Innovative Energy Systems and Technologies

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Green Thermodynamics for Biofuel Synthesis using Bambara Nutshell Catalyst: Toward Sustainable Energy Solutions

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Abstract

In this study, we discussed the green chemistry principle applied to the thermodynamics of biofuel production. As a case study, the study applies green chemistry principles to investigate the thermodynamics of biodiesel synthesis using a heterogeneous catalyst derived from Bambara nutshell (BNS), a renewable agro-waste material. The BNS catalyst was prepared through carbonization and sulfonation and characterized using fourier transform infrared spectroscopy (FTIR) and scanning electron microscope (SEM), revealing functional groups and porous morphology favorable for catalysis. Biodiesel was synthesized via esterification of oleic acid at temperatures ranging from 50-65 ℃. Thermodynamic parameters including activation energy, enthalpy, entropy, and Gibbs free energy were evaluated across first-, second-, and third-order kinetic models. Results showed that the reaction followed first-order kinetics with the best correlation (R^2 = 0.9980), lowest activation energy (51.64 kJ/mol), and most favorable thermodynamic profile–demonstrating low enthalpy (48.80 kJ/mol), less negative entropy (-148.11 J/mol·K), and the lowest Gibbs free energy across tested temperatures. These outcomes confirm the energy efficiency and feasibility of the process, also in support of green chemistry principle 1, 6, 7, 9 and 12. The integration of green chemistry with thermodynamic modeling highlights the potential of BNS as a sustainable, low-cost catalyst for environmentally friendly biodiesel production.

Keywords

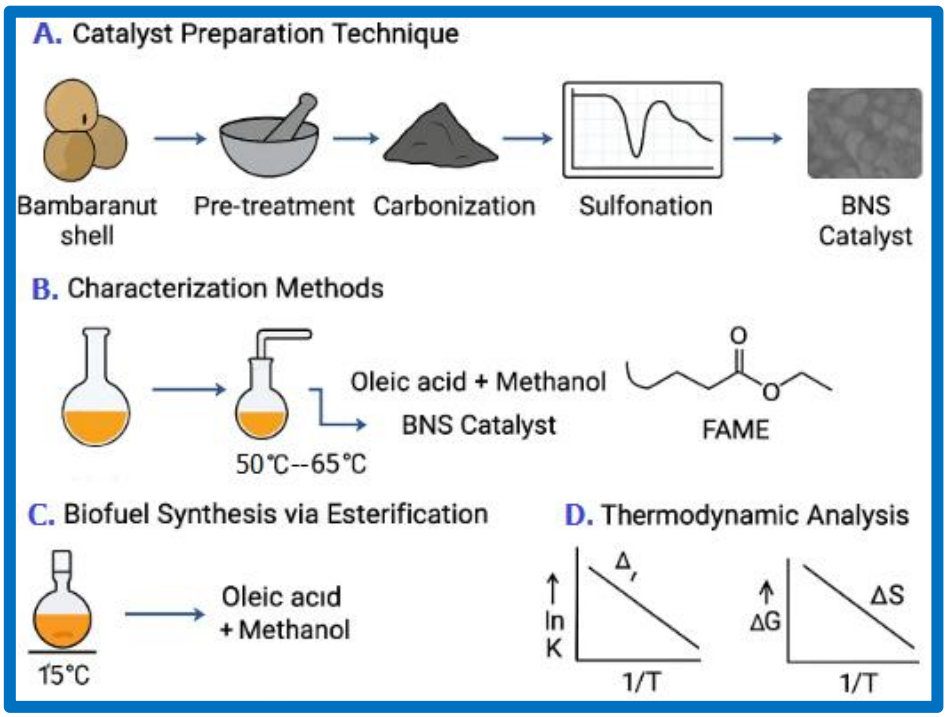
Bambara nutshell, Green chemistry, Biodiesel thermodynamics, Oleic acid, Biocatalyst

Article History

Received: 02 September 2025 Revised: 16 November 2025

Accepted: 03 December 2025 Available Online: 17 December 2025

Graphical Abstract



1. Introduction

In the synthesis of biofuels, green chemistry principles are believed to ensure environmental sustainability, safety, and efficiency [1,2]. In essence, green chemistry is a scientific approach that focuses on designing products and processes that minimize the use and generation of hazardous substances [3,4]. Here, biofuels are produced from biological sources such as vegetable oils, waste cooking oil, and agricultural residues, which are renewable and biodegradable. According to Bedenik & Zidak [5], it reduces dependence on fossil fuels and lowers greenhouse gas emissions. Another key principle is energy efficiency [6]. Green chemistry promotes processes that require less energy, such as using microwave-assisted [7-9] or enzymatic methods for biodiesel production [10], which operate at lower temperatures and pressures compared to traditional methods. The principle of waste prevention is also applied in biofuel synthesis, as well as in new feedstock generation and product substitution [11,12]. When *in-situ* transesterification or an integrating reaction and separation steps are utilized, less waste is generated. Table 1 identify the role of green chemistry in biofuel research, as well as other industrial domains. Utilization of green metrics that includes, atom economy, mass intensity/productivity, reaction mass efficiency and E-factor supports the design of safer and highly efficient biodiesel synthesis reactor [13-15]. Constable et al. [16] introduce us to a new metric called reaction mass efficiency. Those metrics were reportedly useful in optimizing biodiesel production from waste cooking oil [17]. In addition, using non-toxic and reusable catalysts, such as solid acid or base catalysts derived from biomass waste, aligns with the principle of safer chemicals and catalysis [18-20]. Catalysts and their design reduce the need for hazardous substances [21-23] and can be recycled or recovered [24]. Ningaraju et al. [25] exemplify the production of powdered biodiesel synthesis catalysts. Naveenkumar & Baskar [26] employed carbon efficiency, reaction mass efficiency, atom economy, environmental factor and stoichiometric factor, as green chemistry parameters for castor-oil biodiesel production using nano-catalyst. Via transesterification or other means, Outili et al. [27] and Gross et al. [28] accommodated the pretreatment stage and characterization of diverse waste cooking oils in biodiesel synthesis. In some cases, the knowledge of biotechnology or metabolic engineering are chipped in [29]. Economically, green chemistry in biorefinery process is the best step going forward [30,31]. Thus, in most manufacturing settings, it minimizes the cost of recovery and environmental health to ensure a circular economy [3,32]. Hjeresen et al. [33] and Idoko et al. [34] argues that contrary to the common myth, innovative technologies can enhance both environmental sustainability and economic profitability in industry. Hydrogenation, asymmetric catalysis, photo-catalysis, chemo-selective oxidation, nano-catalysis and electro-catalysis are methods that involve the use of metallic catalysts [35-39]. Therefore, green chemistry and biocatalysis are key to transforming the chemical industry of the future by enabling cleaner, safer, and more sustainable production processes [40,41].

