

Rapid Microwave-Assisted Transesterification of Palm Oil Using Sulfonated Graphene Catalyst Prepared Via SLS-Stabilized Turbulence-Assisted Shear Exfoliation

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Abstract

Graphene-based sulfonated catalysts (graphene- SO_3H) were synthesized using the turbulence-assisted shear exfoliation (TASE) method with sodium lauryl sulfate (SLS) as a stabilizing agent. The catalyst was applied for microwave-assisted transesterification of palm oil to produce biodiesel. Characterization of the catalyst via FTIR, XRD, and SEM-EDX confirmed the successful sulfonation and the formation of a porous graphene structure. Among the tested condition, the highest biodiesel yield of 99.07% was achieved using 2 mL SLS and 0.54 g graphene-SLS within 3 min at 65°C. The resulting product met the density and pH requirements, with values of 0.89 g/mL and approximately 7.4, respectively. However, its viscosity of 8.38 cS and acid number of 1.28 mg KOH/g exceeded the limits set by EN 14214, indicating that the product should still be regarded as crude biodiesel rather than standard-compliant fuel-grade biodiesel. Therefore, further post-reaction purification is required to reduce residual impurities affecting viscosity and acid value. This study demonstrates that the combination of graphene- SO_3H catalyst and microwave irradiation is promising as a rapid transesterification route for palm-oil-based biodiesel production, while product purification remains essential for fuel-grade application.

Keywords: Biodiesel, Microwave Induction, Graphene- SO_3H , Palm Oil, Sodium Lauryl Sulfate.

Abstrak

Katalis tersulfonasi berbasis grafena (grafena- SO_3H) disintesis menggunakan metode eksfoliasi geser berbantuan turbulensi (TASE) dengan natrium lauril sulfat (SLS) sebagai agen penstabil. Katalis tersebut diaplikasikan untuk transesterifikasi minyak sawit berbantuan gelombang mikro untuk menghasilkan biodiesel. Karakterisasi katalis melalui FTIR, XRD, dan SEM-EDX mengkonfirmasi keberhasilan sulfonasi dan pembentukan struktur grafena berpori. Di antara kondisi yang diuji, hasil biodiesel tertinggi sebesar 99,07% dicapai menggunakan 2 mL SLS dan 0,54 g grafena-SLS dalam waktu 3 menit pada suhu 65°C. Produk yang dihasilkan memenuhi persyaratan densitas

dan pH, dengan nilai masing-masing 0,89 g/mL dan sekitar 7,4. Namun, viskositasnya (8,38 cS) dan angka asamnya (1,28 mg KOH/g) melebihi batas yang ditetapkan oleh EN 14214, menunjukkan bahwa produk tersebut masih harus dianggap sebagai biodiesel mentah dan bukan biodiesel kelas bahan bakar yang sesuai standar. Oleh karena itu, pemurnian lebih lanjut setelah reaksi diperlukan untuk mengurangi pengotor residu yang memengaruhi viskositas dan nilai asam. Studi ini menunjukkan bahwa kombinasi katalis graphene- SO_3H dan iradiasi gelombang mikro menjanjikan sebagai jalur transesterifikasi cepat untuk produksi biodiesel berbasis minyak sawit, sementara pemurnian produk tetap penting untuk aplikasi kelas bahan bakar.

Keywords: Biodiesel, Induksi Gelombang Mikro, Grafena- SO_3H , Minyak Sawit, Natrium Lauril Sulfat.

1. Introduction

Global energy consumption continues to rise in line with the growth of industry and transportation [1], leading to a serious dependency on fossil fuels that results in issues such as resource depletion, price fluctuations, and increased greenhouse gas emissions, which accelerate climate change [2]. Therefore, the development of renewable energy sources has become a strategic priority in the transition to a sustainable energy system [3]. One of the most promising renewable energy sources is biodiesel, as it is biodegradable, non-toxic, and capable of reducing carbon dioxide emissions compared to fossil fuels [4].

Biodiesel is commonly produced through the transesterification reaction of vegetable oils or animal fats with alcohol using base or acid catalysts [5]. The use of acid catalysts requires higher temperatures and pressures, as well as longer reaction times compared to base catalysts [5]. Homogeneous catalysts such as NaOH or KOH are effective but pose challenges in product separation and generate liquid waste that is environmentally unfriendly [6]. In contrast, heterogeneous catalysts are preferred because they are easy to separate, reusable, and more environmentally friendly [6]. However, various metal oxide- and carbon-based heterogeneous catalysts still face limitations, such as long reaction times and suboptimal conversion [7].

