to produce fuel with suitable carbon chain hydrocarbon composition. The experiments were carried out with monometallic Ni, Co and bimetallic NiCo all impregnated onto TiO<sub>2</sub> without reduction. The deoxygenation parameters were set at 100 g feedstock, 5 wt% catalyst, 250 mL/min nitrogen flow at 100 rpm rotation for 2 h under atmospheric pressure. NiO catalyst showed the highest deoxygenation conversion, obtaining 89.03 % of deoxygenated product after reaction. Although Co<sub>3</sub>O<sub>4</sub> catalyst showed relatively lower deoxygenation activity (52.01 %), the higher selectivity towards C<sub>10</sub>-C<sub>16</sub> hydrocarbons highlighted its potential to be used as catalyst promoter. The use of bimetallic NiCo catalysts in a nitrogen atmosphere was found to be capable of producing hydrocarbons within a suitable carbon chain length for jet fuel use. Overall, the sequential impregnated NiCo is identified as the optimum catalyst as it showed good deoxygenation activity (78.78 %) with the highest selectivity towards C<sub>10</sub>-C<sub>16</sub> hydrocarbons. The outcome of this study provides scientific insights on the deoxygenation of fuel range hydrocarbons under atmospheric pressure without the use of hydrogen.

## 1. Introduction

Catalytic deoxygenation of WCO is capable of producing biofuels by removing existing oxygen content from the feedstock deoxygenated to produce alkanes and alkenes with similar carbon chain length. The process is selective in the range of hydrocarbons and beneficial as high oxygen content fuels have a low heating value, high corrosivity and face instability issues (Khan et al., 2019). These products are suitable to be used as a green diesel, and potentially biojet fuel if the fuel achieves the stringent requirements for jet fuel use (Goh et al., 2020). Noble metals such as Pd, Pt, Co or Ni are commonly used as catalyst for deoxygenation reactions, with the latter ones heavily investigated since it is much cheaper than the others (Goh et al., 2022). However, strong interactions between fatty acids and active metal surfaces result in catalyst deactivation (requiring high H<sub>2</sub> pressure to maintain activity), making non-noble oxophilic metals such as Ni, Co, Cu and Cr more favourable catalyst for H<sub>2</sub> free DO (Phichitsurathaworn et al., 2020). However, in the absence of H<sub>2</sub>, the catalytic activity Ni is still relatively low for fatty acid deoxygenation (Miao et al., 2016). Co catalysts have also shown high catalyst activity and selectivity to olefins, but the larger size cobalt particles have a strong interaction with carboxylic acids which cause successive adsorption of the feed on the active sites, resulting in a rapid deactivation (Phichitsurathaworn et al., 2020). Due to the difficulty of reducing NiO catalyst, several studies have investigated the use of unreduced NiO impregnated on various supports (mesoporous SBA-15, activated carbon, graphene oxide and beta zeolite) to show high catalytic activity and selectivity towards diesel fractions (Baharudin et al., 2019). Additionally, introducing Co into Ni catalysts can also reduce the energy barriers to remove hydroxyl group from carboxyl and break C-O bond on carbonyl groups (Zheng et al., 2023). Besides that, Co<sub>3</sub>O<sub>4</sub> catalysts have been found to be highly selective for fatty acid deoxygenation as reduction of Co<sup>II</sup> and Co<sup>III</sup> to Co<sup>0</sup> during the reaction will increase the number of oxygen vacancies, even without an external hydrogen source (Zeng et

al., 2022). The catalyst support also plays multiple important roles, needing to have an excellent oxygenate removal capabilities and effective coking barrier (Khalit et al., 2021). TiO<sub>2</sub> supports have shown strong catalytic performance and metal-support interaction properties which improves its adsorption geometry, electronic structures and metal dispersion on the support (Ooi et al., 2019).

As of now, there has been minimal studies concerning the use of bimetallic Ni and Co catalyst supported by TiO<sub>2</sub> for the deoxygenation of oils. The aim of our current work is to produce fuel range hydrocarbons from WCO fatty acids in a single step deoxygenation process utilising bimetallic NiCo catalyst supported on TiO<sub>2</sub>. NiCo catalysts were prepared via co-impregnation and subsequent impregnation for comparison. The surface area and pore distribution were compared to determine the best catalyst for WCO deoxygenation. The deoxygenation performance was evaluated based on the yield of deoxygenated product and selectivity of jet-based paraffins (C<sub>10</sub>–C<sub>16</sub>).