Table 1. Areas where green chemistry principles are applied.

Area of Application	Description	Ref.
Pharmaceutical Industry	Designing safer drugs with fewer by-products and minimal use of hazardous solvents	[42-50]
Biofuel Production	Using renewable feedstocks, energy-efficient methods, and non-toxic catalysts.	[51-54]
Agriculture	Developing eco-friendly pesticides and fertilizers with minimal environmental impact.	[55-61]
Polymer and Plastics	Creating biodegradable and recyclable polymers from renewable resources.	[62-69]
Cleaning Products	Formulating non-toxic, biodegradable detergents and solvents.	[70,71]
Textile Industry	Replacing harsh dyes and chemicals with natural and safer alternatives.	[72-78]
Cosmetic and Personal Care	Using natural ingredients and eliminating harmful synthetic chemicals.	[79-84]
Chemical Manufacturing	Reducing waste through atom economy, catalysis, and process intensification.	[85,86]
Food Industry	Applying non-toxic preservatives and sustainable packaging materials.	[87-90]
Water Treatment	Using green oxidants and adsorption materials that are non-toxic and reusable.	[91-97]

Bambara nutshell (BNS) is the hard outer shell of the bambara groundnut, a legume scientifically known as *Vigna subterranea*. This nut is widely grown in sub-Saharan Africa, particularly in Nigeria, Ghana, Burkina Faso, and Cameroon, where it serves as a traditional food crop due to its high protein content and resilience to harsh conditions [98]. The nutshell is usually discarded as agricultural waste after the seeds are consumed or processed. Recently, researchers have explored the potential of bambara groundnut shell as a low-cost and eco-friendly bioenergy or biofuel source [99-101]. When treated through calcination or chemical activation, the nutshell is assumed to have the potential to produce materials rich in metal oxides and carbon, which are effective in catalyzing the transesterification of oils into biodiesel. Its high calcium and potassium content makes it especially suitable as a heterogeneous catalyst. Earlier on, Patzek [102] and Sohel & Jack [103] respectively introduced the application of First Law thermodynamics to analyze the production of biodiesel from soybean and other lignocellulosic feedstock. The thermodynamic study of biodiesel production is crucial for optimizing energy efficiency [104], determining reaction feasibility [105], and guiding the design of sustainable and cost-effective processes [106]. For example, Souza et al. [107] tested the thermodynamic parameters for simulation of palm oil biodiesel production using Aspen Plus. Though, not all biodiesel yield corresponds to the thermodynamic efficiency, according to the assessment conducted by Sohel & Jack [108]. Hence, the current study focuses on studying the thermodynamics of biodiesel production using oleic acid and BNS catalyst, in furtherance of the study on kinetic and optimization of the same acid and catalyst by Kefas et al. [109] and Kefas et al. [110], respectively. Apart from the aforementioned studies, there was no study that highlights the role of BNS in biodiesel production as catalyst. The present study will compute the activation energies, enthalpy, entropy and Gibbs-free energies involved in the esterification production of biodiesel using oleic acid and BNS catalyst. The goal is to produce an efficient process. Despite the sense, the nonsense, according to Ponton [111], lies in producing ethanol from grain or maize—particularly in Europe and the USA—because the process is thermodynamically inefficient, uses only part of the plant, and yields less energy than direct combustion. This study addresses the void in thermodynamic information for biodiesel extraction from most feedstock or catalyst, stated as a significant research gap by Khalighi [112].

2. Methods

2.1 Catalyst Preparation Technique

The bambara nut catalyst was prepared through a multi-stage process involving pretreatment, carbonization, and sulfonation. Initially, the BNS were thoroughly washed to eliminate surface impurities and then dried in an air-blast oven at 105 ℃ to reduce moisture content. The dried shells were ground and sieved to a particle size of 0.125 mm. Approximately 18 g of the sieved BNS powder was carbonized in a furnace at 300 ℃ for 150 min under an inert nitrogen atmosphere. Following carbonization, 4 g of the resulting biochar was subjected to sulfonation by mixing with 100 mL of concentrated sulfuric acid (H_2SO_4) and heated in an oil bath at a temperature of 95 ℃ while being magnetically stirred for 350 min under a continuous nitrogen flow. Post-sulfonation, the sample was repeatedly washed with distilled water to remove residual sulfonate ions and acid impurities. The washed catalyst was then dried in an oven at 105 ℃ for 720 min to remove adsorbed moisture and subsequently stored for characterization and use.

2.2 Characterization Methods

The characterization of the BNS catalyst was carried out using fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) techniques to determine its chemical functionality and surface morphology. FTIR spectroscopy was conducted in the range reported in Sahani et al. [113] and as conducted by Noreen et al. [114]. That is, 400-4000 cm^{-1} to identify the presence of surface functional groups responsible for catalytic activity. SEM analysis was carried out at an accelerating voltage of 15 kV to examine the surface morphology of the catalyst.