In the past decade, carbon-based nanomaterials, particularly graphene and its derivatives, have gained significant attention as potential catalysts for biodiesel production due to their high specific surface area, thermal stability, and numerous active sites for Brønsted acid reactions [8]. Sulfonated graphene (graphene- SO_3H) has been reported to accelerate the transesterification reaction with high efficiency [9], [10]. However, most synthesis methods involve graphene oxide, which is relatively expensive, requires hazardous chemicals (such as concentrated H_2SO_4 and KMnO_4), and demands considerable time and energy [11].

Additionally, the efficiency of biodiesel production can also be enhanced through the use of microwave heating. This technology is capable of distributing heat uniformly at the molecular level, thereby accelerating the reaction rate and reducing energy requirements

compared to conventional heating methods [12], [13]. However, most previous studies have focused on conventional metal oxide or sulfonated carbon catalysts with reaction times ranging from 30 to 90 minutes [14], [15].

Based on the research gap, this study proposes an alternative approach involving the synthesis of non-oxide graphene-SO₃H catalysts using sodium lauryl sulfate (SLS) as a stabilizing agent via the turbulence-assisted shear exfoliation (TASE) method [16], [17], which is then applied in the transesterification reaction of palm oil under microwave induction heating. The primary focus of this study is to investigate the effect of varying SLS concentrations on the morphology, chemical structure, and catalytic activity of graphene-SO₃H, as well as to assess the process efficiency through yield parameters and the physical properties of the resulting biodiesel. Thus, this study is expected to contribute to the development of a faster, more efficient, and environmentally friendly biodiesel production process utilizing non-oxide graphene-based catalysts.

2. Method

2.1 Preparation of Graphene-SLS

Graphene synthesis was carried out using the TASE method [17], employing distilled water containing SLS surfactant with variations of 1, 2, and 5 mL as the working fluid. After the TASE process, a black liquid [16] was obtained and allowed to stand for 24 hours, after which the liquid and its precipitate were separated. The black liquid was then dried in an oven at 80°C until graphene-SLS powder was formed.

2.2 Preparation of Sulfonated Graphene Catalyst (Graphene-SO₃H)

Graphene-SLS was then calcined using a furnace at 350°C for 5 minutes. The sulfonated graphene synthesis was carried out using 0.27, 0.41, and 0.54 g of the calcined graphene-SLS, which was sonicated in water for 2 hours. Sodium nitrite (0.947 g) and sulfanilic acid (0.789 g) were added to the solution at 25°C and the mixture was stirred for 24 hours. After the reaction, the solution was filtered through a Millipore membrane and washed with 100 mL of 1 N HCl and 100 mL of acetone. The resulting solution was then dried at 25°C to obtain graphene-SO₃H powder [18].

2.3 Biodiesel Synthesis Using Graphene-SO₃H Catalyst

Graphene-SO₃H (0.025 g) was used as the heterogeneous catalyst for palm oil transesterification with methanol at a methanol-to-oil molar ratio of 20:1. The reaction was carried out at 65°C for 3 min under microwave irradiation. Transesterification was performed using a Sharp microwave oven, model R-220MA-WH, with an input power of 750 W and a rated microwave output power of 450 W. The appliance was operated using a 220 V, 50 Hz power supply, while the microwave irradiation frequency was 2.45 GHz. The system was used as a multimode cavity microwave applicator. The reaction mixture was placed in a 50 mL borosilicate glass beaker and heated using the medium power setting. After the reaction, the mixture was transferred into a separating funnel and allowed to separate into two layers: the

upper biodiesel-rich phase and the lower glycerol-rich phase. The complete workflow of catalyst preparation and biodiesel synthesis is presented in Figure 1.

