

Research Article

Utilising Polyester and Steel Slag-Derived Metal/Carbon Composites as Catalysts in Biodiesel Production

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Synthetic textiles such as polyesters are essential for daily life. However, large-scale production generates large amounts of waste. This study introduces a new approach for valorising polyester textile waste (PTW) by transforming it into a catalyst for biodiesel production via pyrolysis. Specifically, a metal/carbon composite (PTW + steel slag [SS] composite—PSC) with enhanced catalytic properties was prepared by pyrolysing PTW with SS. The alkaline metals in SS facilitate the carbonisation of PTW via decarboxylation, resulting in a PSC rich in carbon, iron, and alkaline compounds. This composite featured mesopores that were larger than the micropores (MPs) typically found in PTW char. The use of porous material (silica) in thermally induced transesterification has been proven to be an efficient method for biodiesel production, achieving a yield of 97.20 wt.% in 1 min (faster than the 93.82 wt.% yields in 60 min observed from conventional alkali-catalysed transesterification). However, the high reaction temperature ($\geq 360^{\circ}\text{C}$) poses economic/technical challenges. To overcome this, PSC has been employed as a catalyst in thermally induced transesterification, leveraging its mesoporous structure and high alkaline content, particularly calcium oxide. The PSC achieved a biodiesel yield of 98.10 wt.% at a markedly lower reaction temperature of 120°C within 1 min. This performance was not attainable using silica or PTW char under similar conversion conditions. These findings highlight the potential of PSC produced through the pyrolysis of PTW and SS as effective catalysts for biodiesel production. This process is a promising strategy for converting waste into valuable resources and mitigating the environmental impacts associated with polyester waste.

Keywords: biodiesel; metal/carbon composite; steel slag; textile waste; transesterification; waste valorisation

1. Introduction

Synthetic textiles are integral to our daily lives and serve various purposes, from clothing to household items and industrial products [1]. Over the last two decades, global textile production has nearly doubled, reaching 109 million tons in 2020 [2]. Among the various types of textiles, polyester (a synthetic fibre made of polyethylene terephthalate) stands out because of its desirable properties, including high tensile strength and resistance to biological and chemical degradation [3]. In 2020, polyesters accounted for 52% of the global fibre production [4]. However, their extensive use and production have led to massive waste generation [5]. Polyester textile waste (PTW) is disposed of in

landfills and incinerated [6]. Owing to its non-biodegradable nature, PTW in landfills contributes to long-term environmental issues, such as the accumulation of microplastics in soil and groundwater [7]. Although the incineration of polyester effectively reduces its volume and recovers energy through heat, it also risks releasing harmful chemicals, including polycyclic aromatic hydrocarbons (HCs), unburned HCs, and volatile organic compounds, owing to challenges in maintaining optimal combustion conditions [8–10]. Therefore, there is a need for alternative thermochemical processes, such as pyrolysis, to mitigate the formation of toxic substances while converting waste into valuable resources. Pyrolysis offers a promising solution for breaking

down PTW into useful products without the extensive release of pollutants, making it an environmentally and economically viable strategy for waste management.

Pyrolysis is a thermochemical process that decomposes carbonaceous substances into liquid-, gas-, and solid-phase pyrogenic products under oxygen-free conditions [11]. During the pyrolysis of PTW, valuable gases, such as syngas (a mixture of H_2 and CO), and liquid chemicals, such as benzoic acid, can be recovered [12]. Thus, extensive research has focused on optimising the recovery of gaseous and liquid products [13]. In addition, pyrolysis generates char, a solid residue with a high carbon content [14]. However, the physical and chemical properties of PTW char are often insufficient for its use as an effective adsorbent or catalyst compared to commercially available materials, requiring further activation processes [15]. To enhance the utility of PTW char, pyrolysis with metal-containing wastes such as steel slag (SS) offers a viable alternative. SS, a by-product of steel production, is generated in large quantities, with 130–200 million tons discarded annually as waste [16]. SS is disposed of in landfills, which poses adverse environmental and health risks owing to the potential leaching of alkaline and heavy metals into the soil [17]. Therefore, the pyrolysis of PTW and SS provides a method for waste valorisation and helps mitigate environmental impacts. During the pyrolysis of PTW and SS, PTW melts, disperses on the surface of the SS, and undergoes carbonisation to form a metal/carbon composite [18]. SS, which contains large amounts of alkaline metals (calcium), can enhance the catalytic properties of the resulting PTW char through a catalytic effect [19]. The composite formed by this process, referred to as a PTW + SS composite (PSC), holds significant promise as a catalyst for various applications.

This study focused on the use of a PSC fabricated by the pyrolysis of PTW with SS as a catalyst for biodiesel production. Biodiesel is synthesised via the transesterification of triglycerides (TGs) with methanol (MeOH), typically using homogeneous acid or base catalysts [20]. Although effective, this method has several drawbacks, including long reaction times and low tolerance to impurities such as free fatty acids (FAs) and water, which contribute to unwanted reactions such as saponification and hydrolysis [21]. Moreover, separating the catalyst from biodiesel poses a significant challenge [22]. To address these issues, researchers have investigated the use of heterogeneous acid/base catalysts for transesterification [23]. Although these efforts resolve the problems associated with homogeneous catalysts, they often require high operational temperatures and pressures, which complicate the process [24]. An alternative approach involves thermally induced transesterification using porous materials such as silica. This method is technically advantageous because it exhibits a high tolerance to impurities and operates at ambient pressure (1 bar) within 1 min [25]. However, to achieve a high biodiesel yield (>95 wt.%) required high temperatures (>360°C), which is economically impractical [26]. In this context, a PSC derived by pyrolysing PTW with SS offers a promising solution. Alkaline metals in SS provide strong basic sites that facilitate transesterification, thereby enhancing biodiesel production by offering active catalytic sites [27, 28]. To

thoroughly understand the catalyst properties and effectiveness, the thermolytic behaviours of PTW and SS were characterised using thermogravimetric analysis (TGA), whereas the morphological properties of the PSC were characterised using Brunauer–Emmett–Teller (BET) analysis. Finally, the PSC was evaluated as a catalyst for thermally induced transesterification, and the resulting biodiesel yields were measured at various experimental temperatures. These yields were then compared with those obtained using silica and PTW char under the same conditions.

2. Materials and Methods

2.1. Sample Preparation and Chemicals. The PTW was collected from a textile manufacturing factory in Seoul, Korea. The PTW was rinsed with deionised water and dried at 50°C for 24 h. The dried PTW was cut into uniform pieces. The SS was obtained from POSCO (Pohang, Korea). To eliminate moisture, SS was dried at 105°C for 24 h. Subsequently, the PTW pieces and SS were blended in a 1:1 mass ratio (PTW + SS). Soybean oil was purchased from a local market in Seoul, Korea. Nitrogen (N_2) with 99.999% purity was acquired from Green Gas (Korea). A standard gas mixture containing H_2 , CO, and CH_4 was obtained from RIGAS (Seoul, Korea). Dichloromethane (DCM; 99.8%) and MeOH (99.9%) were purchased from Daejung Chemicals & Metals (Gyeonggi, Korea). Silica gel (pore size: 6 nm) and a standard mixture containing 37 FA methyl esters (FAMES) were acquired from Sigma–Aldrich (MO, USA). Potassium hydroxide (KOH; 95.0%) was purchased from Samchun Chemicals (Seoul, Korea).

2.2. Sample Characterisation. The composition of the PTW was determined using a Fourier-transform infrared (FT-IR) spectrometer (Cary 630, Agilent, CA, USA), ranging from 650 to 4000 cm^{-1} . The carbon and hydrogen contents in the PTW and SS samples were determined using an elemental analyser (EA3000, EuroVector, Pavia, Italy). The inorganic contents of the PTW and SS were measured using inductively coupled plasma-optical emission spectroscopy (ICP-OES; 5800, Agilent). The thermolytic behaviours of PTW, SS, PTW + SS, and soybean oil were characterised using a TGA apparatus (STA 448 F5, Netzsch, Bayern, Germany). The sample (10 mg of PTW, SS, or soybean oil, and 20 mg of PTW + SS) was loaded into an alumina crucible and thermally degraded from 35 to 900°C at a heating rate of 10°C min^{-1} under a N_2 atmosphere. The total N_2 flow rate was 100 mL min^{-1} , with 80 mL min^{-1} for purge gas and 20 mL min^{-1} for protective gas. Each TGA test was conducted in triplicates to ensure reliability.