2.3 Biofuel Synthesis via Esterification Process

The synthesis of biodiesel via esterification using the BNS catalyst was conducted in a controlled batch setup. A 250 mL three-neck round-bottom flask equipped with a magnetic stirrer and a reflux condenser was used as the reactor

[115]. In each run, a specific quantity of oleic acid, methanol, and the prepared BNS-based heterogeneous acid catalyst was introduced into the reactor. The methanol-to-oleic acid molar ratio was maintained at 10:1, and the catalyst loading was set at 3 wt.% of the total reaction mixture. The reaction mixture was stirred at 600 rpm to ensure proper mixing, almost close to 700 rpm used by Feyzi et al. [116]. In contrast, the reaction temperature was maintained at various set points of 50, 55, 60 and 65 ℃ using an atmospheric pressure microwave heating system. Zanjani et al. [117] employed a temperature between 45-55 ℃. But in some studies, 65 ℃ is the optimal temperature employed [113,118,119]. The reaction was allowed to proceed for 180 min, with samples withdrawn at predefined intervals of 1, 5, 10, 30, 60, 90, 110, 130, 150, and 180 min for analysis. Upon reaching each time interval, approximately 7 mL of the reaction mixture was collected into a centrifuge tube and immediately cooled in ice to halt the reaction. After completion of the reaction, the catalyst was separated from the mixture by filtration. Excess methanol and the water formed during the reaction were removed from the product mixture through vacuum distillation. The resulting biodiesel layer was analyzed to determine the free fatty acid (FFA) conversion using the AOCS Ca 5a-40 method. The FFA values of the feed and product were used to calculate the percentage conversion of oleic acid to fatty acid methyl ester (FAME), indicating the catalytic efficiency of the BNS catalyst in the esterification process.

2.4 Thermodynamic Analysis

Thermodynamic analysis of the esterification reaction was conducted to determine the activation energy, enthalpy, entropy, and Gibbs free energy associated with the conversion of oleic acid to biodiesel. The rate constants at different reaction temperatures of 50, 55, 60 and 65 ℃ were first calculated from kinetic experiments assuming first-, second-, and third-order reaction models, in accordance with Kefas et al. [109]. The Arrhenius equation was applied in its linearized form by plotting the natural logarithm of the rate constant ($\ln k$) against the reciprocal of absolute temperature (1/T in K^{-1}). From the slope and intercept of the Arrhenius plots, the activation energy (E_a) and the pre-exponential factor (A) were determined. Subsequently, the Eyring equation employed by Abubakar et al. [120], in its linearized form, was used to estimate the enthalpy (ΔH) and entropy (ΔS) of activation. This was achieved by plotting $\ln(k/T)$ against 1/T and calculating the slope and intercept, from which ΔH was derived from the slope (-ΔH/R) and ΔS from the intercept ($\ln(k_B/h) + \Delta S/R$) [121]. Here, R is the universal gas constant, k_B is Boltzmann's constant, and h is Planck's constant. The Gibbs free energy of activation (ΔG) was then computed at each reaction temperature using the relation $\Delta G = \Delta H - T\Delta S$. To garner insights into the energy profile and molecular orderliness of the reaction system, the determined thermodynamic parameters are then interpreted. Afterwards, the equilibrium constant, K^* was computed using the expression $e^{-\Delta G/RT}$.

3. Results and Discussion

3.1 Catalyst Characterization

FTIR spectrum of the BNS catalyst reveals the presence of several key functional groups that contribute to its catalytic potential in biofuel production. A broad peak observed around 3444 and 3349 cm^{-1} corresponds to -OH and -NH stretching vibrations, indicating the presence of hydroxyl and amine groups. Such groups are often associated with improved adsorption and catalytic interaction with reactants during transesterification reactions. Another peak at 2944 cm^{-1} is attributed to C-H stretching, which suggests the presence of aliphatic hydrocarbon chains. The appearance of peaks around 1659 and 1638 cm^{-1} reflects C=O stretching and C=C or N-H bending, respectively, which are commonly found in lignin-based or carbonaceous materials derived from biomass. It supports the idea that the BNS underwent thermal and chemical changes to form active carbon-like or ash-supported catalytic surfaces. In Table 2, the band near 1106 and 1068 cm^{-1} indicates C-O or C-N bonds, which confirms the presence of ether or amine functionalities. They are believed to participate in hydrogen bonding or surface interactions that improve the thermodynamic favorability of ester bond formation in biodiesel synthesis. Other peaks such as those at 791 and 677 cm^{-1} are associated with aromatic or out-of-plane bending, both highlighting the complex but reactive surface of the catalyst employed.

As such, the FTIR spectrum confirms that the BNS catalyst contains a rich variety of surface functional groups, many of which are capable of facilitating low-energy, solvent-free biofuel synthesis. This aligns directly with green chemistry principles. Principle 1 on prevention, is the use of an agricultural waste-like BNS prevents waste accumulation. Next, 'Principle 7' involving the use of renewable feedstocks, is because the catalyst itself is derived from a renewable biomass source. In 'Principle 6', which is 'design for energy efficiency', the presence of reactive sites reduces the energy barrier for biodiesel production. As for 'Principle 9', which is catalysis, the catalyst enhances reaction rates and selectivity without being consumed. Principle 12 has to do with an 'inherently safer chemistry'. That is to say, using naturally derived catalysts herein, limits toxicity and hazard risk. Figure 1 is a SEM image taken at 100 μm magnification, while Figure 2 is taken at a lower magnification of 500 μm. The main difference is the level of detail. Evidently, Figure 1 shows a closer, more detailed view of the surface, whereas Figure 2 shows a broader, more general view of the catalyst particles and their overall structure.

Table 2. BNS catalyst ftir peaks.

S/No.	Wavenumber (cm^{-1})	Observation/Note
1.	3966.5796	Broad peak (O-H/N-H stretching)
2.	3444.9798	Strong, likely O-H stretching (alcohols)
3.	3349.5111	N-H stretching (amines)
4.	2944.4404	C-H stretching (alkanes)
5.	2715.3777	Possibly aldehyde C-H stretch
6.	2476.487	Possibly CO_2 or -SH group
7.	2365.0312	CO_2 or atmospheric interference
8.	2000.2434	Possibly triple bond C≡C stretch
9.	1894.6182	C=O overtone region
10.	1659.6484	C=O stretching (amide I)
11.	1638.8687	C=C or N-H bending (amide II)
12.	1466.6667	C-H bending (methylene group)
13.	1106.5451	C-O or C-N stretching
14.	1068.8566	C-O stretching (ethers)
15.	791.7657	Aromatic C-H bending
16.	677.3359	Out-of-plane bending (C-H)

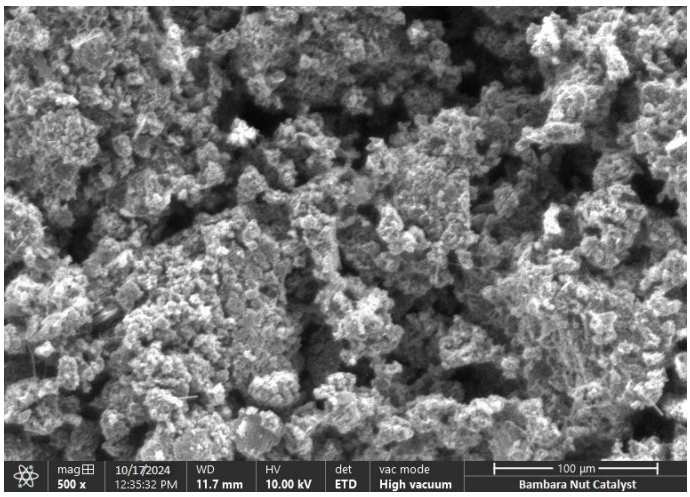


Figure 1. A 100 μm BNS catalyst SEM image.