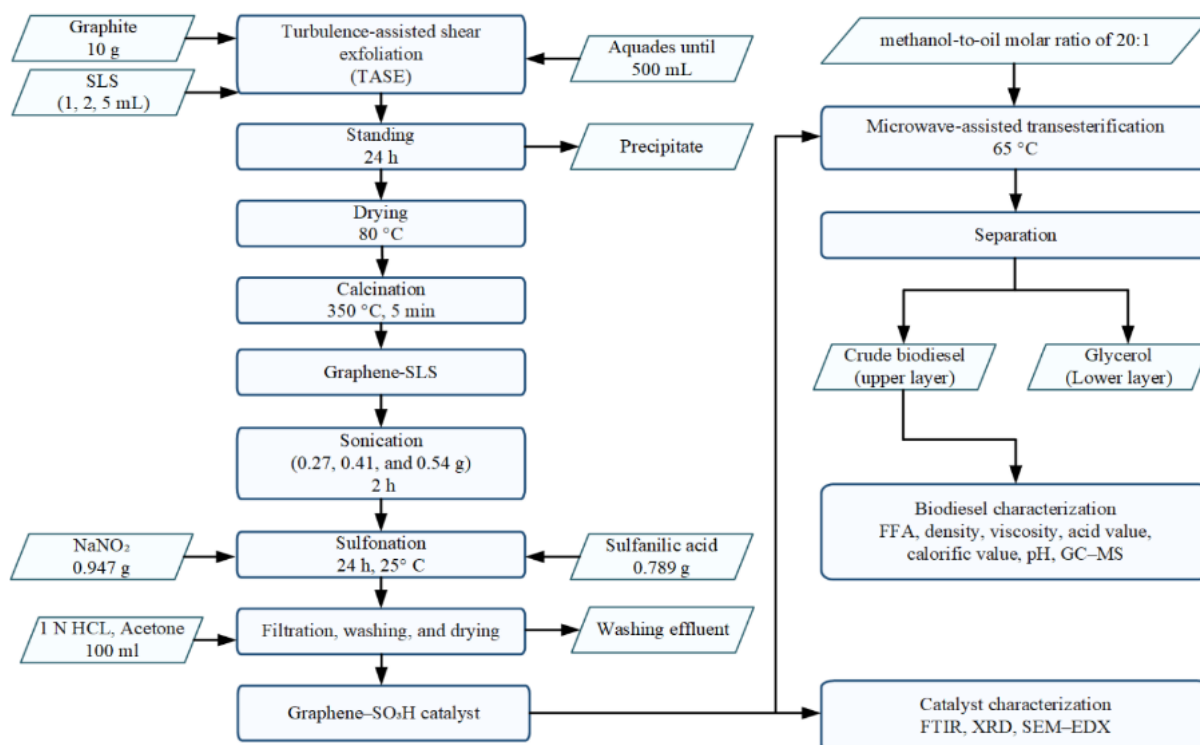


Figure 1. Flow Diagram of The Experimental Procedure

2.4 Characterization

The analysis of free fatty acid content in the raw palm oil feedstock was adopted from the study [19]. The chemical structure and functional groups of the graphene-SO₃H catalyst were characterized using Fourier Transform Infrared Spectroscopy (FTIR) with a PerkinElmer Spectrum IR Version 10.6.1 instrument. The crystallinity of the graphene-SO₃H catalyst was analyzed via X-ray diffraction using an XRD XPERT-PRO Panalytical instrument with a copper (Cu) anode, operating at 30 mA and 40 kV. The average crystallite size (nm) was measured using the Scherrer Equation (1) [20].

$$D = \frac{k \lambda}{\beta \cos \theta} \quad (1)$$

where k is the shape factor (0.9), λ (1.54Å) is the X-ray wavelength, β is the full width at half maximum peak, and θ is the diffraction or Bragg angle at the (200) peak. Morphological analysis of the graphene-SO₃H catalyst was performed using Scanning Electron Microscopy (SEM) with a SU 3500 SEM instrument. The compounds present in the biodiesel were analyzed using Gas Chromatography-Mass Spectrometry (GC-MS) with a Shimadzu GC-MS-TQ8050 NX instrument. The physical and chemical properties of the biodiesel produced were analyzed according to the SNI 7182:2015 and EN 14214 standards, which include acid value, density, viscosity, pH, and calorific value.

3. Result and Discussion

3.1 Free Fatty Acid (FFA) Content Analysis of Palm Oil

The Fortune brand commercial palm oil used as feedstock had an FFA content of 0.02 mg KOH/g oil, a density of 0.90 g/mL, and a viscosity of 8.50 cSt. FFA content is an important parameter for assessing feedstock suitability for transesterification because high FFA levels may react with base catalysts, promote soap and emulsion formation, and reduce conversion efficiency [21]. Since the FFA content was below 1%, the feedstock was suitable for direct transesterification without pre-treatment [19]. The low FFA level also helps minimize soap formation and glycerol separation problems during biodiesel production [19], [22].