2.3. PSC Fabrication and Its Characterisation. To fabricate the PSC, PTW and SS were pyrolysed under an N_2 atmosphere. For comparison, PTW char was also produced by the pyrolysis of PTW under the same conditions. Pyrolysis tests were performed in a batch reactor comprising a cylindrical quartz tube (inner diameter: 22 mm, outer diameter: 25 mm, length: 1000 mm) with two vacuum fittings. PTW (10 g) and PTW + SS (20 g) were placed at the centre of the batch

reactor, which was then covered with a tubular furnace to provide heat. The sample was heated from 35 to 700°C at a heating rate of 10°C min⁻¹ under a N₂ atmosphere, followed by isothermal holding at 700°C for 1 h. During the pyrolysis, the N₂ flow rate was maintained at 800 mL min⁻¹. The non-condensable gaseous effluents (H₂, CO, CH₄, and CO₂) from the pyrolysis of PTW and SS were quantified using micro-gas chromatography (micro-GC). To quantify the gas effluents, a standard gas mixture was used to calibrate the micro-GC. The liquid pyrolysates were analysed using GC/mass spectrometry (GC/MS; 5977B, Agilent). The surface areas, pore diameters, and pore volumes of the PTW char and PSC were determined using a BET instrument (BELSORP-mini II, MicroTrac, Osaka, Japan). The surface morphologies of the PTW char and the PSC were examined using scanning electron microscopy/energy-dispersive X-ray spectroscopy (SEM/EDX; Cube-II Mini-SEM, Emcraft, Kyonggi-do, Korea). The inorganic content of the PTW char and PSC were measured using ICP-OES.

2.4. Alkali-Catalysed and Thermally Induced Transesterification. The alkali-catalysed transesterification of soybean oil was performed in the presence of a KOH catalyst. Prior to transesterification, 3 mL of MeOH and 0.1 g of KOH were mixed for 10 min at 400 rpm to obtain a homogeneous mixture. The homogeneous mixture was added to soybean oil (10 g) and incubated for 60 min (at 63°C). After the reaction was completed, the converted biodiesel was separated from the glycerol using a separatory funnel via gravimetric separation. Due to their polarity, excess MeOH and KOH were included in the glycerol (bottom layer). The separated biodiesel (top layer) was then diluted with DCM for GC analysis. Thermally induced transesterification of soybean oil was conducted using three types of porous materials (silica, PTW char, and PSC). A batch reactor comprising a bulkhead union (SS-400-61, Swagelok, OH, USA) and two stoppers (SS-400-P, Swagelok) were prepared. One end of the batch reactor was sealed with a stopper, and then 200 µL of MeOH, 7.5 mg of soybean oil, and porous material (320 mg of silica, 400 mg of PTW char, or 500 mg of PSC) were added. The other end of the reactor was sealed with a stopper to create a closed system. The reactor was positioned at the centre of a furnace. The inner temperature of the batch reactor was monitored in real-time using a K-type thermocouple. Once the inner temperature reached the desired temperature, the reactor was removed from the furnace and cooled to 24°C using a cooling fan. In contrast to alkali-catalysed transesterification, thermally induced transesterification does not require an additional step to separate biodiesel from the reaction mixture due to the use of heterogeneous materials. Thus, the produced biodiesel was directly diluted with DCM for GC analysis. Subsequent qualitative and quantitative analyses of the biodiesel were performed using GC/MS and a GC/flame ionisation detector. Both GC systems were equipped with a DB-WAX Ultra Inert column (length: 30 m, diameter: 0.25 mm, and film: 0.25 µm). For GC calibration, a standard mixture containing 37 FAME components was used. The conversion yield of biodiesel was calculated using the following equation:

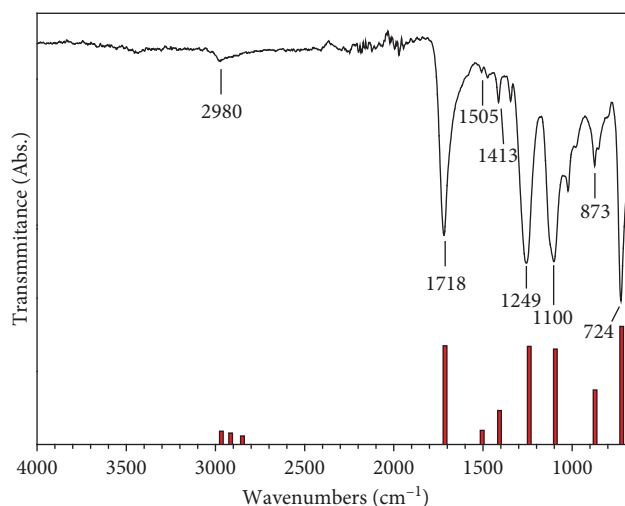


FIGURE 1: FT-IR transmission spectra of PTW (black) and polyester (red bar) as [29–32]. FT-IR, Fourier-transform infrared; PTW, polyester textile waste.

$$\text{Biodiesel yield (wt.\%)} = \frac{\sum \text{Weight of converted FAME}}{\text{Weight of soybean oil}} \times 100.$$

3. Results and Discussions

3.1. Characterisation of PTW and SS. Before fabricating the metal/carbon composite (PSC), FT-IR spectroscopy was performed on the PTW to identify its specific chemical characteristics. The FT-IR spectrum was recorded over a range from 650 to 4000 cm⁻¹. The resulting FT-IR spectrum of PTW is illustrated in Figure 1, which also includes the FT-IR spectrum of the standard polyester for comparison. This reference spectrum helps confirm the presence and composition of polyesters in PTW.

FT-IR analysis of the PTW revealed several characteristic peaks indicative of its chemical composition. The peak at 2980 cm⁻¹ corresponds to the =C–H bond in the benzene ring [29]. The peak at 1718 cm⁻¹ indicates the presence of the C=O bond in the ester [30], whereas the peak at 1505 cm⁻¹ corresponds to the C=C bond in the benzene ring [29]. The 1413 cm⁻¹ peak is associated with the C–C–H bond in the benzene ring, and the 1249 cm⁻¹ peak corresponds to the C–O–C bond in the ester [31]. The 1100 cm⁻¹ peak also represents another C=O bond in ester [31]. The peaks at 873 and 724 cm⁻¹ correspond to the para-disubstituted benzene ring and C–H bond, respectively [32]. The FT-IR spectrum of PTW closely resembles that of polyester fibres, confirming that PTW comprises polyester fibres.

Elemental and ICP analyses were conducted to determine the elemental composition and metal content of PTW and SS, and the results are summarised in Table 1. As shown in Table 1, PTW consisted of 62.61 wt.% carbon and 4.27 wt.% hydrogen, with no presence of inorganic species. The high carbon content of PTW suggests its potential

TABLE 1: Elemental and ICP analyses of the PTW and SS samples.

Sample	Elemental analysis (wt.%)			ICP analysis (wt.%)		
	Carbon	Hydrogen	Calcium	Aluminium	Iron	Magnesium
PTW	62.61	4.27	—	—	—	—
SS	4.89	1.60	36.81	2.82	5.15	1.42

Abbreviations: ICP, inductively coupled plasma; PTW, polyester textile waste; SS, steel slag.

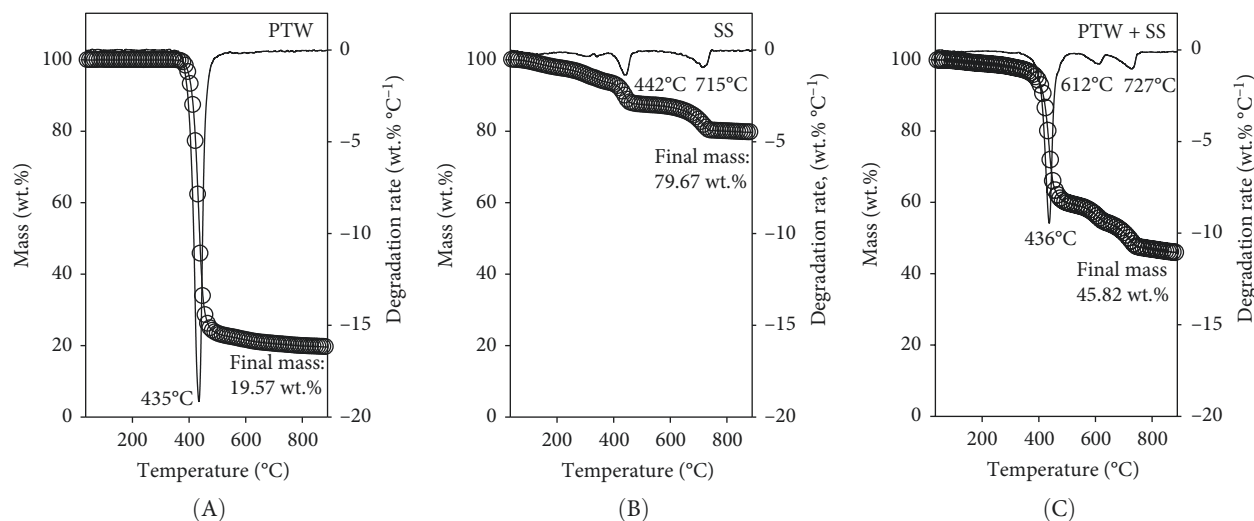


FIGURE 2: Thermolytic mass change and degradation rate of (A) PTW, (B) SS, and (C) PTW + SS. PTW, polyester textile waste; SS, steel slag.

to serve as a porous structure on the metal/carbon composite. In contrast, SS comprises 4.89 wt.% carbon and 1.60 wt.% hydrogen, along with 36.81 wt.% calcium, 2.82 wt.% aluminium, 5.15 wt.% iron, and 1.42 wt.% magnesium. The high inorganic content of SS offers its potential to function as an active site, contributing to the catalytic effects in the metal/carbon composite.