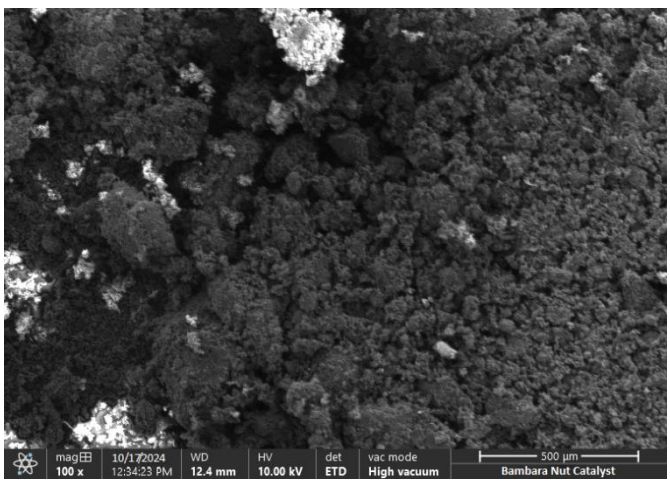


Figure 2. A 500 µm BNS catalyst SEM image.

Figure 1 reveals fine surface features comprising of pores, cracks, and rough textures on the catalyst surface. They are important for understanding how the catalyst can interact with reactants. Pores increase the surface area, which is critical for catalytic activity. More surface area means more active sites are available for the reaction. Additionally, the cracks and uneven texture help in adsorbing and interacting with reactant molecules during the esterification process. On the other hand, Figure 2 reveals the overall shape, particle size distribution, and macro-structural arrangement of the catalyst. It helps in assessing the uniformity, agglomeration, or clustering of the particles. While SEM shows morphology and surface structure, it does not provide chemical information. It does not indicate thermal stability, acidity, or reusability of the catalyst. Also, SEM cannot quantify how well the catalyst performs during the reaction—it

only gives a visual suggestion. Specialized analysis, involving Brunauer-Emmett-Teller (BET), X-Ray Diffraction (XRD) and Thermogravimetric Analysis (TGA) are needed to add the missing ingredient, as conducted by Saikia et al. [122]. Despite that, the visible porosity supports green chemistry by implying low-energy pathways and efficient reactant conversion.

3.2 Thermodynamic Parameters

Figures 3 (a-c) show Arrhenius plots for first-, second-, and third-order reactions, respectively. Each graph plots $\ln k$ against 1/T to determine activation energy [123]. In the first-order plot (Figure 3a), it is observed that it has a nearly perfect straight line, showing a strong correlation. Majority of studies, including those by Salim et al. [124], aligns with the first-order description. The second-order (Figure 3b) also follows a linear trend but less accurately. Third-order (or Figure 3c) plot shows a scattered and weakly linear trend. Figure 1c looks different because the third-order model does not fit the data well. The R^2 value is low, and the points do not align in a straight line, which suggests that the reaction does not follow third-order kinetics. It is unlikely that simple adjustments can make the data follow a third-order model. The deviation likely comes from the actual mechanism of the reaction. However, using different experimental conditions, catalysts, or higher accuracy measurements might offer better alignment—but this is speculative. Presumably, the rate constants (k) change with temperature for each reaction order., so that k_1 and k_2 increase consistently with temperature. Basically, it supports the expected behavior of the reactions. However, k_3 values are less predictable.