3.2 Biodiesel Yield and Physical Properties Analysis

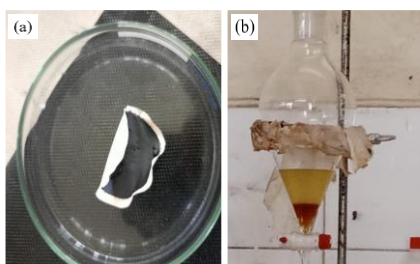


Figure 2. (a) Graphene-SO₃H; (b) Biodiesel Synthesis

Figure 2(a) shows the graphene-SO₃H catalyst, while Figure 2(b) shows the biodiesel product obtained after transesterification. Graphene-SO₃H was applied to palm oil-methanol feedstock under microwave irradiation at 65°C for 3 min. Microwave irradiation accelerates transesterification through rapid volumetric heating of polar molecules and ionic species in the reaction medium, allowing faster and more uniform heat transfer than conventional heating [23], [24].

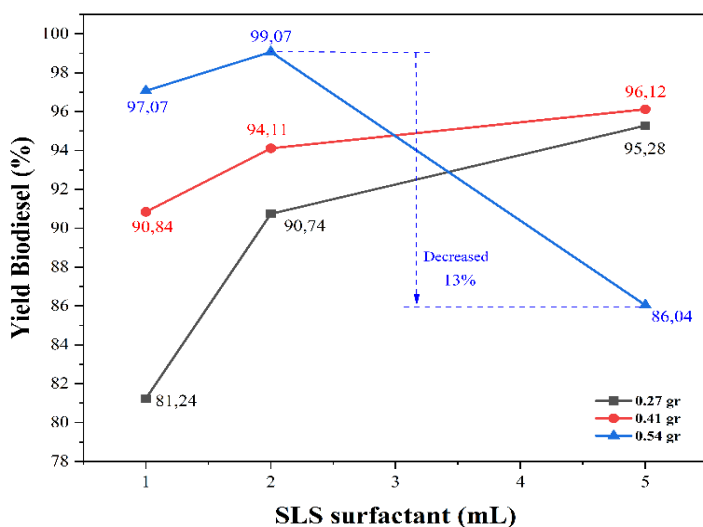


Figure 3. Biodiesel Yield with Graphene-SO₃H Catalyst Prepared Using Different Graphene-SLS Loadings after Calcination

Figure 3 shows the biodiesel yield obtained using graphene–SO₃H catalysts prepared with different SLS concentrations and graphene-SLS loadings after calcination. The biodiesel yield generally increased with increasing graphene-SLS loading from 0.27 to 0.54 g. This increase can be attributed to the greater number of accessible catalytic sites available for the transesterification reaction. SLS also improved graphene dispersion by stabilizing exfoliated graphene sheets through electrostatic repulsion, thereby reducing agglomeration and improving catalytic accessibility [25], [26]. The highest biodiesel yield of 99.07% was obtained using 2 mL SLS and 0.54 g graphene-SLS, whereas the lowest yield of 81.24% was obtained using 1 mL SLS and 0.27 g graphene-SLS.

At the highest graphene-SLS loading of 0.54 g, increasing the SLS volume above 2 mL reduced the biodiesel yield by approximately 13 percentage points. This reduction may be attributed to excessive SLS adsorption or micelle-like coverage on the graphene–SO₃H surface. Such coverage can partially block –SO₃H active sites, limit triglyceride access to the catalyst surface, and increase mass-transfer resistance in the heterogeneous transesterification system [27], [28]. Conversely, insufficient SLS may fail to provide enough electrostatic repulsion to prevent graphene agglomeration. Therefore, both insufficient and excessive SLS addition can reduce the effective catalytic surface area and lower biodiesel yield [25], [26].

The physicochemical properties of the produced biodiesel are summarized in Table 1. The density values ranged from 0.89 to 0.90 g/mL and were within the acceptable ranges specified by SNI 7182:2015 and EN 14214. The calorific value ranged from 37.59 to 40.23 kJ/g, indicating that it was not the limiting parameter in this study. However, the viscosity values of 8.32–8.80 cSt and acid values of 1.06–1.51 mg KOH/g exceeded the limits specified by SNI 7182:2015, ASTM D6751, and EN 14214. Therefore, the product should still be classified as crude biodiesel rather than standard-compliant fuel-grade biodiesel. The elevated viscosity and acid value may be associated with residual glycerides, glycerol, free fatty acids, catalyst residues, surfactant residues, water, alcohol, or incomplete phase separation [29], [30], [31]. Further post-reaction purification is required to improve the final biodiesel quality. Recommended purification steps include catalyst removal by filtration or centrifugation, methanol recovery, glycerol separation, washing or adsorption treatment, final filtration, and drying [30], [32], [33].