## 2. Materials and methods

Nickel (II) nitrate hexahydrate, cobalt (II) nitrate hexahydrate and titanium (IV) oxide were provided by Aladdin. The feedstock used in this study is WCO obtained from school canteen in the Shanghai JiaoTong University China-UK Low Carbon College campus. Repeated high-temperature cooking of vegetable oils result in a mixture of unstable intermediate hydrocarbons and polymerized derivative of glycerides. In total, the GCMS detected 63.4 % of carboxylic acid within the WCO, with Table 1 showing the fatty acid composition of the feedstock. Besides carboxylic acid, other compounds present in the WCO included esters (25.4 %), alcohols (7.2 %) and aldehydes (2.3 %).

*Table 1: Fatty acid composition of WCO feedstock*

Fatty acid chain	Composition (%)
C <sub>10</sub>	10.19
C <sub>12</sub>	0.34
C <sub>14</sub>	4.03
C <sub>16</sub>	14.68
C <sub>18:0</sub>	13.20
C <sub>18:1</sub>	52.31
C <sub>18:2</sub>	1.3
C <sub>18:3</sub>	0.6
Others	3.35

### 2.1 Catalyst preparation

The Ni and Co catalyst were prepared via the wetness impregnation method without reduction. Calcination in an inert atmosphere (N<sub>2</sub> flow) has been found to thermally activate the catalysts while avoiding the use of hydrogen (Hafriz et al., 2022). In a typical procedure, 2 g of metal (II) nitrate hexahydrate was dissolved in deionized water and slowly stirred for 2 h before being dripped into 10 g of TiO<sub>2</sub> support. After drying at 100 °C overnight, the solution was subjected to calcination. The calcination temperature significantly affects the particle size (Zorkipli et al., 2016) and has to be greater than 280 °C since that is the decomposition temperature of nickel nitrate (Wei et al., 2021), whereas the decomposition temperature for cobalt nitrate is around 250 °C (Malecka et al., 2015). In this study, the calcination temperature was set at 500 °C, heated at 10 °C/min for 4 h. After calcination, the catalyst was allowed to cool down before being grinded into a fine powder. The impregnation of bimetallic NiCo catalyst was also conducted two methods, namely immediate co-impregnation of both metals (NiCo (C)) and the subsequent impregnation of Ni followed by Co (NiCo (S)). The TiO<sub>2</sub> catalyst, labelled as TiO, was subjected to similar calcination conditions before being used for WCO deoxygenation.

### 2.2 Catalyst characterisation

The produced catalysts were tested with the Brunauer-Emmett-Teller (BET) method to determine the specific surface area and the Barrett-Joyner-Halenda (BJH) method to evaluate the total pore volume and pore sizes, by using a Quantachrome, Autosorb-iQ apparatus. Initially, each sample was degassed at a 10 °C/min rate up to 150 °C and held for 6 h to eliminate the physisorbed water. The samples were then refrigerated and filled up with inert gas. The adsorption-desorption process of N<sub>2</sub> gas was conducted in a vacuum chamber on the catalyst surface. Elemental composition analysis of the samples was carried out using X-ray fluorescence (XRF) analysis using an Axios Max instrument.

### 2.3 Deoxygenation procedure for WCO

The hydrogen free deoxygenation was conducted in a 250 mL flask heated with a heating mantle. A mass flow controller was used to ensure constant N<sub>2</sub> flow at 250 mL min<sup>-1</sup>. 100 g of feedstock and 5 wt% of a selected catalyst were weighed and added to the flask simultaneously, which was purged with N<sub>2</sub> gas for a few mins prior to the reaction. The reaction mixture was heated to 350 °C at a set stirring speed and maintained for 2 h, similar to conditions of previous studies (Hafriz et al., 2022). The decomposition of triglycerides generally occurs between reaction temperatures of 350 to 400 °C, whereby the C-O and C-C bond cleavage activities will be favoured and form a mixture of olefins, paraffins and aromatic hydrocarbons (Asikin-Mijan et al., 2022). After the designated time, the reaction vessel was allowed to cool down to room temperature before the N<sub>2</sub> gas flow was stopped. During the reaction, the volatile products were condensed at 5 °C and collected. The gaseous product was released without analysis. The WCO and DO products were characterised using gas chromatography mass spectroscopy (GCMS) analysis (Agilent 7890A-5975C). The fraction peaks from the chromatogram were identified via the National Institute of Standards and Testing (NIST) library. The hydrocarbon distribution for the deoxygenation products (alkanes and alkenes) are then split based on gasoline (<C<sub>10</sub>), jet fuel (C<sub>10</sub>-C<sub>16</sub>) and diesel (>C<sub>16</sub>) range fractions (Asikin-Mijan et al., 2022). The higher heating values of the samples were determined using a bomb calorimeter according to the ASTM D240 testing method.

