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Original Research Article

Rice Husk Char as a Catalyst Precursor for Biodiesel Production

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Abstract: The production of biodiesel from waste cooking oil has garnered significant attention due to its potential to address environmental and economic concerns using rice husk char as a catalyst, which presents a promising avenue for sustainable fuel development. The efficient optimisation of rice husk char (RHC) as a catalyst for biodiesel production from waste cooking oil presents challenges in achieving high catalytic activity and scalability. RHC was prepared, and the catalyst structural properties and morphology were characterised using Fourier-transform infrared (FTIR) spectroscopy and Scanning Electron Microscopy (SEM). The critical method involved the incorporation of RHC impregnated with titanium oxide as a catalyst precursor in the transesterification process for biodiesel production. Reaction parameters such as temperature, type of catalyst, and reaction time were systematically varied to assess their impact on the catalytic efficiency. Response surface methodology (RSM) was applied using Box-Behnken Design (BBD) in this study to maximise the transesterification reaction biodiesel production. The results showed that the catalyst produced the highest biodiesel yield calcined treated rice char, 72%. In contrast, the FTIR and SEM showed that titanium oxide was present, and the catalyst impregnation was promising.

Keywords: Biodiesel; Box-Behnken design; rice husk char; titanium oxide; transesterification

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1. Introduction

A – b Biodiesel is a promising alternative to petroleum diesel due to its environmental advantages, renewability, and lower exhaust emissions (Moreno-Caballero *et al.*, 2022). It is biodegradable, renewable, nontoxic, and lacks sulfur or aromatics, making it a strategic alternative to conventional diesel (Moreno-Caballero *et al.*, 2022). Rudolf Diesel introduced biodiesel as an alternative fuel during the First World War (Alex & Roy, 2020). Biodiesel, derived from vegetable oils, is a realistic alternative to conventional diesel, producing fewer harmful emissions and containing no polluting chemicals like sulfur (Alhassan & Aliyu, 2019). Biodiesel's non-toxic and biodegradable nature further supports its consideration as an alternative fuel (Rastogi *et al.*, 2021). Biodiesel, composed of alkyl monoesters of fatty acids, can be used in diesel engines without modification, making it a feasible alternative fuel source (Tat *et al.*, 2000). It has been shown that the simple alcohol esters of fatty acids in biodiesel are an acceptable alternative fuel for diesel engines (Haas *et al.*, 2003). Experimental biodiesel fuels are environmentally friendly and attractive alternatives to conventional diesel (Georgogianni *et al.*, 2007). Biodiesel has been identified as a renewable fuel source, offering considerable advantages over traditional fossil diesel (Gebremariam, 2023). Biodiesel is a promising, renewable, environmentally friendly biofuel that can be used in diesel engines with minimal modifications (Tesfa *et al.*, 2011). Microwave irradiation for biodiesel production highlights alternative energy sources for sustainable fuel production (Refaat *et al.*, 2008). Biodiesel offers a promising alternative to diesel oil, being renewable and sharing properties similar to diesel. Biodiesel has demonstrated superiority over conventional diesel fuel in terms of combustion efficiency, emissions cleanliness, cetane number, biodegradability, flash point, and lubrication (Hafeez *et al.*, 2020).

Biochar, an organic pyrolysis byproduct abundant in carbon, has existed for an extended period, potentially thousands of years—ancient. Ancient civilisations, such as the Amazonians, utilised biochar-based soil amendments to enhance agricultural productivity and fertility. Terra preta is an example of a historical application of biochar that demonstrates its potential to improve soil properties and sequester carbon. Biochar has been recognised as a valuable catalyst in biodiesel production, providing a sustainable and efficient alternative for biofuel synthesis. Research has indicated that biochar-based catalysts, derived from biomass waste, demonstrate high catalytic efficiency, with some achieving up to a 96% yield in biodiesel production (Nguyen, 2024). Biochar as a catalyst in biodiesel production is emphasised as an environmentally friendly alternative, utilising byproducts of the biodiesel industry for catalyst preparation (Tobío-Pérez *et al.*, 2022).