Table 1. Physicochemical Properties of Crude Biodiesel Produced Under Different SLS and Graphene-SLS Variation

Sample		Density (g/mL)	Viscosity (cSt)	Acid Value (mg KOH/g)	Calorific Value (kJ/g)	pH
SLS (mL)	Graphene-SLS (g)					
1	0.27	0.89	8.60	1.28	38.22	7.36
	0.41	0.90	8.80	1.06	37.59	7.23
	0.54	0.89	8.74	1.51	38.35	6.95
2	0.27	0.89	8.45	1.34	39.36	7.13
	0.41	0.90	8.59	1.28	39.87	7.10
	0.54	0.89	8.38	1.28	40.00	7.39
5	0.27	0.90	8.32	1.34	39.67	7.19
	0.41	0.89	8.57	1.34	40.03	7.14
	0.54	0.90	8.37	1.28	40.23	6.95

Sample		Density (g/mL)	Viscosity (cSt)	Acid Value (mg KOH/g)	Calorific Value (kJ/g)	pH
SLS (mL)	Graphene-SLS (g)					
Standards						
SNI 7182:2015		0.85-0.89	2.3-6	0.5 (max)	Not specified	~ 7
ASTM D6751		Not specified	1.9-6	0.5 (max)	Not specified	~ 7
EN 14214		0.86-0.90	3.5-5	0.5 (max)	≥ 35	~ 7

3.3 Gas Chromatography-Mass Spectrometry (GC-MS) Analysis of Biodiesel

Figure 4 shows the GC-MS chromatogram of the biodiesel sample obtained under the highest-yield condition, namely 2 mL SLS and 0.54 g graphene-SLS. The identified compounds are listed in Table 2. The GC-MS results confirmed the formation of fatty acid methyl ester (FAME) components, including hexadecanoic acid methyl ester, octadecanoic acid methyl ester, and 9-octadecenoic acid methyl ester. These compounds correspond to methyl palmitate, methyl stearate, and methyl oleate, which are commonly found in palm-oil-derived biodiesel [34], [35], [36].

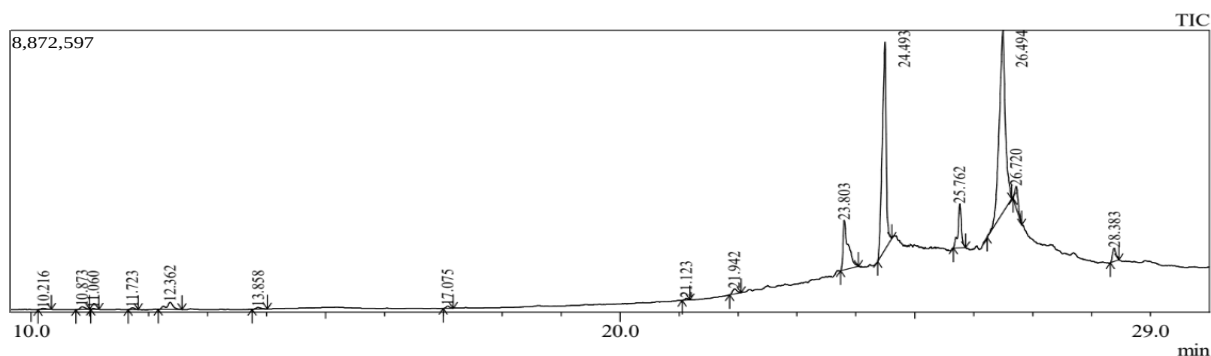


Figure 4. GC-MS Chromatogram of Biodiesel Produced Using 2 mL SLS and 0.54 g Graphene-SLS

However, several non-FAME or free-fatty-acid-type compounds were also detected, including tetradecanoic acid, cis-vaccenic acid, and octadecanoic acid. Cis-vaccenic acid showed the largest peak area of 43.03%, followed by octadecanoic acid methyl ester at 32.30%. The presence of these non-FAME compounds indicates that the product was not fully purified and may still contain residual acidic or lipid-derived impurities. This finding is consistent with the elevated acid value reported in Table 1 and with previous reports that residual free fatty acids and other impurities can affect biodiesel quality [29], [31]. Therefore, the GC-MS results support the formation of biodiesel components, but also confirm that further post-reaction purification is required before the product can be classified as standard-compliant fuel-grade biodiesel.