To determine the temperature required for fabricating PSC through the pyrolysis of PTW and SS, the thermolytic behaviours of the PTW and SS samples were characterised using a TGA unit. Each TGA test was conducted from 35 to 900°C at a heating rate of 10°C min⁻¹ under an N₂ atmosphere. The mass changes and thermal degradation rates of PTW, SS, and PTW + SS with increasing temperatures are shown in Figure 2.

As shown in Figure 2A, mass changes in the PTW were observed in the two temperature regimes. In the first regimes (340–540°C), 77.49 wt.% of mass change occurred, which was attributed to the liberation of volatile matter (VM) through PTW pyrolysis. In the second regime (540–900°C), a small mass change (2.94 wt.%) was realised, and the thermal degradation rate was noticeably decelerated, indicating that the remaining residue underwent carbonisation through dehydrogenation, resulting in char formation. These findings suggest that PTW char should be fabricated at >540°C. As shown in Figure 2B, the total mass change in the SS is 20.33 wt.%, resulting in a final mass of 79.67 wt.%, which is nearly identical to its ash content (79.16 wt.%). This suggests that the mass change in SS can be attributed to the degradation of inorganic species containing calcium,

aluminium, iron, and magnesium (Table 1). The thermolysis of SS was observed under two temperature regimes. In the first temperature regime (380–500°C), the mass change was attributed to the thermal decomposition of calcium hydroxide (Ca(OH)₂) through dehydration [33]. In the second temperature regime (610–740°C), mass change was associated with the release of CO₂ from calcium carbonate (CaCO₃). Consequently, Ca(OH)₂ and CaCO₃ were likely transformed into calcium oxide (CaO) [27]. As shown in Figure 2C, the thermal degradation of PTW + SS occurs between 340 and 740°C, with three distinct degradation peaks at 436, 612, and 727°C. The peaks at 436 and 727°C correspond to the thermal degradation of PTW and SS, respectively. Unlike the TGA results for PTW and SS alone, a distinct peak at 612°C was observed for PTW + SS. The residual masses of the PTW and SS at 500°C were 23.39 and 87.56 wt.%, respectively; therefore, the expected residual mass of a 1:1 blend of PTW + SS at 500°C would be 59.88 wt.%. However, the actual residual mass of the PTW + SS was 4.4 wt.% higher than this expected value, indicating that chemicals from the thermolysis of PTW interacted with and deposited onto SS. These findings suggest that the distinguishable peak at 612°C was likely due to the liberation of chemicals deposited on SS originating from PTW pyrolysis. Considering that most VM from the pyrolysis of PTW and SS is liberated below 640°C, the fabrication of PSC was conducted at 700°C.

3.2. Pyrolysis of PTW and SS. TGA is limited to detecting mass changes in PTW + SS with respect to temperature [34]. To further elucidate the mechanism of PSC formation,

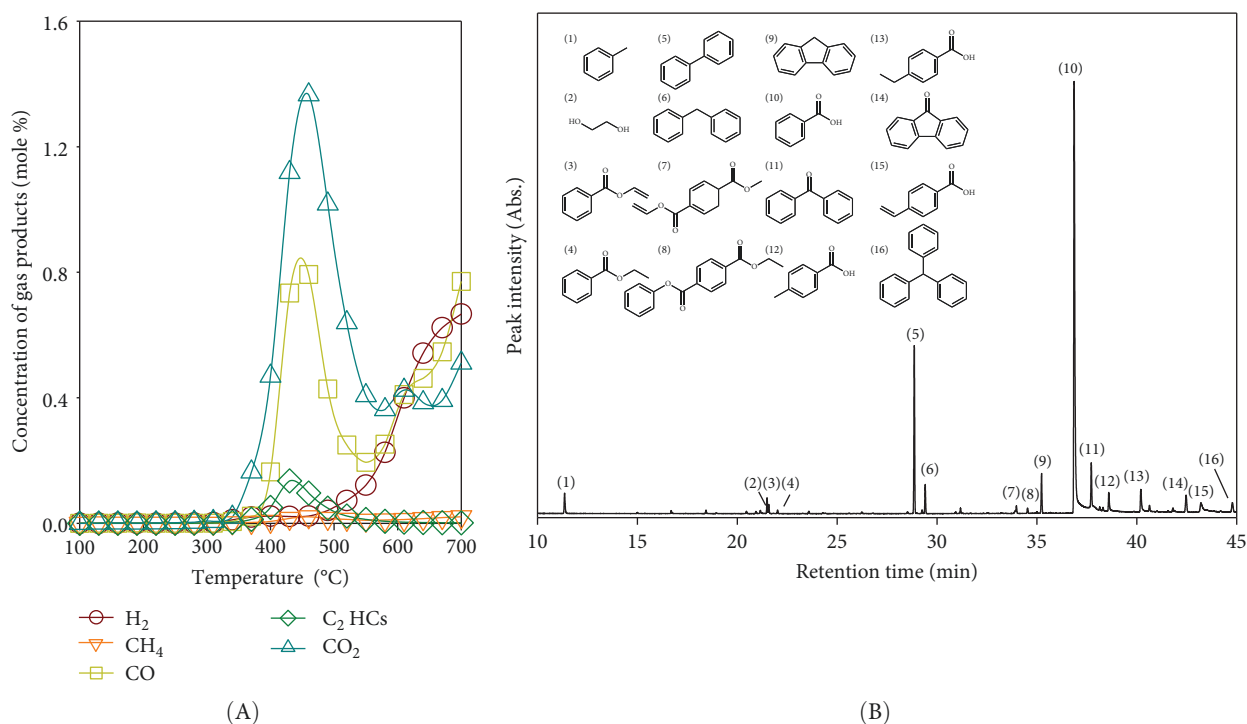


FIGURE 3: (A) Concentration profiles of H₂, CH₄, CO, C₂-HCs, and CO₂ and (B) GC/MS chromatogram of liquid pyrolysates from pyrolysis of PTW and SS. GC, gas chromatography; HCs, hydrocarbons; MS, mass spectrometry; PTW, polyester textile waste; SS, steel slag.

pyrolysis of PTW and SS was conducted from 35 to 700°C under N₂. The concentration profiles of non-condensable gases (H₂, CH₄, CO, C₂-HCs, and CO₂) released from the co-pyrolysis of PTW with SS are depicted in Figure 3A.

As shown in Figure 3A, the evolution of H₂ is observed at ≥490°C, which indicates that carbonisation through dehydrogenation occurs at ≥490°C. As the temperature increased, the formation of H₂ intensified, suggesting that the carbonisation rate of PTW + SS increased proportionally with the pyrolysis temperature. Based on this, the resulting PSC was isothermally maintained at 700°C to provide sufficient time for carbonisation. The formation of H₂ commenced at a higher temperature than that of other gases (CH₄, CO, C₂ HCs, and CO₂), which was attributed to the relatively higher bond energy of the CH bond (416 kJ mol⁻¹) compared to the C—O (346 kJ mol⁻¹) and CC (358 kJ mol⁻¹) bonds [35–37]. The formation of CO and CO₂ was observed at ≥370°C, which was attributed to deoxygenation (decarboxylation and decarbonylation) from the cleavage of the ester bond in PTW [38, 39]. Interestingly, the evolution patterns of CO and CO₂ during the pyrolysis of PTW and SS differ from the general trends observed during the pyrolysis of polyesters [40]. Specifically, additional formation of CO and CO₂ was observed at ≥550°C. The increased formation of CO and CO₂ could be explained by two possible pathways. First, the deoxygenation of chemicals deposited onto the SS likely occurred. Between 400 and 550°C, the concentrations of CO and CO₂ from the pyrolysis of PTW with and were lower than those from the pyrolysis of PTW alone (data not shown). These findings suggest that carbon was deposited onto SS through the interaction between chemicals from the pyrolysis

of PTW and SS and subsequently released as CO and CO₂ at ≥550°C. Second, CO₂ evolved through the thermal degradation of CaCO₃ into CaO and likely contributed to the formation of CO through gas-phase reactions with the VM liberated from the thermolysis of PTW. Our previous studies demonstrated this pathway [41]. This suggests that calcium may be present as CaO in the resulting PSC. The chemical structure of the resulting PSC was closely related to the formation of condensable VM liberated during the pyrolysis of PTW and SS [42]. For an in-depth study, condensable VM (liquid pyrolysate) was further analysed using GC/MS (Figure 3B). The peak areas of the chemicals identified in the liquid pyrolysate are summarised in Table 2.