Additionally, studies have shown that biochar can enhance reaction kinetics, reduce reaction temperatures, and improve the overall efficiency of biodiesel production compared to traditional methods (Kim *et al*., 2022). Numerous studies have investigated the synthesis and application of biochar-based catalysts for biodiesel production, highlighting their effectiveness in transesterification reactions. Biochar has proven to be successful as a catalyst in the upgrading of bio-oil from pyrolysis, showcasing its versatility in biofuel production (Wang *et al*., 2022). Furthermore, biochar has been utilised as a catalyst in producing syngas and biodiesel from agricultural waste, demonstrating its potential in promoting sustainable energy practices (Kim *et al*., 2022). The application of biochar as a catalyst in biodiesel production extends to various feedstocks, including sewage sludge, peanut waste, and palm kernel shells, illustrating the adaptability of biochar in different production processes (Jung *et al*., 2022; Nuradila *et al*., 2017). Biochar has been used as a porous medium and alkaline catalyst for transesterification reactions, underscoring its role in facilitating biodiesel synthesis (Cho *et al*., 2021).

Rice husk and rice straw agricultural residues are generated from cultivation and processing. Rice husk, the outer layer of rice grains, and rice straw, the stalks left after grain harvesting, are abundant and widely available agricultural byproducts. Both materials are rich in cellulose, hemicellulose, and lignin, making them lignocellulosic biomass with potential as renewable resources. Rice husk char has been identified as a valuable catalyst in various applications, such as biodiesel production and adsorption studies. Chen *et al*. (2013) highlighted the potential of rice husk ash as a catalyst precursor for biodiesel production. Wang *et al*. (2020) emphasised the use of rice husk char as an additive for phosphate recovery, demonstrating its versatility in different processes.

Maiti *et al*. (2006) also focused on the physical and thermochemical characterisation of rice husk char, showcasing its potential as a biomass energy source. Moreover, Li *et al*. (2017) demonstrated the complete utilisation of rice husk to produce synthesis gas, with the biochar from rice husk pyrolysis catalytic reforming. Furthermore, Balasubramanian *et al*. (2021) highlighted the unique properties of charred rice husk as an adsorbent material with diffusion and catalytic properties.

2. Materials and Methods

2.1. Materials

The restaurant in Bintulu, Sarawak supplied the W,C O utilised this study. Unlike the yellow culinary oil, the restaurant's CO is a deep brown. Methanol, sulphuric acid (concentration of 95% to 97%), titanium dioxide (Merck KGaA, Germany), and paraffin oil (HmbG Chemicals, Hamburg, Germany) were utilised in this investigation. The rice husk was acquired from a local vendor in Bintulu, Sarawak.

2.2. Catalyst Preparation

A 40 g sample of RHC was pulverised with 400 mL of concentrated sulfuric acid (95–97%) in a 1000 mL vessel heated in an oil bath at 150°C for 12 hours. The RHC was ground into powder. Following the mixture had cooled to room temperature, it was combined with distilled water, agitated, and filtered. Following this, the precipitate underwent several washes with boiling distilled water until no sulfate ions remained in the filtrate. After filtration, the sample was dried in an RHC air-drying oven at 80 °C for twenty-four hours. This method was adapted from Azman *et al*. (2021) with slight modifications. Then the rice husk char was impregnated to synthesise the TiO_2: RHC catalyst based on the w/w ratio 20%:80% and 40%:60%, by constant stirring for six (6) hours. Afterwards, RHCt doped with the metals was calcined at 800°C for 4 hours.

2.3. Catalyst Characterisation

The catalyst was characterised using Fourier Transform Infrared Spectroscopy (FTIR) and scanning electron microscopy (SEM). The qualitative analysis of the sample was done using the FTIR spectroscopy technique using Shimadzu IRAffinity-1 Fourier-Transform Infrared Spectroscopy (FTIR). A scanning electron microscope (SEM) (JEOL 7500F-1) with a magnification of 10,000 was used to examine the surface morphology of the catalyst.

2.4. Experimental Design and Data Analysis

The optimisation of reaction parameters in the transesterification process was evaluated using response surface methodology (RSM) based on Box-Behnken design (BBD) with minor modification from Borah *et al*. (2019) with the Statistica software. The reaction parameters, i.e., Type of catalyst (X_1), temperature of the reaction (X_2), and reaction time ($X_{3)}$, were varied to obtain the maximum biodiesel conversion (Y). The experimental design consisting of experimental units is shown in Table 1. These experiments facilitated the study of the influence of each independent variable (Type of catalyst, temperature of reaction, and reaction time) and relations between these variables on the FAME conversion i.e., the dependent variable.