Table 2. Identified Compounds in Biodiesel Produced Using 2 mL SLS and 0.54 g Graphene-SLS

Peak	R.Time	Area %	Height %	Component
1	10,27	0,21	0,22	2,4-Decadienal
2	10,87	0,48	0,48	2,4-Decadienal, (E,Z)-
3	11,06	0,10	0,15	2-Decenal, (E)-
4	11,72	0,26	0,33	2,4-Decadienal, (E,Z)-

Peak	R.Time	Area %	Height %	Component
5	12,36	1,84	1,29	Copaene
6	13,86	0,35	0,31	Caryophyllene
7	17,08	0,30	0,38	Undecanoic acid, 10-methyl-, methyl ester
8	21,12	0,20	0,32	Tridecanoic acid, 12-methyl-, methyl ester
9	21,94	0,90	0,94	Tetradecanoic acid
10	23,80	9,88	9,13	Hexadecanoic acid, methyl ester
11	24,49	32,30	38,33	Octadecanoic acid, methyl ester
12	25,76	5,75	8,06	9-Octadecenoic acid (Z)-, methyl ester
13	26,49	43,03	33,65	Cis-Vaccenic acid
14	26,72	2,62	3,95	Octadecanoic acid
15	28,38	1,77	2,46	Myristic acid glycidyl ester

3.4 X-Ray Diffraction (XRD) Analysis

Figure 5 shows the XRD pattern of the graphene–SO₃H catalyst prepared using 2 mL SLS and 0.54 g graphene-SLS. The main diffraction peak appeared at $2\theta = 26.55^\circ$, with a d-spacing of approximately 3.35 Å. This peak corresponds to the characteristic (002) plane of graphitic carbon, indicating that the catalyst retained a layered graphene/graphitic structure after sulfonation [37], [38]. A broad and weak diffraction feature was also observed around $2\theta = 24.80^\circ$, suggesting lower structural ordering in the sulfonated graphene material. Another weak peak appeared around $2\theta = 44.54^\circ$, which can be associated with turbostratic graphitic carbon. The broadening and low intensity of these peaks indicate that the graphene–SO₃H catalyst contains small crystallite domains and partially disordered carbon structures [14], [38]. The crystallite size calculated using the Scherrer equation ranged from 17.39 to 40.67 nm, with an average crystallite diameter of 26.57 nm. These values indicate that the graphene–SO₃H catalyst contains nanoscale crystalline domains while retaining a largely disordered carbon structure. This structure is beneficial for heterogeneous catalysis because disordered carbon domains and layered graphene structures can provide accessible surface sites for –SO₃H functional groups.

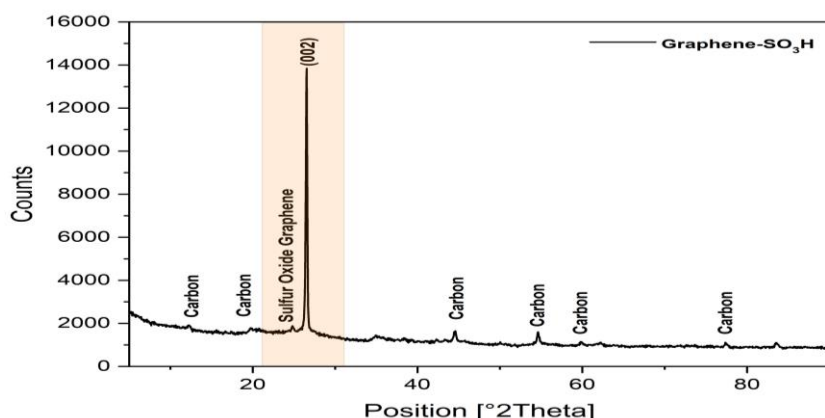


Figure 5. XRD Pattern of Graphene–SO₃H Catalyst Prepared Using 2 mL SLS and 0.54 g Graphene-SLS

3.5 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

Figure 6 shows the FTIR spectra of graphene–SO₃H catalysts prepared using different SLS variations. The analyzed samples represent the highest biodiesel yield condition, namely

2 mL SLS and 0.54 g graphene-SLS, the lowest yield condition, namely 1 mL SLS and 0.27 g graphene-SLS, and the decreased-yield condition, namely 5 mL SLS and 0.54 g graphene-SLS. A broad absorption band around $\sim 3400\text{ cm}^{-1}$ indicates the presence of hydroxyl (O–H) groups, which may be associated with adsorbed water or oxygen-containing groups on the graphene surface [17], [39]. The peaks around ~ 2920 and 2850 cm^{-1} correspond to C–H stretching vibrations, while the peak at $\sim 1614\text{ cm}^{-1}$ is attributed to aromatic C=C stretching in the graphene framework [17], [40].