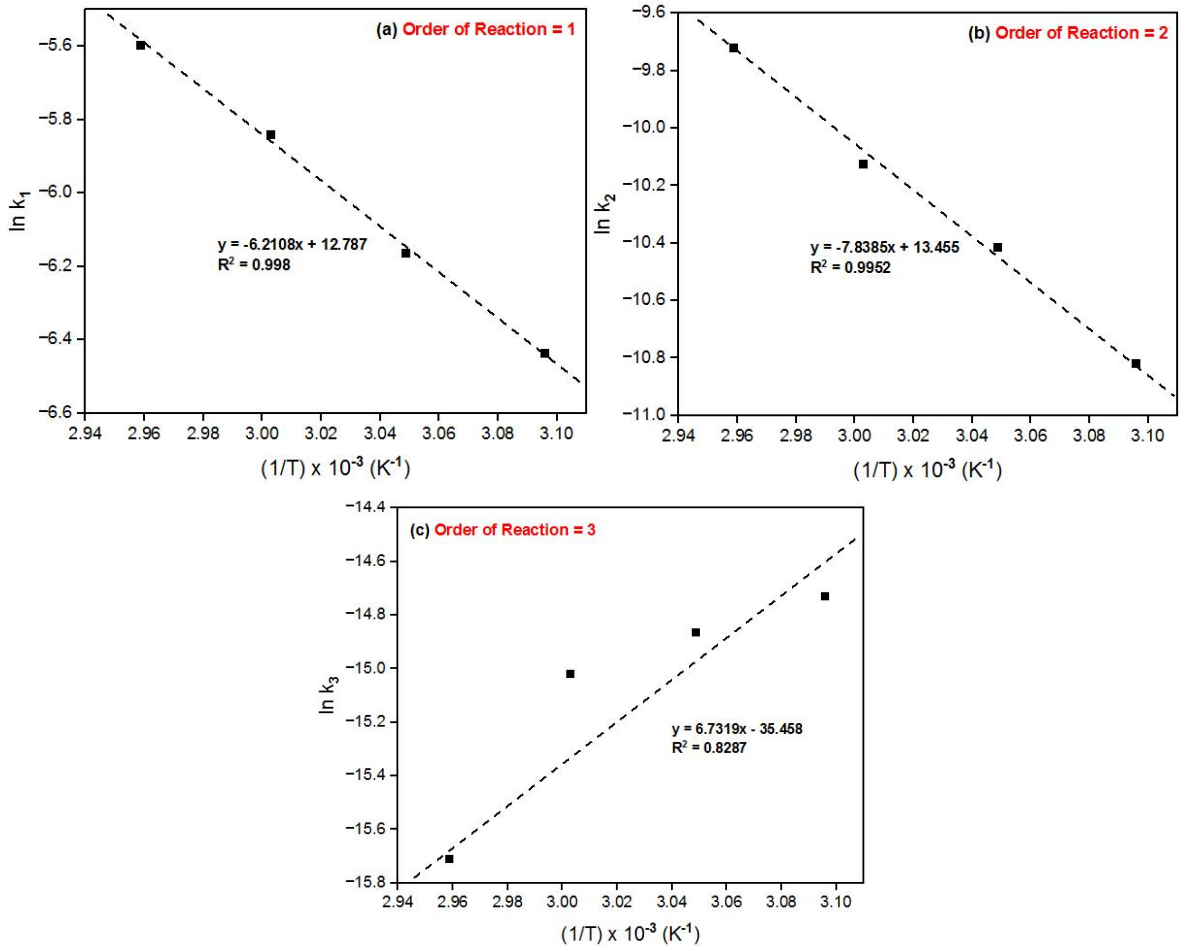


Figure 3. Arrhenius plot based on presumed reaction order: (a) 1, (b) 2, and (c) 3.

From Figure 3 and Table 3, it is observed that the first-order reaction model has the highest R^2 value of 0.9980. Therefore, it best explains the experimental data compared to second-order (R^2 = 0.9952) and third-order (R^2 = 0.8287) models. Using the same microwave heating employed in this study, Farid et al. [125] found that the first order model is the best. A positive E_a means that the reaction needs an input of energy to start; this is common in most chemical reactions. A negative E_a, which is not observed in this study, would imply that the reaction becomes faster as temperature decreases, which is rare and often suggests a complex or surface-driven mechanism. Clearly, E_a= 51.64 kJ/mol is lower in the first-order reaction because the reaction mechanism involves fewer molecular interactions and requires less energy to proceed. In the second-order case, the reaction may involve more complex interactions between molecules, thus needing more energy (up to 65.17 kJ/mol) to reach the transition state. In this situation, it reflects the nature of the reacting species and the pathway they follow. The k_o indicates how often molecules collide in the right orientation to react. A far lower k_o value of 3.02 min^{-1} was reported by Yatish et al. [126] during thermodynamics of biodiesel synthesis using *Butea monosperma* oil. A high value means frequent and effective collisions. While high k_o

can support faster reactions, it must be considered along with E_a. In this study, the reported k_o values; especially for n = 1 and 2 are too high compared to 7.94 min^{-1} in Sahani & Sharma [127]. A closer k_o value from previous study by Rodrigues et al. [121] is 4.35×10^8 min^{-1} or 2.61×10^{10} h^{-1}. A lower k_o as in 3.988×10^{-16}, doesn't mean poor performance, especially if the catalyst facilitates lower energy barriers.

Table 3. Arrhenius kinetic parameters computed.

Order	Parameter	Value
n = 1	E_a (kJ/mol)	51.64
	k_o (min^{-1})	3.575×10^5
	R^2	0.9980
n = 2	E_a (kJ/mol)	65.17
	k_o (min^{-1})	6.973×10^5
	R^2	0.9952
n = 3	E_a (kJ/mol)	-55.97
	k_o (min^{-1})	3.988×10^{-16}
	R^2	0.8287

Table 3 shows that the first-order reaction has a relatively low E_a. By implication, less energy is needed to drive the process, which aligns with the green chemistry principle of energy efficiency (Principle 6). Using a catalyst that lowers E_a supports environmentally friendly processes by reducing the overall energy demand. Several studies have computed the foregone Arrhenius constants in their respective studies, as shown in Table 4. Apart from food oils explored by Noreen et al. [114], non-food oils can be employed [128]. A general trend observed is that first-order kinetics is the most frequently reported and favored reaction order across multiple feedstocks and catalyst types. It suggests that the transesterification reaction often follows a pseudo-first-order mechanism when alcohol is in excess. The E_a values vary widely–from as low as 12.12 kJ/mol for rapeseed oil with K_2CO_3/Al_2O_3 catalyst, to as high as 77.6 kJ/mol for sunflower oil using $MgO\text{-}La_2O_3$ nanocatalysts. Such variation can be attributed to differences in catalyst nature, oil properties, and reaction conditions. Notably, lower activation energies typically reflect higher catalytic efficiency and easier reaction pathways, as seen with catalysts like sulfuryl chloride (25.05 kJ/mol for *Datura metel* oil) and barium lanthanum oxide (34.44 kJ/mol for Mahua oil).

Table 4. Previous report on activation energies determined for various catalyst.

Best Order	Activation Energy (kJ/mol)	Acid or Oil Type	Catalyst	Ref.
1.287	12.12	Rapeseed oil	K_2CO_3/Al_2O_3	[117]
1	72.86	Sunflower oil	Al-Sr nanocatalysts	[119]
1	34.44	*Madhuca indica* (Mahua) oil	Barium lanthanum oxide	[127]
1.5	27.06	Soybean oil	CH_3ONa	[129]
2	77.6	Sunflower oil	$MgO\text{-}La_2O_3$ nanocatalysts	[116]
1	50.1 & 48.55	Cooking oil and castor oil	Potassium modified ceria oxide	[130]
1	57.82	Cooking oil	NR	[131]
NR	19.15	Cooking oil	Spent coffee grounds	[132]
1	59.31	Fish oil	Nano magnetic	[115]
1	25.051	*Daturametel Linn* seed oil	Sulfuryl chloride	[133]
NR	33.14	*Moringa oleifera* oil	$KOCH_3$	[134]
1	42.5	Soybean oil	Calcium oxide-magnetite nanocomposites	[135]
2	28.35	Candlenut oil	Non-catalytic	[136]
1	51.64	Oleic acid	BNS	This Study

Based on the linearity and R^2 values in Figure 4 and Table 5, it can be said that the first-order ($R^2 = 0.9976$) gives the 'best fit', second-order with $R^2 = 0.9951$ is describe as portraying a 'good fit' and, the third-order model presents a 'poor fit'. In Shalfoh et al. [137]'s study using sodium hydroxide (NaOH) as catalyst, the second-order model performed best. Again, Figure 3 (Arrhenius plot) and Figure 4 (Eyring plot) both shows how well each reaction order fits the experimental data. In both figures, the first-order reaction shows the most linear behavior, indicating consistent kinetics and thermodynamics. The third-order plots in both figures are scattered, showing a poor fit. This consistency between figures confirms that the reaction is best described by first-order kinetics.