Table 1. Transesterification reaction parameter optimisation experimental design.

Run	Type of catalyst (X_1)	Temperature (˚C) (X_2)	Time (min) (X_3)	Yield (%)
1	RHCt	65	90	64
2	TR2	65	120	66
3	RHCt	85	90	50
4	TR2	85	120	64
5	RHCt	75	60	72
6	TR2	75	90	66
7	RHCt	75	120	36
8	TR2	75	120	66
9	TR1	65	60	66
10	TR1	85	90	48
11	TR1	65	90	52
12	TR1	85	60	52
13	TR1	75	90	56
14	TR1	75	120	64
15	TR1	75	60	54

2.5. Transesterification Reaction

The transesterification process reported by Borah *et al.* (2019) exhibits a minor alteration in the subsequent reaction. A 100-mL three-necked round-bottom glass flask equipped with a reflux condenser, magnetic stirrer, and thermometer was used to conduct the transesterification process. The molar ratio of methanol to oil employed in the experiment was 6:1. The duration required to complete the transesterification process was one hour. The separatory funnel was used to separate the reaction mixture.

3. Results and Discussions

3.1. Catalyst Characterisation

The FTIR spectra of the catalyst are presented in Figure 1. The peaks observed between 3600–3400cm^{-1} showed OH stretch, respectively. OH, stretch bending has been observed in all the catalysts. This outcome is supported by Hossain *et al.* (2020). The OH groups are associated with cellulosic material or water. It demonstrates that moisture is present in the biomass structure and that pyrolysis-related dehydration processes occur. Additionally, -OH groups are present in this area, caused mainly by silanols (Si-OH) adsorbed on the rice husk surface (Menya *et al.*, 2018; Bakar & Titilove, 2013). The bands observed around 1030 cm^{-1} can be attributed to CO, C=C, and CCO groups related to cellulose, hemicellulose, and lignin (Lazzari *et al*., 2018; Yuan *et al*., 2016). Around 1400-1300cm^{-1} showed the presence of titanium. This indicates the impregnation did occur. The early peak, around 500cm^{-1}, showed where the titanium was introduced into the rice husk char before it became fully incorporated (Al-Taweel & Saud, 2016).