### 3. Results and Discussion

Table 2 shows the BET and XRF analysis of the catalysts. The specific surface area (SSA) of the synthesized catalysts was in the order of TiO<sub>2</sub>>Ni>NiCo (S)>NiCo (C)>Co. It can be observed that the SSA of all the catalysts slightly decreased after being incorporated with the Ni and Co. This could have been caused by the active metal embedding and covering the pores of the support or the destruction of the pore structure (Zheng et al., 2023). Although the SSA of the produced catalysts was relatively small, the average pore diameter and pore volume was similar to that of other TiO<sub>2</sub> supported catalysts (Ai et al., 2023). After impregnation, the average pore diameter also showed slight increase. This may be due to the destruction of pore walls during calcination (Khalit et al., 2021). Besides the SSA, pore diameter and pore volume, the catalyst performance is also dependent on the active metal dispersion. Kohli et al. (2020) found that NiMo/γ-Al<sub>2</sub>O<sub>3</sub> had a better hydrotreating performance compared to NiMo/SBA-15 and NiMo/activated carbon due to the better metal dispersion (despite the latter having about twice SSA). In this study, XRF analysis (shown in the final 3 columns of Table 2) indicates that the measured NiO and Co<sub>3</sub>O<sub>4</sub> loading in catalysts were in accordance to the actual composition. This shows that majority of the catalyst metal was successfully impregnated onto the support.

Table 2: SSA, pore distribution and composition of catalysts measured by XRF

Catalyst	Specific surface area (m <sup>2</sup> /g)	Average pore diameter (nm)	Pore volume (cm <sup>3</sup> /g)	NiO (wt%)	Co <sub>3</sub> O <sub>4</sub> (wt%)	TiO <sub>2</sub> (wt%)
TiO	79.77	14.972	0.389	-	-	98.35
Ni	73.65	15.218	0.398	4.65	-	93.52
Co	65.38	15.928	0.345	-	4.79	93.40
NiCo (C)	67.30	15.175	0.345	2.44	2.51	93.87
NiCo (S)	71.09	15.196	0.357	2.42	2.52	93.88

From the GCMS analysis of WCO feedstock (Table 1), C<sub>16</sub> and C<sub>18</sub> carbon chain constitutes the majority of the fatty acids. Theoretically, DCO<sub>x</sub> and DCO of these fatty acids should produce a corresponding (C<sub>15</sub> and C<sub>17</sub>) alkane and alkene (Romero et al., 2018). Taking stearic acid, C<sub>17</sub>H<sub>35</sub>COOH, as an example, the DCO<sub>x</sub> products would be CO<sub>2</sub> and n-heptadecane, while the DCO products would be CO, water and heptadecane (Yildiz et al., 2020). Figure 1 shows the product distribution of the deoxygenation reaction. Ni catalyst showed the highest deoxygenation performance (89.03 %), followed by NiCo (C) (79.16 %), NiCo (S) (78.78 %), TiO (61.7 %) and Co (52.01 %). The deoxygenation performance of the Ni and Co catalysts was in accordance to the SSA of the catalysts.

The deoxygenation performance for Ni and both bimetallic NiCo catalysts are particularly impressive for deoxygenation without external hydrogen source. Baharudin et al. (2020) found that the NiO catalyzed deoxygenation of palm fatty acid distillate yielded 83 % of deoxygenated product, with an 86 % selectivity for diesel range hydrocarbons. Another hydrogen free deoxygenation study found that at optimised reaction conditions, the deoxygenation of WCO catalysed by NiO supported by modified dolomite was able to achieve 92.14 wt% of WCO conversion but only 68.86 % product yield (Hafriz et al., 2022). H<sub>2</sub> limited deoxygenation conditions lead to isomerization and dehydrogenation of deoxygenation products to form cyclic and aromatic