As shown in Figure 3B and Table 2, various chemicals are produced during the pyrolysis of PTW with SS. The liquid pyrolysate contained benzoic acid, terephthalic acid, and benzene derivatives. These chemical compounds can result from the thermal cracking of ester linkages in PTW. Specifically, at >300°C, ester linkages undergo a six-membered transition, resulting in the transfer of β-H to C=O and the consequent formation of carboxylic acid and vinyl groups at the end of the chain [43, 44]. Subsequent decarboxylation and ethylene cleavage led to the formation of benzoic acid and benzene derivatives [45]. Compared to the liquid pyrolysate from PTW pyrolysis alone (data not shown), benzophenone (19.12 area%), diphenylmethane (11.12 area%), and triphenylmethane (6.58 area%) were additionally formed during the pyrolysis of PTW and SS. These findings can be explained by the alkaline metals in SS, especially CaO, which have a catalytic impact [46, 47]. For instance, CaO catalyses the decarboxylation of benzoic acid, releasing CO₂ and H₂O

TABLE 2: The peak areas of the major chemical species in liquid pyrolysate obtained from pyrolysis of PTW and SS.

No.	RT (min)	Chemical species	Peak area (Abs. $\times 10^5$)
1	11.35	Toluene	9.62
2	21.15	1,2-Ethanediol	1.30
3	21.49	Vinyl benzoate	5.35
4	22.01	Benzoic acid ethyl ester	1.28
5	28.87	Biphenyl	64.89
6	29.41	Diphenylmethane	11.12
7	33.98	1-Methyl 4-vinyl terephthalate	2.23
8	34.53	Ethyl phenyl ester terephthalic acid	2.32
9	35.24	Fluorene	16.20
10	36.87	Benzoic acid	255.67
11	37.72	Benzophenone	19.12
12	38.60	4-Methyl-benzoic acid	9.43
13	40.20	4-Ethylbenzoic acid	12.58
14	42.47	9H-fluoren-9-one	10.76
15	43.22	4-Vinylbenzoic acid	17.64
16	44.79	Triphenylmethane	6.58
	—	Total	446.08

Abbreviations: PTW, polyester textile waste; RT, retention time; SS, steel slag.

and resulting in the formation of benzophenone. These findings indicate an increase in aromatic components, which serve as precursors for char formation [48]. Thus, highly aromatic PSC was produced during the pyrolysis of PTW and SS.

3.3. Characterisation of PSC. The main purpose of this study was to investigate the effectiveness of PSC as catalysts for biodiesel production via thermally induced transesterification. Prior to utilising the PSC as a catalyst, its physical and chemical properties were determined. The surface properties of the PSC were examined using SEM/EDX spectrometry. Images and atomic distributions on the surface of the PSC are shown in Figure 4, with the PTW char included for comparison. The surface of the PTW char exhibits a plate-like morphology in the SEM images, and the mapping image indicates that it comprises carbon. This high carbon content suggests that the pyrolysis temperature and isothermal retention time (RT) were sufficient to form a carbon-rich char through carbonisation. As shown in Figure 4, the PSC exhibits a rough surface. This roughness could be attributed to the fabrication process. Specifically, the PTW transitions to a more fluid state at conversion temperatures, disperses onto the surface of the SS, and undergoes carbonisation to form PSC. These processes resulted in a rough appearance of the PSC surface. From the mapping image of the PSC, the carbon content decreased, whereas the oxygen, calcium, and iron contents increased compared to those of the PTW char. This indicates that PTW provides solid carbon, whereas SS supplies alkaline/heavy metals to the PSC. Consequently, the pyrolysis of the PTW and SS resulted in the formation of a metal/carbon composite that imparted catalytic effects on the surface. However, SEM/EDX analyses are limited to the detection of surface atoms and cannot accurately determine the quantity of metals in PSC. To determine the metal

content of the PSC, ICP-OES analysis was conducted, which confirmed that it contained 35.3 wt.% of calcium, 4.58 wt.% of iron, 2.72 wt.% of aluminium, and 1.71 wt.% of magnesium.

The surface area, total pore volume, and mean pore diameter of PTW char and PSC were determined using BET/Barrett–Joyner–Halenda (BJH) analyses. The results are presented in Figure 5 and summarised in Table 3. Figure 5A shows the hysteresis loops of the PTW char and PSC, which depend on their porous structure and pore size. For the PTW char, a Type I hysteresis loop was observed, indicating the development of narrow micropores (MPs) (< 2 nm) [49]. Indeed, the MP size distribution was dominant in the PTW char (Figure 5B). Compared to the PTW char, the surface area of the PSC decreased, whereas the pore size showed more pronounced development. The hysteresis loop for the PSC can be categorised as Type IV-H3, indicating the development of parallel-plate-shaped mesopores (2–50 nm) [50]. These findings suggest that SS contributes to the formation of mesopores in PSC through the pyrolysis of PTW and SS. Based on these findings, it can be concluded that PSC possess enriched mesopores and inorganic content, making them suitable porous materials and catalysts for biodiesel production via thermally induced transesterification [27].

3.4. Application of PSC in Thermally Induced Transesterification. It is critical to characterise the thermolytic behaviour of soybean oil for thermally induced transesterification. To this end, a TGA test of soybean oil was conducted from 35 to 900°C under a N_2 atmosphere. The mass changes and thermal degradation rates of the soybean oil with increasing temperature are shown in Figure 6.

The mass change in soybean oil was observed from 310 to 490°C, with the fastest thermolytic rate occurring at 419°C. The final residual mass was negligible. These thermolytic patterns are identical to the thermolysis patterns of TGs [51],

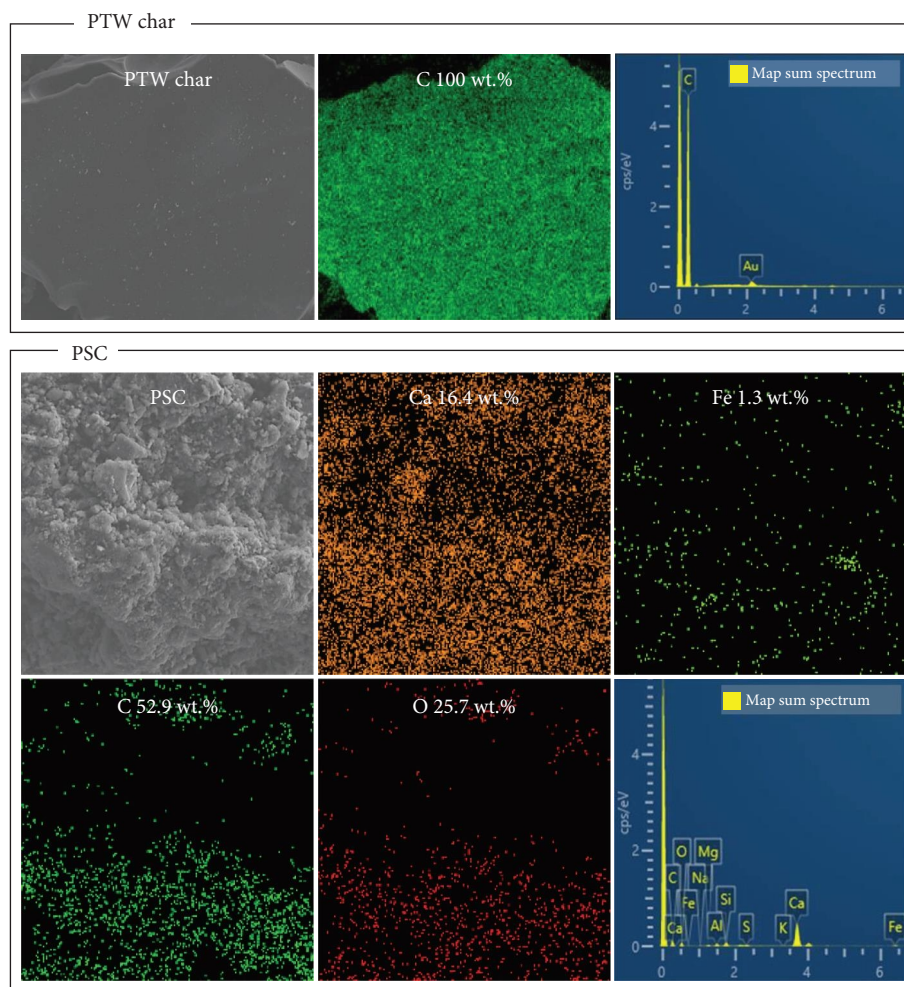


FIGURE 4: SEM/EDX images of PTW char and PSC. PSC, polyester textile waste + steel slag composite; PTW, polyester textile waste; SEM/EDX, scanning electron microscopy/energy-dispersive X-ray spectroscopy.

indicating that soybean oil consists solely of TGs. TGs do not have a specific boiling point because three different FAs are randomly attached to the glycerol backbone [52]. The thermal degradation of TGs begins with the dissociation of FAs from the glycerol backbone, followed by their sequential vaporisation [53]. Thus, the mass loss of the soybean oil was attributed to the vaporisation of FAs from the glycerol backbone in the TGs. The initial step for the transesterification reaction is the dissociation of FAs from glycerol backbones, which indicates that transesterification of soybean oil can be thermally induced at $\geq 310^{\circ}\text{C}$. The boiling point of MeOH is 65°C [54]. This infers that a heterogeneous reaction between MeOH (gas phase) and soybean oil (liquid phase) is developed at $\geq 310^{\circ}\text{C}$. The two different mobilities of the reactants lead to the deceleration of the transesterification reaction kinetics [55]. To expedite the reaction kinetics, porous materials such as silica have been employed to provide a confined space that enhances the collision frequency of the reactants [56]. To ensure the effectiveness of this approach, the pore size of the porous material must exceed 2 nm, which corresponds to the TG length. Therefore, PSC with large mesopores (2–50 nm) could be suitable porous materials for thermally induced transesterification.