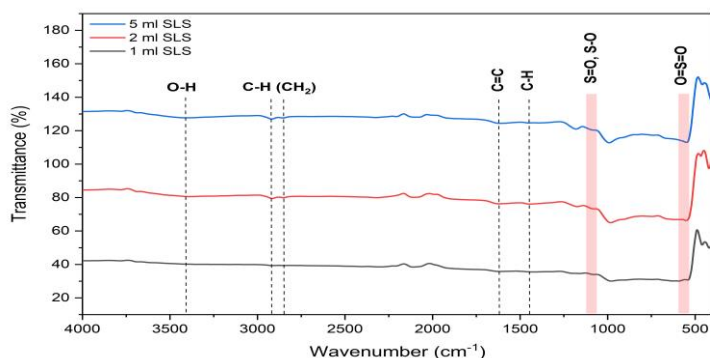


Figure 6. FTIR Spectra of Graphene–SO₃H Catalysts Prepared Using Different SLS Variations

The absorption band around $\sim 1405\text{ cm}^{-1}$ can be assigned to C–H deformation in the aromatic structure. More importantly, the peaks observed around ~ 1108 and 1032 cm^{-1} indicate S=O and S–O stretching vibrations associated with sulfonate groups [41]. In addition, the peaks around ~ 601 and 545 cm^{-1} are related to O=S=O deformation vibrations, which further support the presence of sulfonate functionality [42], [43]. Therefore, the FTIR results confirm the successful introduction of –SO₃H groups onto the graphene structure. These sulfonate groups act as Bronsted acid sites and are essential for the catalytic activity of graphene–SO₃H in the transesterification reaction [44].

3.6 Scanning Electron Microscopy (SEM) and EDX Analysis

Figure 7 presents the SEM images and EDX spectra of graphene–SO₃H catalysts prepared using different SLS and graphene-SLS variations. Figure 7(a) shows the SEM image of the catalyst prepared using 1 mL SLS and 0.27 g graphene-SLS. The catalyst exhibited a rough, dense, and inhomogeneous morphology with visible aggregation and uneven pores. This condition indicates that the amount of SLS was insufficient to disperse the graphene sheets uniformly. As shown in Figure 7(b), the EDX spectrum confirmed the presence of C, O, and S elements, indicating that sulfonation occurred on the graphene surface. However, the poor morphology in Figure 7(a) resulted in fewer accessible active surfaces, which is consistent with the lowest biodiesel yield of 81.24% [45], [46].

In contrast, Figure 7(c) show that the catalyst prepared using 2 mL SLS and 0.54 g graphene-SLS exhibited a more open and porous structure. The graphene flakes were more uniformly distributed, although slight aggregation was still observed. This morphology can improve reactant diffusion and increase access to catalytic active sites, thereby supporting the

highest biodiesel yield of 99.07% [46]. The EDX spectrum in Figure 7(d) also confirmed the presence of C, O, and S elements, indicating successful sulfonation of graphene and the attachment of $-\text{SO}_3\text{H}$ groups on the catalyst surface [14], [44]. For the catalyst prepared using 5 mL SLS and 0.54 g graphene-SLS (Figure 7(e)), the SEM image showed thick stacked layers, a clumped surface, and large irregular pores. This morphology suggests that excessive SLS may promote re-agglomeration or micelle-like coverage on the graphene- SO_3H surface. Such coverage can reduce contact between the reactants and active sites, thereby lowering catalytic efficiency. This observation supports the decrease in biodiesel yield by approximately 13 percentage points compared with the 2 mL SLS condition [25]. The EDX result (Figure 7(f)) still confirmed the presence of C, O, and S, while the higher sulfur signal may be associated with sulfonate groups and possible residual sulfate species from excessive SLS.