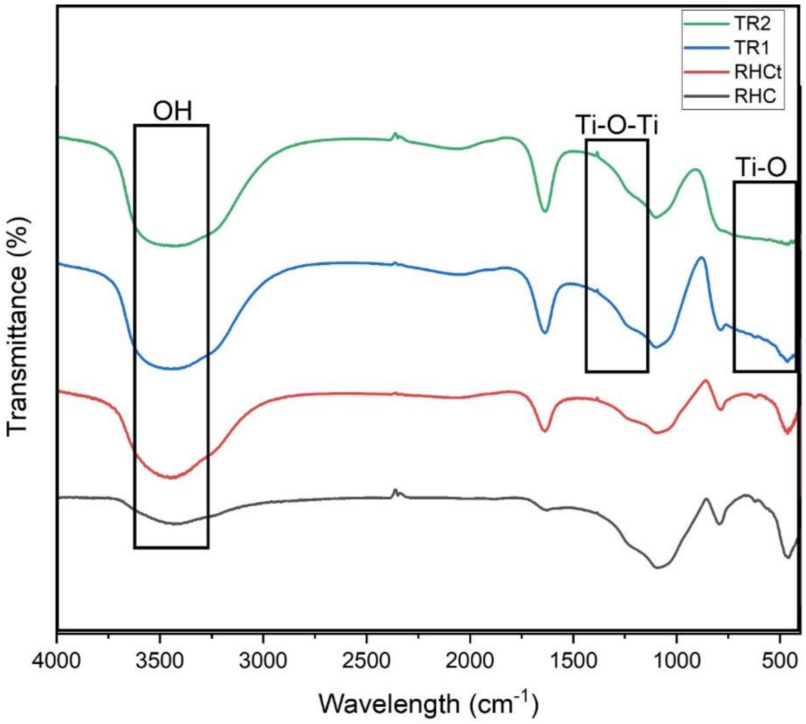

Figure 1. The FTIR spectra of the catalyst

A study of the catalyst surface morphological properties is conducted, as depicted in Figure 2. Figures 2 (a) and 2 (b) display scanning electron microscope (SEM) images of rice husk char and rice husk char treated. Figure 2 (a) illustrates the rice husk's smooth surface and compact structure. Figure 2 (b) illustrates the configuration of rice husk char after the pretreatment process. It showed that, after being treated, the rice husk becomes more porous, which enables it to be incorporated with other materials (Hossain *et al.*, 2020). Biochar's porous structure offers a substantial surface area, including many pores, encompassing both micro-pores and macro-pores (Chen *et al*., 2020). This characteristic enhances its porosity in comparison to untreated rice husk. The rice husk char has a porous structure, leading to a notable augmentation in surface area compared to its untreated counterpart. The augmented surface area of biochar facilitates more locations for chemical reactions, nutrient absorption, water retention, and microbial colonisation, rendering it a desirable compound for soil amendment and carbon sequestration. The porosity of rice husk char allows for improved water retention, nutrient absorption, and pollutant adsorption, making it a valuable material for soil improvement and environmental remediation (Niu *et al*., 2022). Figures 2 (c) and (d) show that the rice husk char is impregnated with titanium oxide. As can be seen, Figure 2(d) shows that titanium oxide is primarily present. The rice husk char was almost covered with titanium oxide, which indicated that the impregnation had successfully occurred. Impregnating rice husk char with titanium oxide can enhance its catalytic properties for various processes (Kusworo *et al*., 2023). The synergistic effects of rice husk char and titanium oxide impregnation can lead to the development of a versatile and efficient catalyst with potential benefits for environmental and industrial applications.

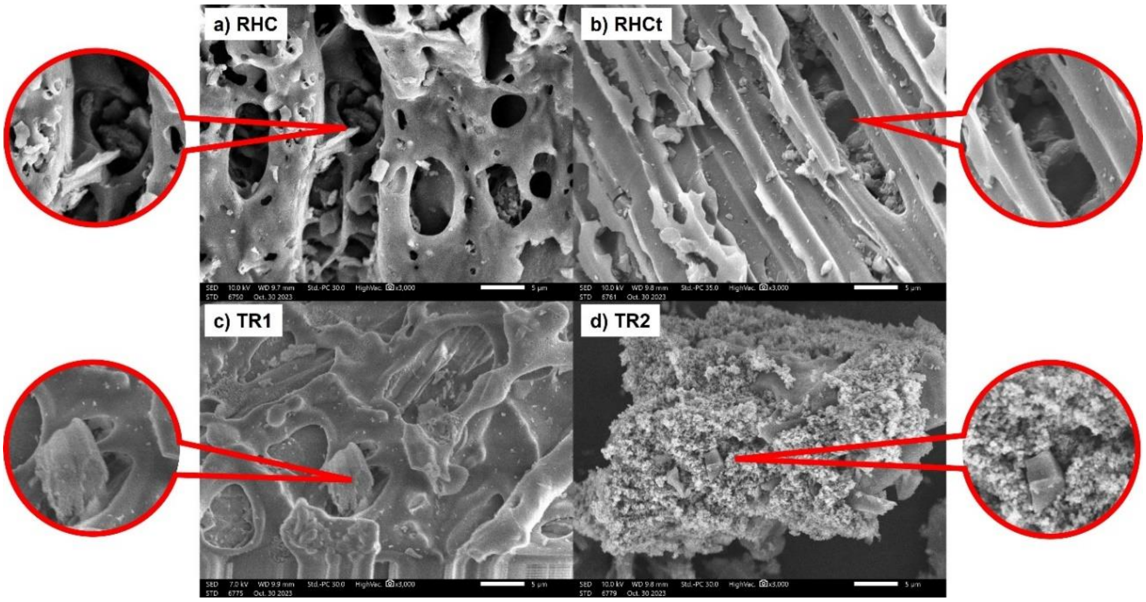

Figure 2. Catalyst morphology through SEM image.