To assess the reliability of the thermally induced transesterification, conventional transesterification of soybean oil was performed using a KOH catalyst (Figure 7). The biodiesel yield from the alkali-catalysed transesterification was 93.82 wt.% after 60 min of reaction at 63°C . However, the yield was <95 wt.%, likely due to the reverse reaction of the transesterification. In contrast, the biodiesel yield obtained from thermally induced transesterification using silica was 97.20 wt.% within 1 min of reaction at 360°C . This indicates that thermally induced transesterification achieves a higher biodiesel yield in a shorter reaction time than alkali-catalysed transesterification. Furthermore, the biodiesel obtained from alkali-catalysed and thermally induced transesterification comprised $\text{C}_{16:0}$, $\text{C}_{18:0}$, $\text{C}_{18:1}$, $\text{C}_{18:2}$, $\text{C}_{18:3}$, $\text{C}_{20:0}$, $\text{C}_{20:1}$, and $\text{C}_{22:0}$ FAMES with nearly identical mass compositions. This demonstrated that thermally induced transesterification is a technically reliable method for biodiesel production.

Nonetheless, a high temperature (360°C) was required to achieve the maximum biodiesel yield of 97.2 wt.% from thermally induced transesterification using silica. Economically, reducing the reaction temperature is necessary. To address this, thermally induced transesterification of soybean oil

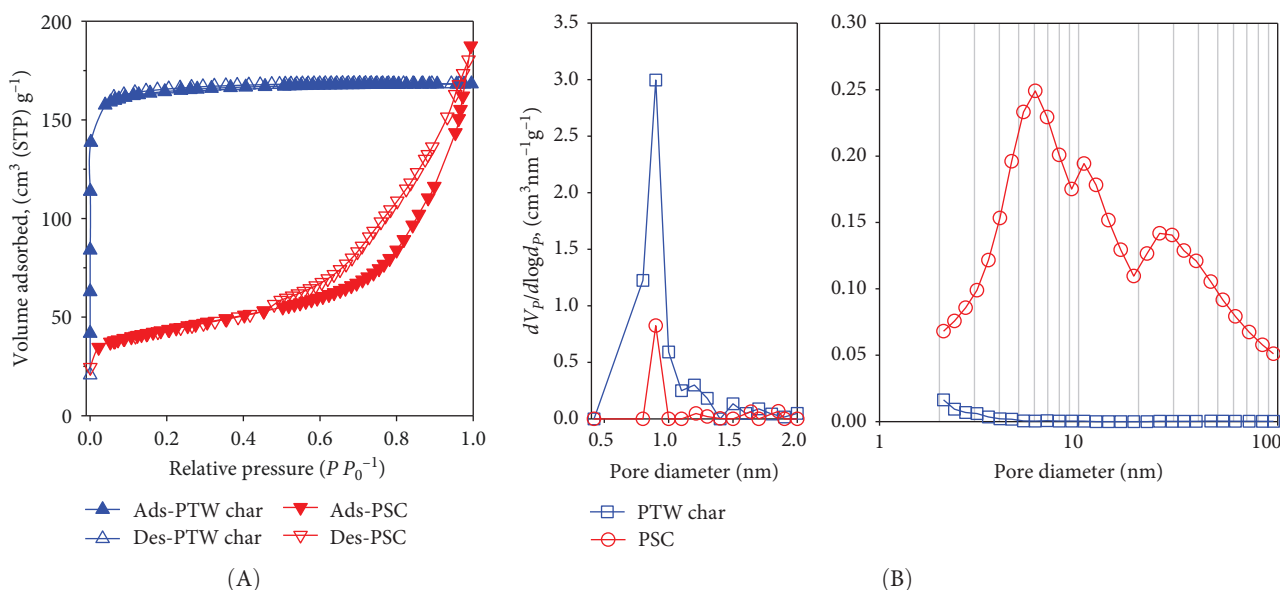


FIGURE 5: (A) N₂ adsorption/desorption isotherms and (B) BJH and MP plots of PTW char and PSC. BJH, Barrett–Joyner–Halenda; MP, micropore; PSC, polyester textile waste + steel slag composite; PTW, polyester textile waste; STP, standard temperature (0°C) and pressure (1 bar).

TABLE 3: Surface area, total pore volume, and mean pore diameter of PTW char and PSC.

Sample	Surface area (m ² g ⁻¹)	Total pore volume (cm ³ g ⁻¹)	Mean pore diameter (nm)
PTW char	652.42	0.2602	1.595
PSC	156.36	0.2827	7.233

Abbreviations: PSC, polyester textile waste + steel slag composite; PTW, polyester textile waste.

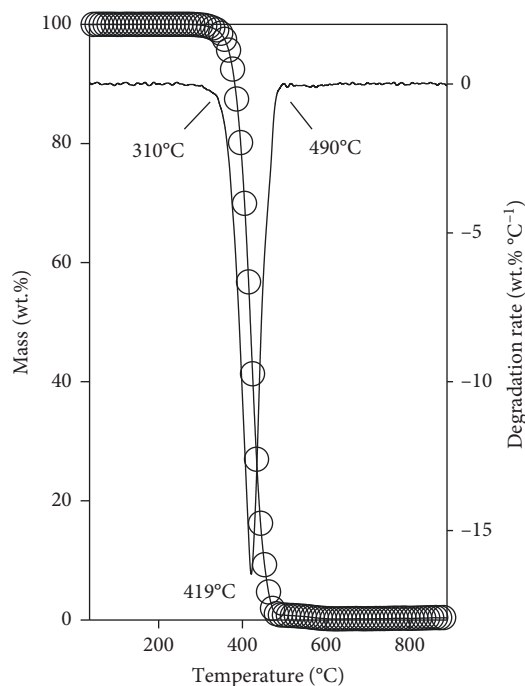


FIGURE 6: Thermolytic mass change and degradation rate of soybean oil.

using PSC was conducted, and silica and PTW char were used for comparison. Thermal energy is the driving force for thermally induced transesterification. Therefore, the thermally induced transesterification of soybean oil was performed at various reaction temperatures in the presence of silica, PTW char, and PSC. The biodiesel yield profiles and their compositional matrices are shown in Figure 8 as a function of temperature.

For silica, biodiesel conversion was initiated at 200°C, and the yield increased with increasing temperature, reaching a maximum of 97.2 wt.% at 360°C. These observations indicated that heat energy was the driving force of thermally induced transesterification. As shown in Figure 8B, FAME compositional ratios obtained using silica are nearly identical, indicating that the thermal cracking of FAME is not realised at <370°C. For the PTW char, biodiesel conversion began at 250°C. Similar to the case of silica, the yield increased with increasing temperature. However, the maximum yield was significantly lower than that with silica, reaching 23.3 wt.% at 370°C. As discussed, PTW char has a high surface area but predominantly comprises MPs (<2 nm), which are smaller than the size of the TGs. This indicated that the low biodiesel yield was due to the presence of MPs in the PTW char. In addition, the compositional ratio of C_{18:2} FA in biodiesel