molecules, which is proven by the presence of aromatic compounds within each product. This is an important factor to consider as aromatic compounds improve fuel lubricity and compatibility with existing fuelling systems, but too much of it causes the fuel to have low energy density and high soot formation (Yunus et al., 2022). For both the TiO and Co catalysed deoxygenation, there remains a significant amount of fatty acids remaining in the product, which will increase the acid value of the product and worsen its cold flow properties (Hafriz et al., 2022). The Co catalysed deoxygenation showed poor deoxygenation performance, which has also been reported by Zeng et al. (2022), as  $\text{Co}_3\text{O}_4$  catalysts face difficulty for the deoxygenation of unsaturated fatty acids (such as the majority oleic acid). For the Ni catalysed deoxygenation, majority of the alkenes produced were identified as heptadecenes. This was similar to the findings of Mohammed et al. (2022), who suggested that the primary deoxygenation pathway of WCO were the hydrogenation of oleic acid to stearic acid then DCO of stearic acid to heptadecane.

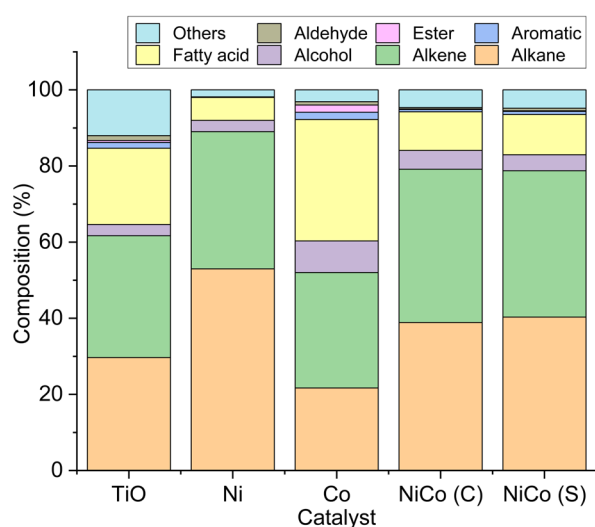


Figure 1: Composition of deoxygenated products for each catalyst

Figure 2 shows the selectivity for alkanes and alkenes for  $<C_{10}$ ,  $C_{10}-C_{16}$  and  $>C_{16}$  carbon chains. For the TiO reaction, the higher fraction of light hydrocarbons ( $<C_{10}$ ) indicate that majority of the fatty acids undergo cracking to form shorter chain hydrocarbons. The incorporation of Ni and Co catalysts improved selectivity towards jet fuel and diesel range as carboxylic acids are reduced to alkanes and alkenes. It is worth noting that for all Ni and Co reactions,  $C_{15}$  alkanes and alkenes was the dominant carbon chain produced, despite  $C_{18}$  fatty acids being the majority fatty acid chain. This scenario has also been reported by previous studies (Aliana-Nasharuddin et al., 2020), which attributed this to the cracking via C–C cleavage of heptadecane and heptadecene to form  $C_{15}$  hydrocarbons. The presence of these hydrocarbons in the product suggests that all Ni and Co catalysed reactions had a simultaneous deoxygenation and cracking pathway.

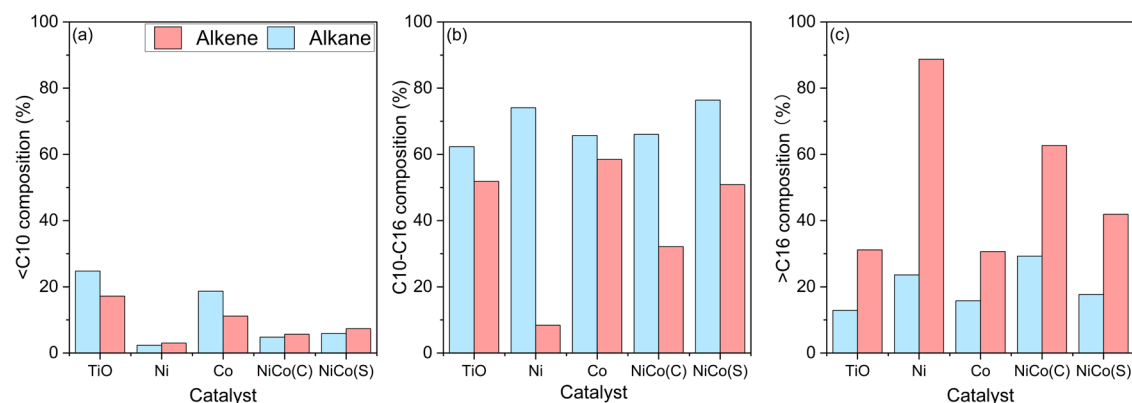


Figure 2: Carbon chain selectivity for alkanes and alkenes of each catalyst ((a)  $<C_{10}$  (gasoline), (b)  $C_{10}-C_{16}$  (jet fuel), (c)  $>C_{16}$  (diesel))