3.2. The Impact of Operational Factors and Statistical Analysis on the Output of Biodiesel

Equation 1 examined the regression equation for biodiesel conversion (%) produced by regression analysis in coded terms.

$$Y= -9.21538+47.44487X_1-8.09103X_2-4.25000X_3 \quad (1)$$

The analysis of variance (ANOVA) was employed to investigate the model validity and assess the importance of variables and their interactions. The findings of this study are displayed in Table 2. The model F-value of 3.86 suggests a strong level of statistical significance. Furthermore, when the "Prob > F" values are below 0.0500, it indicates that the model terms are statistically significant. Table 3 presents the considerable model terms associated with the conversion of biodiesel, namely A, B, C, AB, AC, and BC. As seen in Table 2, the sequential sum of squares for temperature (144.50) surpasses that of time (38.27) and the type of catalyst (6.71), so temperature is established as the prevailing factor.

Figure 3 displays the three-dimensional surface plot of the catalyst type and temperature. The conversion of biodiesel increases when using the TR1 type of catalyst. But there is a decrease in conversion with an increase in temperature. On the other hand, RHCt showed that the higher the reaction temperature, the lower the yield produced. Higher temperatures can decompose biodiesel precursors such as triglycerides, fatty acids, and alcohol. Thermal breakdown can create unwanted byproducts, such as free fatty acids, reducing biodiesel output. Although high temperatures speed up biodiesel production processes, they can also have negative consequences that diminish output. Temperature must be optimised to maximise reaction rate and yield while minimising side reactions and energy expenditure.

Figure 4 below shows that biodiesel conversion decreases with the RHCt type of catalyst. However, there is an increase in conversion with increased reaction time. The surface plot for the interactive effect of catalyst and reaction time is shown in Figure 4 below. The conversion of biodiesel decreases when the RHCt catalyst is used. However, there is an increase in conversion with increased reaction time; the yield increases when using a TR1 catalyst. The potential synergistic effects resulting from the combination of rice husk char with titanium oxide can enhance catalytic activity. Titanium oxide is an active site for the transesterification reaction, while rice husk char promotes mass transfer and supplies supplementary catalytic sites due to its porous structure and surface area. Incorporating titanium oxide into rice husk char can enhance its surface area and porosity, augmenting the number of active sites available for the transesterification reaction. The surface area augmentation facilitates enhanced interaction between the catalyst and reactants, improving conversion rates and increasing biodiesel yields.

The surface plot represented in Figure 5 illustrates the interactive relationship between temperature and reaction time. The conversion of biodiesel goes down with an increase in temperature. However, there is an increase in conversion with an increase in reaction time. The pace of the transesterification reaction generally exhibits an increase at elevated temperatures, mainly attributed to the heightened mobility of reactants and the number of collisions. Nevertheless, elevated temperatures can also facilitate other processes, such as hydrolysis or saponification, thereby diminishing the biodiesel production output. The observed decline in conversion rates at elevated temperatures could be attributed to the heightened occurrence of side reactions that consume reactants and intermediates, resulting in diminished biodiesel production. In addition, elevated temperatures can lead to the thermal breakdown of the biodiesel inputs, such as triglycerides, resulting in a decrease in the amount of reactants available for the transesterification process. The more extended period of the reaction time facilitates a more significant duration for the transesterification reaction to reach its culmination, hence resulting in elevated conversion rates. Extended reaction durations offer further prospects for the transesterification reaction equilibrium to transition towards biodiesel production, thus augmenting the yield of the intended product. Additional side reactions may persist during extended reaction durations; nonetheless, the overall outcome of prolonged reaction times typically leads to an augmentation in biodiesel conversion, primarily attributed to the prevailing impact of the transesterification reaction.

Table 2. ANOVA analysis was conducted to determine the percentage of FAME conversion.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	964.28	6	160.71	3.86	0.0413 significant
A type of catalyst	6.71	1	6.71	0.1610	0.6988
B-Temperature	144.50	1	144.50	3.47	0.0996
C-Time	38.27	1	38.27	0.9186	0.3659
AB	12.00	1	12.00	0.2880	0.6061
AC	513.13	1	513.13	12.32	0.0080
BC	12.00	1	12.00	0.2880	0.6061
Residual	333.32	8	41.66		
Cor Total	1297.60	14			