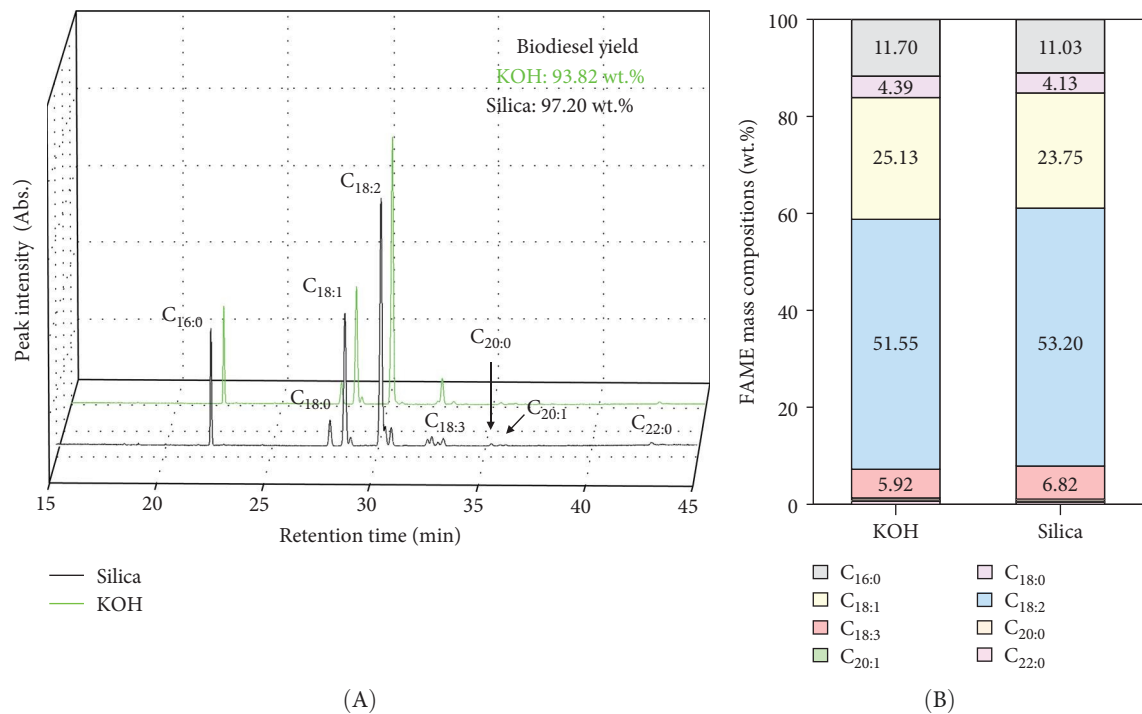


FIGURE 7: (A) GC/MS chromatograms of biodiesel obtained from alkali-catalysed transesterification using KOH at 63°C for 60 min and thermally induced transesterification using silica at 360°C within 1 min, and (B) FAME mass compositions in biodiesel. FAME, fatty acid methyl ester; GC/MS, gas chromatography/mass spectrometry; KOH, potassium hydroxide.

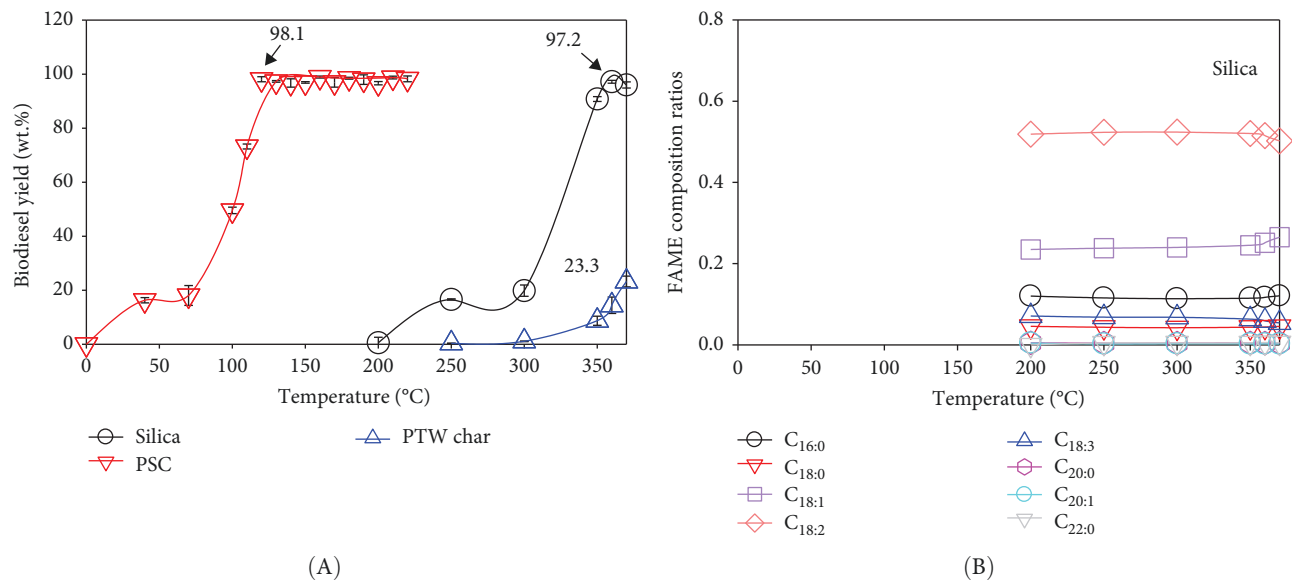


FIGURE 8: Continued.

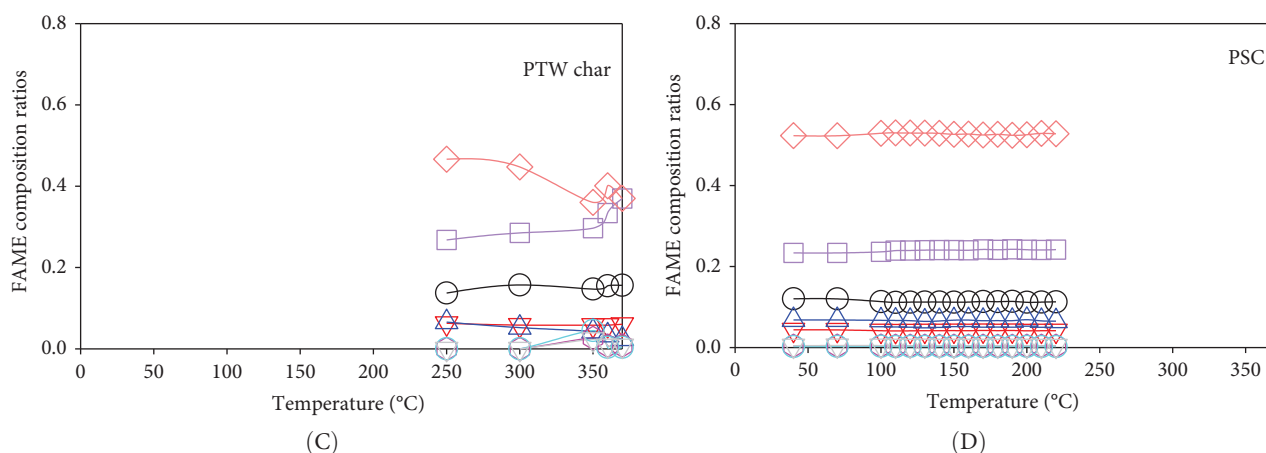


FIGURE 8: (A) Biodiesel yields from thermally induced transesterification of soybean oil as a function of temperature, and their FAME compositional ratios obtained using (B) silica, (C) PTW char, and (D) PSC. FAME, fatty acid methyl ester; PSC, polyester textile waste + steel slag composite; PTW, polyester textile waste.

TABLE 4: Comparison of conditions for biodiesel production.

Feedstock	Type of catalyst	Metal loading (wt.%)	Support material	Reaction condition	Biodiesel yield per oil (wt.%)	Refs.
Heterogeneous reaction						
Sunflower oil	CaO/Al ₂ O ₃	10.1	Al ₂ O ₃	60°C for 300 min	91.5	[58]
Palm oil	K-CaO/Al ₂ O ₃	10.0	Al ₂ O ₃	65°C for 300 min	95.5	[59]
Soybean oil	CaO/AC-2	5.0	Coconut shell	65°C for 300 min	98.6	[60]
Cator oil	Ca/C	38.7	Bagasse	65°C for 180 min	95.4	[61]
Sunflower oil	Ca/C	20.0	Avocado seeds	99.5°C for 300 min	99.5	[62]
Thermally induced reaction (this study)						
Soybean oil	—	—	—	360°C for 1 min	97.2	This study
Soybean oil	Steel slag/C	35.3	Polyester	120°C for 1 min	98.1	This study

obtained using PTW char decreased at >350°C. This reduction can be attributed to the interaction between the pyrolysates fixed in the PTW char and C_{18:2} FA during thermally induced transesterification. This interaction was evidenced by the formation of compounds such as methyl 7-(2-pentyl phenyl)heptanoate and (1-methyl hexadecyl)benzene (data not shown). Unlike PTW char, PSC has well-developed mesopores (2–50 nm), making them more suitable porous materials for thermally induced transesterification. For the PSC, the conversion of biodiesel was initiated at 40°C, and the yield increased with increasing temperature, reaching 98.1 wt.% at 120°C. This was 240°C lower than the temperature required to achieve maximum biodiesel yield using silica. This enhanced efficiency is likely due to the catalytic effect of alkaline metals, particularly CaO, present in the PSC, which accelerates the transesterification reaction [57]. Additionally, the biodiesel yield plateaued at >200°C, and the FAME compositional ratios remained nearly identical at these temperatures. These findings suggest that the yield was saturated, indicating that a high biodiesel yield could be achieved without further cracking of the FAME.

For comparison purposes, the results from thermally induced transesterification are summarised in Table 4 alongside other reported heterogeneous reactions. Given that PSC

contains a high content of Ca (35.3 wt.%), a Ca-based catalyst was selected for comparison. Although the biodiesel yield from the thermally induced reaction was comparable to previous research, the reaction kinetics were significantly faster. Specifically, other studies required long reaction times (>180 min) to achieve a biodiesel yield of >95 wt.% in heterogeneous reactions. In contrast, the thermally induced reaction produced biodiesel in just 1 min. Despite this advantage, one technical limitation remains. The current study was conducted using a small 1 mL volume reactor to avoid heat transfer challenges and gain a fundamental understanding of the reaction. Therefore, further research is necessary to scale up the process to ensure its feasibility for industrial applications in the future.