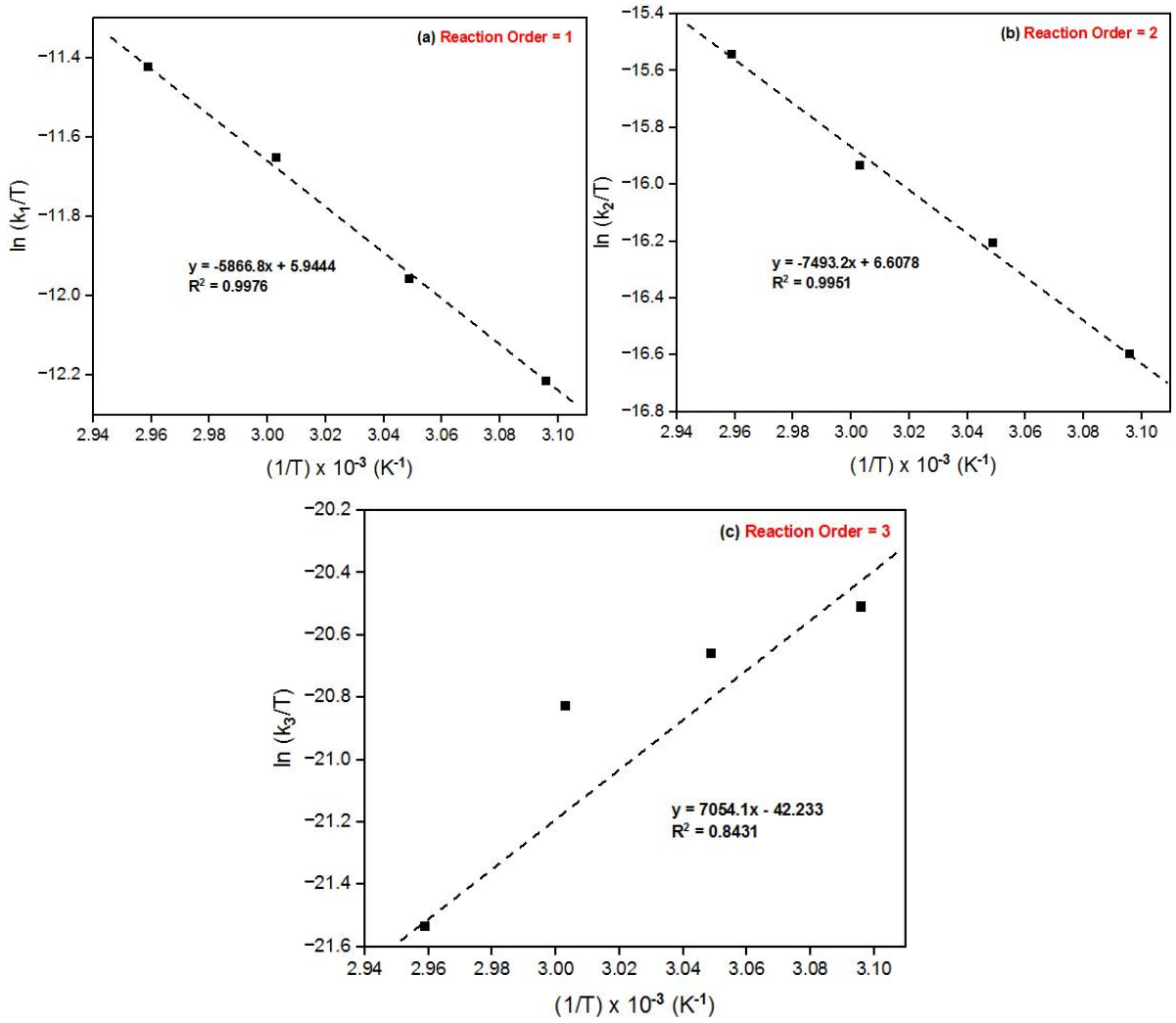


Figure 4. Graphical determination of enthalpy and entropy energy changes: (a) 1, (b) 2, and (c) 3.

Moreover, the first-order reaction performed best in Table 5 with the lowest enthalpy of 48.80 kJ/mol and the best R^2 value. The enthalpy increases in the second order (62.29 kJ/mol) because more energy is required to initiate and sustain interactions between multiple reacting molecules. Thus, making the system less efficient thermodynamically. A positive enthalpy means the reaction is endothermic—it absorbs heat from the surroundings [136]. This can still be favorable in green chemistry if the reaction runs at mild temperatures and the energy input is from renewable sources. Negative ΔH would imply an exothermic reaction, typically more energy-efficient. In this case, even though the enthalpy is positive, it supports green chemistry because the energy required is not excessive, and the catalyst helps reduce overall energy demand. It is better for enthalpy to be low in biodiesel production (as in order 1). Lower ΔH means less energy is needed to drive the reaction, which is more energy-efficient and cost-effective—thus, in line with green chemistry attributes of low energy consumption. As against this, Feyzi & Shahbazi [119] and Feyzi et al. [116] respectively obtained a ΔH = 215.02 and 162 kJ/mol higher than in this study.