When considering the <math>C\_{10}</math> range of hydrocarbons (Figure 2(a)), TiO and Co showed the highest composition. This may be due to the cracking that occurs at elevated temperatures (>360 °C), causing more C–C cleavage and non-condensable products (Khalit et al., 2021). With regards to Co catalysed deoxygenation, since it has large acid-base sites which can help in deoxygenation and cracking, an excess of these sites will lead to overcracking, resulting in the formation of gaseous fractions and lower product yield (Asikin-Mijan et al., 2022). Alternatively, it could be possible to increase the reaction temperature or change the Co loading within the NiCo catalysts to promote cracking of hydrocarbons to improve the yield of  $C_{10}$ – $C_{16}$  hydrocarbons (Figure 2(b)). Besides that, the product selectivity for NiCo (C) and NiCo (S) is relatively different despite having similar catalyst loading and particle size. This change in product distribution can be attributed to the difference in oxygen vacancies, whereby more oxygen vacancies will lead to higher selectivity towards  $C_{16}$  and  $C_{18}$  hydrocarbons (Zheng et al., 2023).

As expected, the Ni and Co catalysed deoxygenation improved the HHV of the products, with monometallic Ni showing the highest HHV. It can be seen that all the deoxygenated products had a higher HHV than Jet A-1 fuel, but only Ni and NiCo (C) had HHV above the ASTM D1655 requirements. The increase in HHV is in accordance to the decrease in oxygen content, as high alkane and alkene content results in a higher HHV. Based on the tested catalysts in this study, NiCo (C) shows the most potential as the HHV of the deoxygenated product exceeds that of commercial jet fuels, with a suitable hydrocarbon composition. However, other fuel properties such as their acid value, viscosity, freezing point and flash point have yet to be tested to fully determine the suitability to replace commercial jet fuels.

*Table 3: HHV of the WCO, deoxygenated products and jet fuels*

Compound	HHV (MJ/kg)
Pure WCO	37.99
TiO	39.61
Ni	44.43
Co	39.28
NiCo (C)	43.48
NiCo (S)	42.15
Jet A-1	42.0 (Asikin-Mijan et al., 2022)
ASTM D1655	Minimum 42.8 (Asikin-Mijan et al., 2022)
Jet propulsion fuel 5 (JP5)	Minimum 42.6 (Yıldız et al., 2020)
Jet propulsion fuel 8 (JP8)	Minimum 42.8 (Yıldız et al., 2020)

#### 4. Conclusions

H<sub>2</sub> free catalysed deoxygenation provides a sustainable alternative for low cost renewable fuel production. In this study, the hydrogen free deoxygenation of WCO into fuel range hydrocarbons was investigated over unreduced monometallic and bimetallic NiCo/TiO<sub>2</sub> catalysts under atmospheric pressure. Among these catalysts, Ni catalysts showed the highest deoxygenation performance, but NiCo (S) had the best selectivity for jet fuel range hydrocarbons. The high surface area, high pore volume and good metal dispersion contributed to the high reaction efficiency of the Ni catalyst, but coimpregnation with Co catalysts allowed for simultaneous deoxygenation and cracking reactions to occur, improving reaction selectivity for potential jet fuel usage. It is recommended that the optimum Co loading within the NiCo catalyst can be investigated to optimise deoxygenation performance and hydrocarbon selectivity in future studies. The higher heating value of NiCo (C) deoxygenated products also exceeded the minimum requirement of ASTM D1655 standards. However, the findings in this study have not produced a direct jet fuel substitute, as it still requires the testing of other fuel properties to determine suitability for commercial application. Overall, bimetallic NiCo/TiO<sub>2</sub> catalysts showed promising reaction performance and selectivity for hydrogen-free WCO deoxygenation.

#### Acknowledgments

The authors would like to thank for the financial support from the research fund for International Excellent Young Scientists (52250610220) awarded by the National Natural Science Foundation of China (NSFC).

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