4. Conclusions

This study investigated the pyrolysis of PTW with SS to enhance the physicochemical properties of PTW char. During the pyrolysis process, the alkaline metals in the SS facilitated the carbonisation of PTW, producing PSC with a high carbon content. CaO was also formed in PSC through the decarboxylation of CaCO₃, providing a catalytic effect beneficial for biodiesel production. The mean pore diameter of

the PTW char increased from 1.6 to 7.2 nm in PSC, which can contribute to facilitating biodiesel conversion through thermally induced transesterification. To confirm this, soybean oil was selected as the model compound. The biodiesel production was tested using commercial silica, PTW char, and PSC. The maximum biodiesel yield obtained using silica was 97.2 wt.% at 360°C, which was significantly higher than the 23.3 wt.% yield from PTW char at 370°C. The low yield of PTW char was attributed to the presence of MPs (<2 nm). In contrast, the PSC achieved a maximum biodiesel yield of 98.1 wt.% at 120°C, which was 240°C lower than the temperature required to achieve similar yields with silica. This remarkable efficiency is due to the catalytic effect of alkaline metals (particularly CaO) and the well-developed mesopores in the PSC. These results demonstrate that PSCs are effective metal/carbon composites for biodiesel production and present a promising strategy for converting industrial waste into valuable resources.

Data Availability Statement

Data will be made available upon request.

Conflicts of Interest

The authors declare no conflicts of interest.

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References

- [1] N. Nørup, K. Pihl, A. Damgaard, and C. Scheutz, "Development and Testing of a Sorting and Quality Assessment Method for Textile Waste," *Waste Management* 79 (2018): 8–21.
- [2] M. Kahoush and N. Kadi, "Towards Sustainable Textile Sector: Fractionation and Separation of Cotton/Polyester Fibers From Blended Textile Waste," *Sustainable Materials and Technologies* 34 (2022): e00513.
- [3] A. Danish and T. Ozbakkaloglu, "2 - Fiber Classifications and Physical and Mechanical Properties of Different Fibers Used in Alkali-Activated Composites," in *Advanced Fiber-Reinforced Alkali-Activated Composites*, eds. A. Çevik and A. Niş, (Elsevier, 2023): 23–58.
- [4] R. M. Frazier, K. A. Vivas, I. Azuaje, et al., "Beyond Cotton and Polyester: An Evaluation of Emerging Feedstocks and Conversion Methods For the Future of Fashion Industry," *Journal of Bioresources and Bioproducts* 9, no. 2 (2024): 130–159.
- [5] K. Ghosal and C. Nayak, "Recent Advances in Chemical Recycling of Polyethylene Terephthalate Waste Into Value Added Products For Sustainable Coating Solutions—Hope Vs. Hype," *Materials Advances* 3, no. 4 (2022): 1974–1992.
- [6] S. Yousef, J. Eimontas, N. Striūgas, et al., "A Sustainable Bioenergy Conversion Strategy For Textile Waste With Self-Catalysts Using Mini-Pyrolysis Plant," *Energy Conversion and Management* 196 (2019): 688–704.
- [7] C. Stone, F. M. Windsor, M. Munday, and I. Durance, "Natural or Synthetic—How Global Trends in Textile Usage Threaten Freshwater Environments," *Science of The Total Environment* 718 (2020): 134689.
- [8] M. K. Awasthi, T. Sar, S. C. Gowd, et al., "A Comprehensive Review on Thermochemical, and Biochemical Conversion Methods of Lignocellulosic Biomass Into Valuable End Product," *Fuel* 342 (2023): 127790.
- [9] A. Williams, J. M. Jones, L. Ma, and M. Pourkashanian, "Pollutants From the Combustion of Solid Biomass Fuels," *Progress in Energy and Combustion Science* 38, no. 2 (2012): 113–137.
- [10] X. Jiang, D. Chen, Z. Ma, and J. Yan, "Models For The Combustion of Single Solid Fuel Particles in Fluidized Beds: A Review," *Renewable and Sustainable Energy Reviews* 68 (2017): 410–431.
- [11] A. K. Panda, R. K. Singh, and D. K. Mishra, "Thermolysis of Waste Plastics to Liquid Fuel: A Suitable Method For Plastic Waste Management and Manufacture of Value Added Products—A World Prospective," *Renewable and Sustainable Energy Reviews* 14, no. 1 (2010): 233–248.
- [12] Y. Peng, Y. Wang, L. Ke, et al., "A Review on Catalytic Pyrolysis of Plastic Wastes to High-Value Products," *Energy Conversion and Management* 254 (2022): 115243.
- [13] P. Straka, O. Bičáková, and M. Šupová, "Slow Pyrolysis of Waste Polyethylene Terephthalate Yielding Paraldehyde, Ethylene Glycol, Benzoic Acid and Clean Fuel," *Polymer Degradation and Stability* 198 (2022): 109900.
- [14] S. Sharifian and N. Asasian-Kolur, "Polyethylene Terephthalate (PET) Waste to Carbon Materials: Theory, Methods and Applications," *Journal of Analytical and Applied Pyrolysis* 163 (2022): 105496.
- [15] R. Blanchard and T. H. Mekonnen, "Synchronous Pyrolysis and Activation of Poly(Ethylene Terephthalate) For The Generation of Activated Carbon For Dye Contaminated Wastewater Treatment," *Journal of Environmental Chemical Engineering* 10, no. 6 (2022): 108810.
- [16] P. Renforth, C. L. Washbourne, J. Taylder, and D. A. C. Manning, "Silicate Production and Availability For Mineral Carbonation," *Environmental Science & Technology* 45, no. 6 (2011): 2035–2041.
- [17] G. Costa, A. Poletti, R. Pomi, and A. Stramazzo, "Leaching Modelling of Slurry-Phase Carbonated Steel Slag," *Journal of Hazardous Materials* 302 (2016): 415–425.
- [18] R. Nisticò, "Polyethylene Terephthalate (PET) in The Packaging Industry," *Polymer Testing* 90 (2020): 106707.
- [19] D. He, L. Yang, Y. Luo, G. Liu, and Z. Wu, "Synergistic Calcium Leaching and Iron Enrichment By Indirect Carbonation of Thermally Modified Steel Slag," *Construction and Building Materials* 411 (2024): 134249.
- [20] N. Singh, R. K. Saluja, H. J. Rao, et al., "Progress and Facts On Biodiesel Generations, Production Methods, Influencing Factors, and Reactors: A Comprehensive Review From 2000 to 2023," *Energy Conversion and Management* 302 (2024): 118157.
- [21] E. M. Shahid and Y. Jamal, "Production of Biodiesel: A Technical Review," *Renewable and Sustainable Energy Reviews* 15, no. 9 (2011): 4732–4745.
- [22] H. Pan, Q. Xia, Y. Wang, et al., "Recent Advances in Biodiesel Production Using Functional Carbon Materials As Acid/Base Catalysts," *Fuel Processing Technology* 237 (2022): 107421.