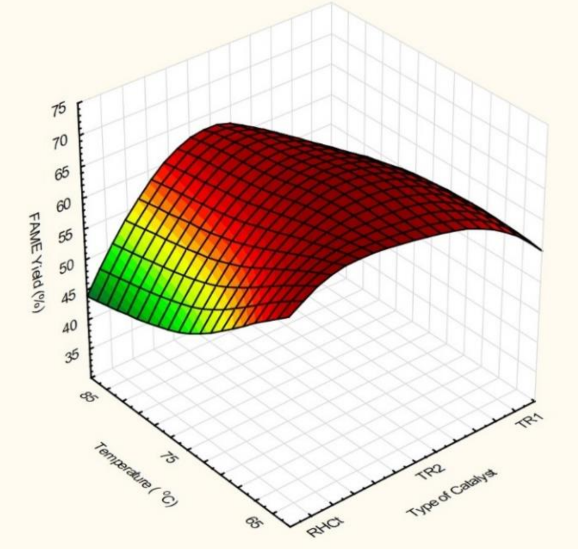

Figure 3. Surface plot for the interactive effect of catalyst type and temperature

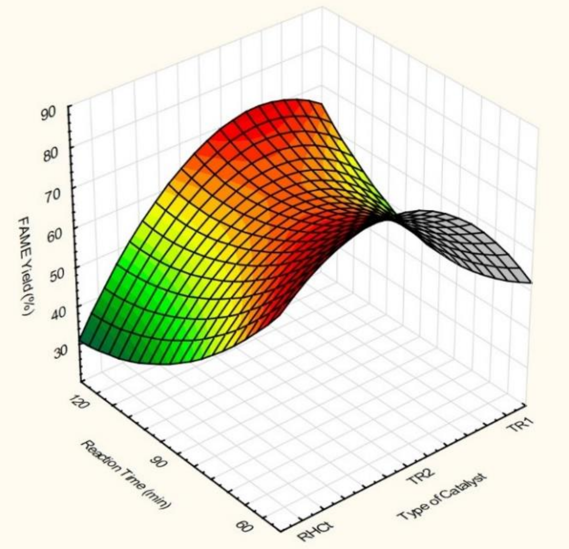

Figure 4. Surface plot for the interactive effect of the catalyst type and reaction time

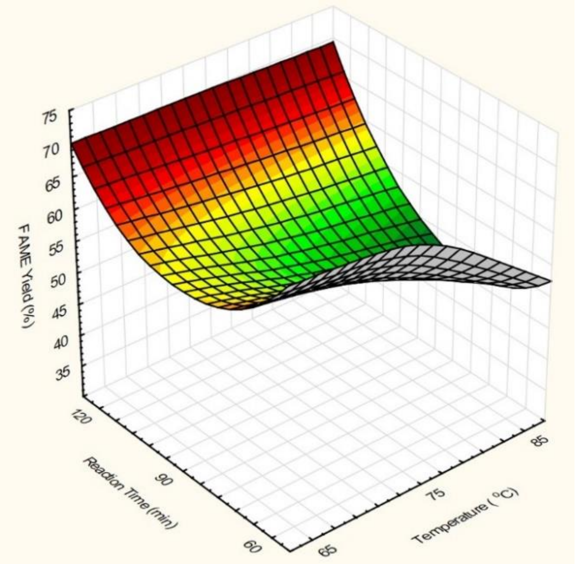

Figure 5. Surface plot for the interactive effect of temperature and reaction time