A negative ΔS means that the system becomes more ordered as the reaction proceeds, as also observed in -241.25 J/mol value obtained by Mathiarasi & Partha [133]. In this esterification process, the reaction moves from two reactants (oleic acid and methanol) to a single biodiesel product, which leads to a decrease in randomness or molecular disorder. So, the negative ΔS here is expected and acceptable. The first-order reaction has the least negative ΔS (-148.11 J/mol·K), while the third-order has the most negative value (-548.82 J/mol·K). A less negative (or closer to zero) entropy is more favorable because it indicates less loss of disorder. Therefore, first-order is best in terms of ΔS. Higher (less negative) ΔS is preferable, because it suggests less restriction in molecular motion and energy distribution, which can support a more spontaneous and efficient reaction. Extremely negative entropy, as in -284.6 J/mol.K in Sun et al. [138] means the system becomes too ordered, which is usually less favorable energetically. Since ΔS is negative for all orders, as temperature (T) increases, -TΔS becomes more positive, thereby increasing ΔG. This explains why Gibbs free energy increases with temperature in all cases in Table 5. According to Nautiyal et al. [139], the negative value of ΔS and the positive values of ΔG and ΔH, explains why the reaction is unspontaneous and endergonic. The moderate negative ΔS and positive ΔG values indicate that the reaction becomes more ordered and requires energy input, which aligns with green chemistry. Figure 5 depicts the increase in ΔG with temperature surge for all order of reaction. Across all reaction

orders, the ΔG values remain positive within 50-65 ℃. It points to the fact that the esterification is non-spontaneous and requires an external energy input; however, the magnitude and trend of ΔG vary with temperature and reaction order.

Table 5. Enthalpy, entropy and Gibbs free energy changes determined.

Order	Parameter	Value
n = 1	ΔH (kJ/mol)	48.80
	ΔS (J/mol.K)	-148.11
	ΔG at 50 ℃ (kJ/mol)	96.66
	ΔG at 55 ℃ (kJ/mol)	97.40
	ΔG at 60 ℃ (kJ/mol)	98.14
	ΔG at 65 ℃ (kJ/mol)	98.88
	K^* at 50 ℃	2.33×10^{-16}
	K^* at 55 ℃	3.14×10^{-16}
	K^* at 60 ℃	4.21×10^{-16}
	K^* at 65 ℃	5.54×10^{-16}
	R^2	0.9976
n = 2	ΔH (kJ/mol)	62.29
	ΔS (J/mol.K)	-142.63
	ΔG at 50 ℃ (kJ/mol)	108.39
	ΔG at 55 ℃ (kJ/mol)	109.11
	ΔG at 60 ℃ (kJ/mol)	109.84
	ΔG at 65 ℃ (kJ/mol)	110.57
	K^* at 50 ℃	3.04×10^{-18}
	K^* at 55 ℃	4.00×10^{-18}
	K^* at 60 ℃	5.27×10^{-18}
	K^* at 65 ℃	6.82×10^{-18}
	R^2	0.9951
n = 3	ΔH (kJ/mol)	-58.64
	ΔS (J/mol.K)	-548.82
	ΔG at 50 ℃ (kJ/mol)	118.80
	ΔG at 55 ℃ (kJ/mol)	121.55
	ΔG at 60 ℃ (kJ/mol)	124.30
	ΔG at 65 ℃ (kJ/mol)	127.04
	K^* at 50 ℃	5.30×10^{-20}
	K^* at 55 ℃	3.48×10^{-20}
	K^* at 60 ℃	2.59×10^{-20}
	K^* at 65 ℃	1.98×10^{-20}
	R^2	0.8431

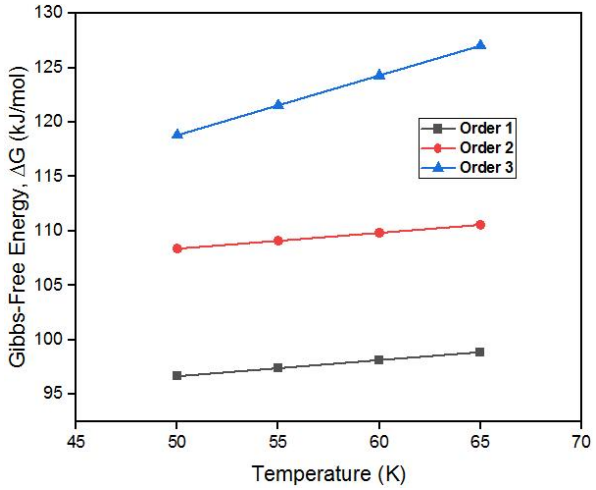


Figure 5. Change in Gibbs free energy with reaction temperature rise.

Higher Gibbs free energy is not preferable. As reported by Farid et al. [125] and Tulashie & Kotoka [134], a lower or negative ΔG is more favorable because it indicates that the reaction is spontaneous. Roy et al. [130] realized a ΔG of 90.85 and 92.66 kJ/mol in consonance with the current thermodynamic study at n = 1, where the average is 97.77 kJ/mol. In green chemistry, spontaneous or low-energy reactions are desired to reduce energy input and environmental impact. So, higher ΔG goes against this goal. At 65 ℃, the ΔG for order 3 is almost close to the findings of Zeng et al. [129] who obtained 137.43 kJ/mol. The K^* reflects the extent to which a reaction proceeds toward product formation at equilibrium, and it is directly influenced by the thermodynamic parameters–ΔH, ΔS and ΔG. For the first-order reaction, despite the positive ΔG values across all temperatures which suggest non-spontaneity, the slight increase in K^* from 2.33×10^{-16} at 50 ℃ to 5.54×10^{-16} at 65 ℃ implies that the reaction becomes marginally more favorable at higher

temperatures. This trend is consistent with the behavior of endothermic reactions, where increased temperature shifts the equilibrium toward the products. Nevertheless, the K^* in second-order are lower than those of the first-order case, which suggest a lower extent of reaction. Correspondingly, K^* decreases with temperature in third-order, aligning with expectations for an exothermic reaction, where increasing temperature drives the equilibrium backward. Chemical reactions occasionally cause most of the irreversibility of the process [140]. Thus, based on K^*, first-order reaction model provides the best thermodynamic and statistical consistency. It is important if similar studies is extended to producing bioethanol, biobutanol and biohydrogen [141], including at supercritical conditions [142,143]. In Table 6, E_a, ΔH, ΔS and ΔG reported previously in the literature for other catalyst types are documented. For industrial implementation, we recommend pilot-scale trials addressing catalyst stability/recyclability, metal leaching, and mass-transfer limitations to ensure long-term performance and environmental compliance.

Table 6. Biodiesel thermodynamic parameters from the literature.