- [23] T. Issariyakul and A. K. Dalai, "Biodiesel From Vegetable Oils," *Renewable and Sustainable Energy Reviews* 31 (2014): 446–471.
- [24] S. Sinha, A. K. Agarwal, and S. Garg, "Biodiesel Development From Rice Bran Oil: Transesterification Process Optimization and Fuel Characterization," *Energy Conversion and Management* 49, no. 5 (2008): 1248–1257.
- [25] E. E. Kwon, H. Yi, J. Park, and J. Seo, "Non-Catalytic Heterogeneous Biodiesel Production Via a Continuous Flow System," *Bioresource Technology* 114 (2012): 370–374.
- [26] S. Jung, M. Kim, K.-Y. A. Lin, Y.-K. Park, and E. E. Kwon, "Biodiesel Synthesis From Bio-Heavy Oil Through Thermally Induced Transesterification," *Journal of Cleaner Production* 294 (2021): 126347.
- [27] Y. Tang, J. Xu, J. Zhang, and Y. Lu, "Biodiesel Production From Vegetable Oil By Using Modified CaO As Solid Basic Catalysts," *Journal of Cleaner Production* 42 (2013): 198–203.
- [28] B. Nath, B. Basumatary, N. Wary, et al., "Agricultural Waste-Based Heterogeneous Catalyst For the Production of Biodiesel: A Ranking Study via the VIKOR Method," *International Journal of Energy Research* 2023, no. 1 (2023): 1–23.
- [29] S. Farah, T. Tsach, A. Bentolila, and A. J. Domb, "Morphological, Spectral and Chromatography Analysis and Forensic Comparison of PET Fibers," *Talanta* 123 (2014): 54–62.
- [30] J. Feng, S. Xu, G. Pan, et al., "Clean Preparation of Washable Antibacterial Polyester Fibers by High Temperature and High Pressure Hydrothermal Self-Assembly," *Nanotechnology Reviews* 10, no. 1 (2021): 1740–1751.
- [31] Y. Cai, L. Li, T. Wang, et al., "The Optimization of Whiteness of Polyester Fabric Treated With Nanoparticles of 2,2'-(Vinylenedi-p-Phenylene) bis-Benzoxazole (OB-1) by the Taguchi Method," *Colloids and Surfaces A: Physicochemical and Engineering Aspects* 676 (2023): 132320.
- [32] A. B. Rezaie, M. Montazer, and M. M. Rad, "Photo and Biocatalytic Activities Along With UV Protection Properties on Polyester Fabric Through Green in-situ Synthesis of Cauliflower-Like CuO Nanoparticles," *Journal of Photochemistry and Photobiology B: Biology* 176 (2017): 100–111.
- [33] Z. Ma, H. Liao, and F. Cheng, "Synergistic Mechanisms of Steelmaking Slag Coupled With Carbide Slag for CO₂ Mineralization," *International Journal of Greenhouse Gas Control* 105 (2021): 103229.
- [34] D. López-González, M. Fernandez-Lopez, J. L. Valverde, and L. Sanchez-Silva, "Thermogravimetric-Mass Spectrometric Analysis on Combustion of Lignocellulosic Biomass," *Bioresource Technology* 143 (2013): 562–574.
- [35] C. Wei, D. Srivastava, and K. Cho, "Thermal Expansion and Diffusion Coefficients of Carbon Nanotube-Polymer Composites," *Nano Letters* 2, no. 6 (2002): 647–650.
- [36] K. Fan, J. Liu, X. Wang, et al., "Towards Enhanced Tribological Performance as Water-Based Lubricant Additive: Selective Fluorination of Graphene Oxide at Mild Temperature," *Journal of Colloid and Interface Science* 531 (2018): 138–147.
- [37] F. Zhang, Z. Song, J. Zhu, et al., "Factors Influencing CH₄/CO₂ Reforming Reaction Over Fe Catalyst Supported on Foam Ceramics Under Microwave Irradiation," *International Journal of Hydrogen Energy* 43, no. 20 (2018): 9495–9502.
- [38] L. Gong, Y. Pan, L. Cui, and X. Zhang, "Atomic Insights Into the Thermal Degradation of Polyethylene Terephthalate Combining STA-FTIR and DFT Methods," *Fuel* 364 (2024): 131067.
- [39] J. W. F. Chia, O. Sawai, and T. Nunoura, "Reaction Pathway of Poly(ethylene) Terephthalate Carbonization: Decomposition Behavior Based on Carbonized Product," *Waste Management* 108 (2020): 62–69.
- [40] D. Kwon, S. Yi, S. Jung, and E. E. Kwon, "Valorization of Synthetic Textile Waste Using CO₂ as a Raw Material in the Catalytic Pyrolysis Process," *Environmental Pollution* 268 (2021): 115916.
- [41] S. Jung, S. Lee, H. Song, Y. F. Tsang, and E. E. Kwon, "Sustainable Valorization of E-Waste Plastic Through Catalytic Pyrolysis Using CO₂," *ACS Sustainable Chemistry & Engineering* 10, no. 26 (2022): 8443–8451.
- [42] Y. Huang, S. Liu, M. A. Akhtar, et al., "Volatile-Char Interactions During Biomass Pyrolysis: Understanding the Potential Origin of Char Activity," *Bioresource Technology* 316 (2020): 123938.
- [43] G. Montaudo, C. Puglisi, and F. Samperi, "Primary Thermal Degradation Mechanisms of PET and PBT," *Polymer Degradation and Stability* 42, no. 1 (1993): 13–28.
- [44] M. Dziecioł and J. Trzeczynski, "Studies of Temperature Influence on Volatile Thermal Degradation Products of Poly (Ethylene Terephthalate)," *Journal of Applied Polymer Science* 69, no. 12 (1998): 2377–2381.
- [45] A. Dhahak, C. Grimmer, A. Neumann, et al., "Real Time Monitoring of Slow Pyrolysis of Polyethylene Terephthalate (PET) by Different Mass Spectrometric Techniques," *Waste Management* 106 (2020): 226–239.
- [46] X. Lin, Z. Zhang, Z. Zhang, J. Sun, Q. Wang, and C. U. Pittman, "Catalytic Fast Pyrolysis of a Wood-Plastic Composite With Metal Oxides as Catalysts," *Waste Management* 79 (2018): 38–47.
- [47] S. Kumagai, R. Yamasaki, T. Kameda, et al., "Aromatic Hydrocarbon Selectivity as a Function of CaO Basicity and Aging During CaO-Catalyzed PET Pyrolysis Using Tandem μ -Reactor-GC/MS," *Chemical Engineering Journal* 332 (2018): 169–173.
- [48] Z. Chen, X. Zhang, F. Yang, et al., "Deactivation of a Y-Zeolite Based Catalyst With Coke Evolution During the Catalytic Pyrolysis of Polyethylene For Fuel Oil," *Applied Catalysis A: General* 609 (2021): 117873.
- [49] Z. Wang, X. Jiang, M. Pan, and Y. Shi, "Nano-Scale Pore Structure and Its Multi-Fractal Characteristics of Tight Sandstone by N₂ Adsorption/Desorption Analyses: A Case Study of Shihezi Formation From the Sulige Gas Field, Ordos Basin, China," *Minerals* 10, no. 4 (2020): 377.
- [50] C. Shan, T. Zhang, X. Liang, et al., "On the Fundamental Difference of Adsorption-Pores Systems Between Vitrinite- and Inertinite-Rich Anthracite Derived From the Southern Sichuan Basin, China," *Journal of Natural Gas Science and Engineering* 53 (2018): 32–44.
- [51] J.-M. Jung, J.-I. Oh, D. Kwon, et al., "Synthesis of Fatty Acid Methyl Esters via Non-Catalytic Transesterification of Avocado Oil With Dimethyl Carbonate," *Energy Conversion and Management* 195 (2019): 1–6.
- [52] J.-M. Jung, M. Kim, S. Jung, et al., "Direct Conversion of *Camellia japonica* Seed into Biodiesel Through Non-Catalytic Transesterification," *Industrial Crops and Products* 174 (2021): 114194.
- [53] P. Chand, C. V. Reddy, J. G. Verkade, T. Wang, and D. Grewell, "Thermogravimetric Quantification of Biodiesel Produced via Alkali Catalyzed Transesterification of Soybean Oil," *Energy & Fuels* 23, no. 2 (2009): 989–992.
- [54] N. Ahmad, F. Javed, J. A. Awan, et al., "Biodiesel Production Intensification Through Microbubble Mediated Esterification," *Fuel* 253 (2019): 25–31.
- [55] M. Kim, J.-M. Jung, S. Jung, et al., "Biochar as a Catalyst in the Production of Syngas and Biodiesel From Peanut Waste,"

- International Journal of Energy Research* 46, no. 13 (2022): 19287–19299.
- [56] J. W. Goodrum, “Volatility and Boiling Points of Biodiesel From Vegetable Oils and Tallow,” *Biomass and Bioenergy* 22, no. 3 (2002): 205–211.
- [57] S. F. Basumatary, K. Patir, B. Das, et al., “Production of Renewable Biodiesel Using Metal Organic Frameworks Based Materials as Efficient Heterogeneous Catalysts,” *Journal of Cleaner Production* 358 (2022): 131955.
- [58] D. M. Marinković, J. M. Avramović, M. V. Stanković, O. S. Stamenković, D. M. Jovanović, and V. B. Veljković, “Synthesis and Characterization of Spherically-Shaped CaO/ γ -Al₂O₃ Catalyst and Its Application in Biodiesel Production,” *Energy Conversion and Management* 144 (2017): 399–413.
- [59] N. Chanthon, N. Munbupphachart, K. Ngaosuwan, et al., “Metal Loading on CaO/Al₂O₃ Pellet Catalyst as a Booster For Transesterification in Biodiesel Production,” *Renewable Energy* 218 (2023): 119336.
- [60] H. Sun, K. Sun, F. Wang, et al., “Catalytic Self-Activation of Ca-Doped Coconut Shell For in-Situ Synthesis of Hierarchical Porous Carbon Supported CaO Transesterification Catalyst,” *Fuel* 285 (2021): 119192.
- [61] L. Zhang, J. Xin, K. Liu, G. Wei, D. Xiong, and W. Lan, “Effective Transesterification of Castor Oil to Biodiesel Catalyzed by Novel Carbon-Based Calcium Composite,” *Energy Conversion and Management* 307 (2024): 118368.
- [62] L. di Bitonto, H. E. Reynel-Ávila, D. I. Mendoza-Castillo, A. Bonilla-Petriciolet, C. J. Durán-Valle, and C. Pastore, “Synthesis and Characterization of Nanostructured Calcium Oxides Supported Onto Biochar and Their Application as Catalysts For Biodiesel Production,” *Renewable Energy* 160 (2020): 52–66.