3.3 Characterisation of Biodiesel by Using FTIR

Figure 6 below shows the FTIR spectra of waste cooking oil and biodiesel. The FTIR biodiesel output and WCO spectra with various catalyst types in the mid-infrared range of 500 to 4000 are^{-1} is depicted in Figure 6. An FTIR analysis was performed to determine the proportion of biodiesel present in the biodiesel transesterification process, with consideration given to the specific catalyst employed. From a chemical perspective, biodiesel may combine mono-alkyl esters from long-chain fatty acids (C16–C18) obtained from vegetable oils and animal fats (da Silva *et al.*, 2017). The methyl ester peaks exhibited prominent bands corresponding to the C=O and O–CH3 groups, indicating the presence of the transesterification phenomenon. Identifying the O–CH$_3$ group is of utmost importance since it plays a crucial role in assuring the successful occurrence of transesterification. No significant peaks were seen in the bands 1800–2700 cm^{-1} and 3200–4000 cm^{-1} for biodiesel and WCO. Moreover, the spectra depicted in Figure 4 exhibited prominent peaks in specific bands of 2922.16 and 2852.72 cm^{-1} before C–H vibrations occurred. Biodiesel has the O–CH$_3$ group, but WCO does not. This suggests that WCO has been effectively transformed into biodiesel, as evidenced by the presence of the O–CH$_3$ group in the band 1188–1200 cm^{-1}. A previous investigation by Goli and Sahu (2018) showed that the O–CH$_3$ peak is at a wavenumber of 1170.16 cm^{-1}. The C–O group is observed in the peaks depicted in Figure 6, namely at bands 1159.22 cm^{-1}, which may be attributed to the category of (C–O) ester group stretching vibrations. A narrow band with a wavelength ranging from 1750 to 1500 cm^{-1} was generated by the cis C=C bond. The aliphatic groups of CH$_2$ and CH$_3$ were seen to have a band at 1460.11 cm^{-1}. Additional diagnostic peaks for biodiesel were seen at wavenumbers of 1743.65 cm^{-1}, precisely corresponding to the C=O group of glycerides. An inverse relationship exists between the quantity of free fatty acid and its propensity to convert into fatty acid methyl ester. The absorption sequence within the 600–1500 cm^{-1} range is often characterised by a high degree of complexity due to the diverse range of bending and stretching within the molecule.

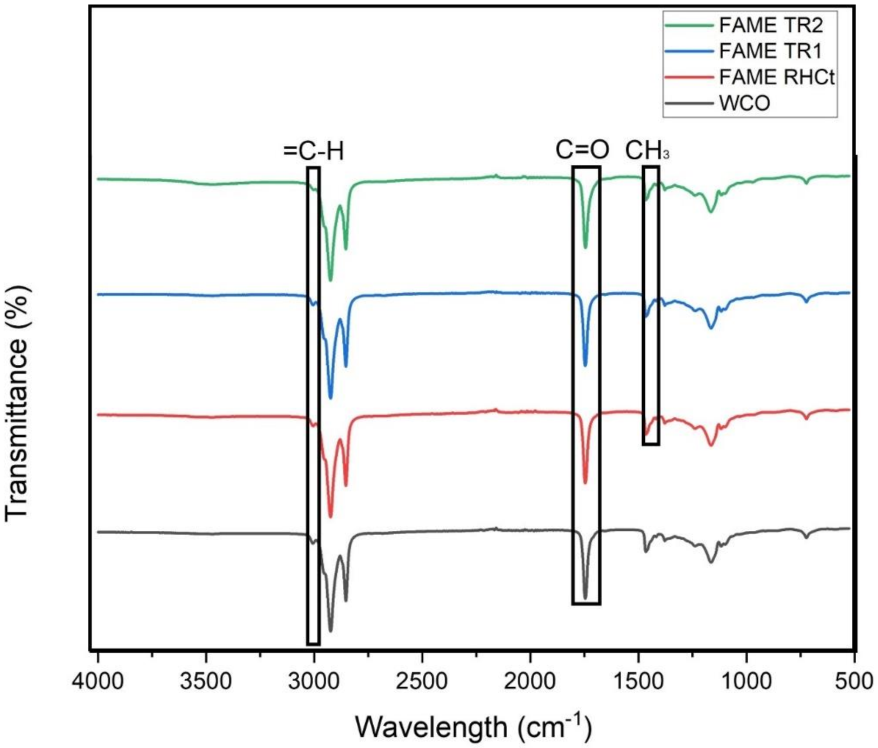

Figure 6. The FTIR spectra of WCO and biodiesel.

4. Conclusions

In summary, for all the catalysts that have been tested, Rice Husk Char (RHC), Rice Husk Char treated (RHCt), Titanium Dioxide/Rice Husk Char treated 20/80 (TR1), Titanium Dioxide/Rice Husk Char treated 40/60 (TR2), RHCt was found to be the most effective catalyst with a temperature of 75°C and a reaction time of 60 minutes. The yield found is 72%. Compared to previous studies, it has been found that the yield achieved was lower. The research also successfully determined the physical and chemical properties of the catalyst by visualising it under a scanning electron microscope (SEM) for four catalysts chosen: RHC, RHCt, TR1 and TR2, as shown in the result.

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Conflicts of Interest: The authors declare no conflict of interest.

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