Catalyst Type	Reaction Condition	Thermodynamic Parameter	Ref.
H.ZA & DH.ZA	H.ZA optimum: 80 ℃, MeOH/oil 14:1, 3 wt.% & 120 min; DH.ZA optimum: 60 ℃, MeOH/oil 12:1, 3 wt.% & 60 min	E_a : H.ZA = 35.9 & DH.ZA = 32.714 kJ·mol^{-1}. ΔH*: H.ZA = 33.23 & DH.ZA = 30.03 kJ·mol^{-1}. ΔS*: H.ZA = −195.59 & DH.ZA = −195.91 J K^{-1} mol^{-1}	[144]
SO_4^{2-}/ZnO-β-zeolite (β-zeolite supported sulfated metal-oxide)	3 wt. % catalyst loading, 200 ℃ reaction temp., 15:1 molar ratio of methanol to oil & 8 h reaction time	E_a = 38.58 kJ mol^{-1}	[145]
CaO/Ag nanocatalyst	NR	E_a = 43.73 kJ mol^{-1}	[146]
KOH/$ZnAl_2O_4$ nanocatalyst	4 wt.% catalyst, 90 min & 9:1 M:O-MR	E_a = 42.57 kJ mol^{-1}	[147]
Biochar-based catalyst from rubber-seed shells	65 ℃	E_a = 36.6 kJ mol^{-1}, ΔH = 33.9 kJ mol^{-1} & ΔS = −0.180 kJ mol^{-1}	[148]
Alumina	Methanol to oil molar ratio = 6:1 & 323, 328 and 333 K	E_a = 46.19 kJ mol^{-1}; ΔH = 43.78 kJ mol^{-1}; ΔS = −0.129 kJ mol^{-1} K^{-1} & ΔG = 90.68, 92.13, and 93.58 kJ mol^{-1}	[149]
SO_3H-functionalized eucalyptus tree bark biochar	80 ℃, 5 h reaction time, catalyst loading = 4 wt.% & 8:1 methanol/OA molar ratio	E_a = 81.77 kJ mol^{-1}; ΔH = 78.94 kJ mol^{-1}; ΔS = 135.3 J mol^{-1} K^{-1} & ΔG = 33.03 kJ mol^{-1}	[150]
BNS	3 wt.% catalyst loading, 600 rpm stirring, 10:1 methanol-oleic acid ratio, 180 min reaction time & 50-65℃	E_a = 51.64 kJ mol^{-1}; ΔH = 48.8 kJ mol^{-1}; ΔS = -148.11 J mol^{-1} K^{-1} & ΔG = 96.66 kJ mol^{-1} (at 50 ℃)	This Study

Note: ZA–Zeolite-A; H.ZA–Hydrated Phase ZA; DH.ZA–Dehydrated Phase ZA.

Table 6 does not report the recyclability of the catalyst employed. In this study, the BNS catalyst was only able to sustain one effective reaction cycle. Presumably, there is limited structural or active site stability under the reaction conditions. It suggests that further modification or reinforcement of the catalyst is required to improve its reusability and practical applicability. A study by Li et al. [151] shows that microwave heating achieved a 'k' value about 10-20 × higher than conventional heating and significantly reduced the E_a. Nga et al. [152] explicitly report their FexOy/CaO catalyst sieved to 0.125 mm, which is similar to BNS size used herein. However, the particle activity cannot be analyzed under different conditions looked at in both studies. Also, no particle size of the heterogeneous catalyst reported in Pham et al. [153], was mentioned using microwave. The 0.125 mm (≈125,000 nm) BNS catalyst is about 500,000% larger in particle size compared to the 20-30 nm electrocatalyst reported in Yelegen et al. [154]. Compared to previous biomass-derived catalysts, this study employs the BNS catalyst under milder reaction temperatures (50-65 ℃) and moderate methanol-to-oil ratios. The study demonstrated an effective performance without requiring the high temperatures (≥80 ℃) or prolonged reaction times often reported in earlier works. Many previous catalysts showed lower E_a values. In contrast, the thermodynamic profile of BNS (ΔH = 48.8 kJ mol^{-1} and ΔS = -148.11 J mol^{-1} K^{-1}) indicates a more ordered transition state that aligns with a controlled and energy-efficient reaction environment. Unlike studies that rely on highly functionalized or metal-supported catalysts, this work uses a simpler, low-cost biomass precursor, to support sustainability and ease of preparation. Thus, BNS catalyst balances practical reaction conditions with competitive thermodynamic behavior. It distinguishes it from previous systems in Table 6 that either require harsher operating conditions or more complex modification steps.

4. Conclusion

This study successfully demonstrated the application of green chemistry principles in the thermodynamic assessment of biodiesel synthesis using a catalyst derived from BNS, an abundant agricultural waste. The characterization of the BNS catalyst confirmed the presence of key functional groups and porous morphology, which contributed to its high catalytic activity. Kinetic and thermodynamic evaluations revealed that the esterification reaction best follows a first-order

mechanism, with favorable activation energy, enthalpy, and Gibbs free energy values. Among the three reaction orders studied, the first-order model showed the highest linearity (R^2 = 0.9980), the lowest activation energy (51.64 kJ/mol), and the most thermodynamically efficient profile with the least enthalpy (48.80 kJ/mol), less negative entropy (-148.11 J/mol·K), and lowest Gibbs free energy across all temperatures tested. These values indicate an energy-efficient, entropy-driven, and feasible reaction pathway. The use of BNS aligns with several green chemistry principles, including waste valorization, energy efficiency, catalysis, and the use of renewable resources. The findings had validated the thermodynamic viability of BNS in biodiesel production and also emphasized its potential to reduce environmental impact and enhance sustainability. BET surface area analysis, Barrett-Joyner-Halenda (BJH) pore size distribution, TGA, XRD, and Inductively Coupled Plasma Optical Emission Spectroscopy/Mass Spectrometry (ICP-OES/ICP-MS) should be conducted for enhanced description of the catalyst performance. In the next study, atom economy, mass intensity/productivity, reaction mass efficiency and E-factor are green metrics that should be computed.

Conflicts of Interests

The authors declare that they have no conflicts of interests.

Generative AI Statement

The authors declare that no Gen AI was used in the creation of this manuscript.

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