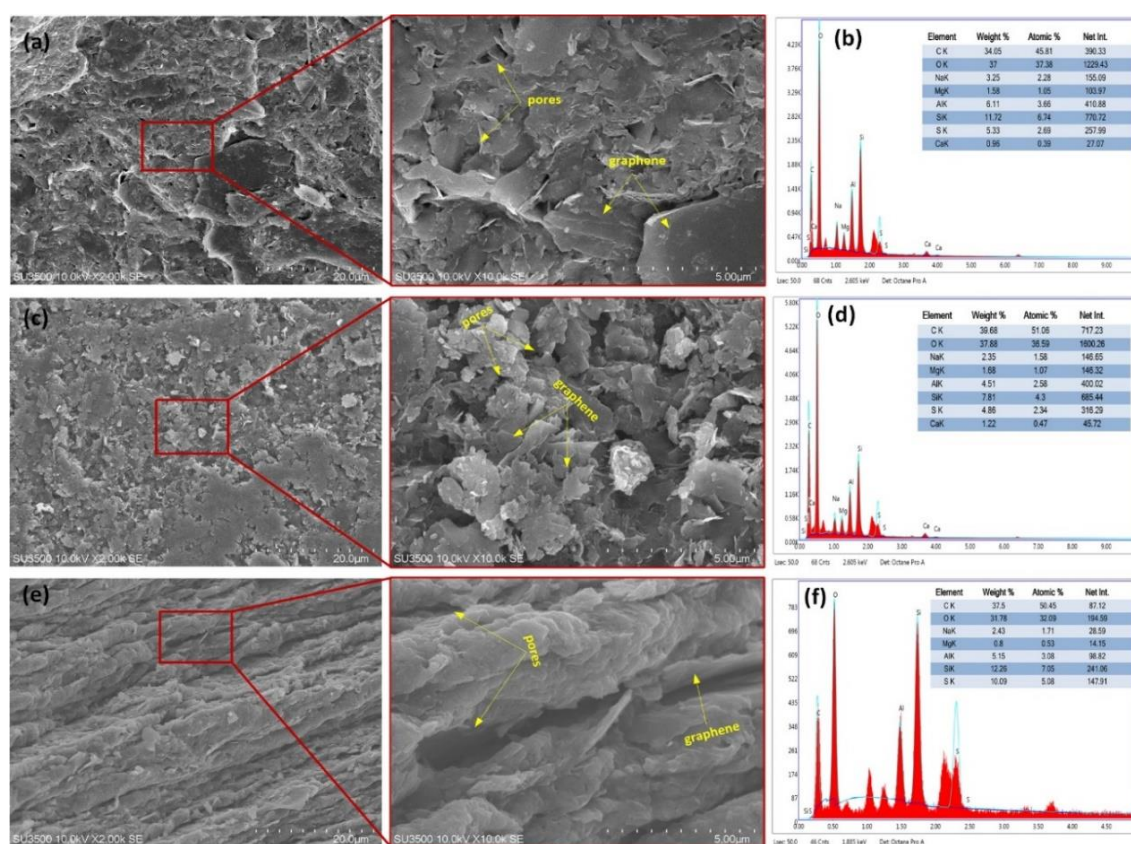


Figure 7. (a) SEM Image and (b) EDX Spectrum of The Graphene- SO_3H Catalyst Prepared Using 1 mL SLS and 0.27 g Graphene-SLS; (c) SEM Image and (d) EDX Spectrum of The Graphene- SO_3H Catalyst Prepared Using 2 mL SLS and 0.54 g Graphene-SLS; (e) SEM Image and (f) EDX Spectrum of The Graphene- SO_3H Catalyst Prepared Using 5 mL SLS and 0.54 g Graphene-SLS

4. Conclusion

The rapid microwave-assisted transesterification of palm oil using a sulfonated graphene catalyst was successfully carried out within 3 min. The highest biodiesel yield of 99.07% was obtained using a catalyst prepared with 2 mL SLS and 0.54 g graphene-SLS. This result indicates that an appropriate amount of SLS plays an important role in improving graphene

dispersion and maintaining accessible catalytic active sites. However, excessive SLS addition reduced the biodiesel yield by approximately 13 percentage points, most likely due to graphene re-agglomeration or micelle-like coverage that limited reactant access to the catalyst surface. The physicochemical analysis showed that the produced biodiesel met the density and pH requirements, but its viscosity and acid value were still above the standard limits. Therefore, the product should be classified as crude biodiesel and requires further post-reaction purification before being considered fuel-grade biodiesel. GC-MS analysis confirmed the formation of FAME components, including methyl palmitate, methyl stearate, and methyl oleate, while the presence of non-FAME or free-fatty-acid-type compounds further supported the need for purification. Catalyst characterization confirmed the successful formation of graphene-SO₃H. FTIR analysis showed characteristic absorption bands related to sulfonate groups, particularly S=O, S-O, and O=S=O vibrations. XRD analysis revealed a graphitic diffraction peak at approximately 26.55°, indicating that the catalyst retained a layered carbon structure after sulfonation. SEM analysis showed that the catalyst prepared with 2 mL SLS and 0.54 g graphene-SLS had a more porous and homogeneous morphology, while EDX analysis confirmed the presence of C, O, and S elements. Overall, graphene-SO₃H prepared through SLS-assisted exfoliation showed strong potential as a heterogeneous acid catalyst for rapid biodiesel production, although further purification and process optimization were required to obtain standard-compliant fuel-grade biodiesel.

Appreciation

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