

Gasification Product Characteristics from Torrefied Spent Coffee Grounds in A Fixed-Bed Reactor

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Abstract

Biomass resources, particularly spent coffee grounds (SCG), offer promising potential for renewable energy generation via gasification. However, the inherent limitations of raw SCG, including high moisture and volatile content, necessitate pretreatment to enhance its suitability as a feedstock. This study evaluates the effectiveness of torrefaction as a thermal pretreatment method for SCG prior to steam gasification in a fixed-bed reactor. Comparative analyses of untreated and torrefied SCG revealed significant improvements in fuel characteristics, with torrefaction at 250 °C for 60 minutes reducing moisture content from 8.35% to 0.74%, volatile matter from 78.7% to 76.32%, and increasing fixed carbon from 12.71% to 21.74%. Optimal gasification performance was achieved at 850 °C and a holding time of 50 minutes, yielding 85.06% bio-gas, with minimal char (12.84%) and tar (2.10%) production. Characterisation of the resulting biochar indicated an increase in fixed carbon and a reduction in moisture, both of which are beneficial for syngas generation. Tar's functional group analysis revealed a high concentration of aliphatic C-H bonds, suggesting it could serve as a biofuel alternative.

1. Introduction

The global demand for cleaner, more sustainable energy alternatives has driven growing interest in renewable energy sources. Renewable energy refers to energy derived from natural resources that are replenished faster than they are consumed. These resources are abundant, environmentally friendly, and widely available. In contrast, fossil fuels such as coal, crude oil, and natural gas are categorised as non-renewable because their formation takes hundreds of millions of years.

Among renewable energy sources, biomass holds significant potential to meet the world's growing energy demands. Biomass originates from recently living organic materials such as wood, agricultural residues, municipal solid waste, animal manure, and aquatic plants [1]. Its versatility allows it to be converted into various energy forms either directly or indirectly [2], contributing to the production of transportation fuels, electricity, and heat. A notable biomass source gaining attention is spent coffee grounds (SCG). With coffee ranking as the second-most-traded commodity globally, its production generates approximately 6 million tons of SCG waste annually [3]. SCG is rich in lignin and cellulose and is typically discarded via incineration, landfilling, or composting [4]. Recently, there has been growing interest in valorising SCG for bioenergy production, driven by the need for more sustainable waste management within the coffee industry [5].

Among the various thermochemical conversion technologies, gasification stands out as an efficient process for converting solid biomass into syngas, a combustible gas mixture that includes hydrogen (H_2), carbon monoxide (CO), and methane (CH_4). Gasification involves the partial oxidation of biomass at elevated temperatures in a controlled environment with limited oxygen supply [6]. The resulting syngas can be used for power generation, direct combustion, or as a feedstock for producing liquid fuels and chemicals.

Despite its advantages, the efficiency of biomass gasification is often hindered by the inherent limitations of raw biomass, such as high moisture and volatile matter. Pretreatment methods such as torrefaction are mild thermal processes carried out at 200–300 °C. It can enhance the physicochemical properties of biomass, making it more suitable for gasification.

Therefore, this research aims to analyse the proximate composition and functional group characteristics of both untreated and torrefied SCG, determine the optimum gasification parameters (temperature and holding time) for maximising product yields (bio-char, tar, and bio-gas) and investigate the functional group of the bio-char and tar produced from the gasification of torrefied SCG.

2. Method

2.1 Preparation of SCG

Spent coffee grounds were collected from a local café in Perlis, Malaysia. The raw SCG samples were dried in an oven at 110 °C for 24 hours to reduce moisture. After drying, the samples were ground using a mechanical crusher and sieved to obtain particle sizes of 300-600 µm. The processed SCG was then stored in an airtight container to prevent moisture absorption and oxidation from the surrounding environment.

2.2 Characterisation of Untreated and Torrefied Samples

Two primary analyses were conducted to characterise the SCG and its conversion products: proximate analysis and functional group analysis. The proximate analysis was used to determine the moisture content (MC), ash content (AC), volatile matter (VM), and fixed carbon (FC) of the untreated and torrefied SCG. The ASTM method was used for proximate analysis. The fixed carbon content was calculated using Eqn. (1).

$$\% \text{ fixed carbon} = 100\% - (\% \text{ moisture content} + \% \text{ volatile matter} + \% \text{ ash content}) \quad (1)$$

The Fourier-transform infrared (FTIR) spectroscopy was employed to identify the functional groups present in untreated SCG, torrefied SCG, and tar. The analysis was conducted using a Spectrum 400 FTIR/FTNIR spectrometer equipped with an Attenuated Total Reflectance (ATR) accessory. Samples were scanned within the spectral range of 500–4000 cm^{-1} with 200 scans at a resolution of 4.0 cm^{-1} .

2.3 Torrefaction of SCG

Torrefaction was conducted using a fixed-bed reactor, as shown in Fig. 1. The reactor was operated at three distinct temperatures: 200 °C, 250 °C, and 300 °C, each maintained for 60 minutes. For each run, 5 grams of SCG were placed in a cylindrical stainless-steel tube and inserted into the reactor. Before heating, the reactor was purged with nitrogen at a flow rate of 0.5 L/min for 10 minutes to establish an inert atmosphere. The temperature was subsequently elevated to the specified setpoint and maintained at that level throughout the experiment, with a continuous supply of nitrogen.

Once the torrefaction process was complete, the reactor was allowed to cool gradually to ambient temperature. Once it had reached room temperature, the ultimate weight of the torrefied SCG was determined. Subsequently, the weight of the solid product was recorded to calculate the yield of torrefied SCG, as described in Eqn. (2).

$$\text{Mass yield} = \frac{\text{Mass of torrefied SCG (g)}}{\text{Mass of untreated SCG (g)}} \times 100\% \quad (2)$$

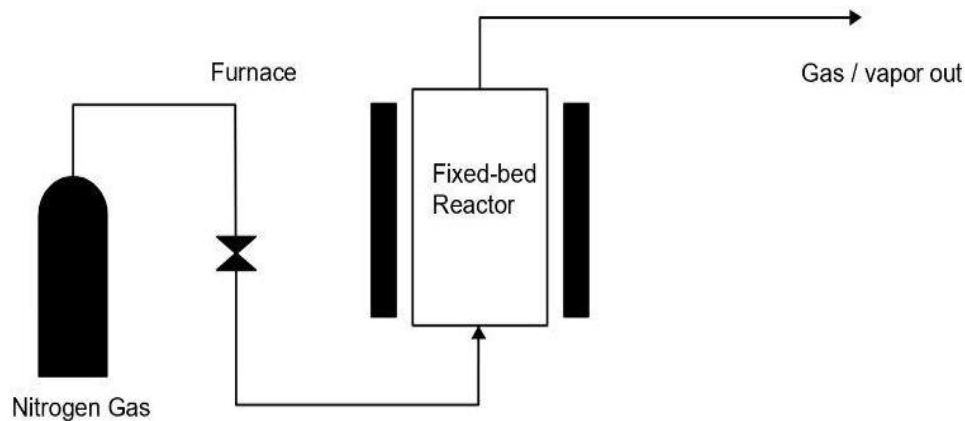


Fig. 1 Schematic diagram of torrefaction via fixed-bed reactor

2.4 Gasification of SCG

The gasification of SCG was carried out in a fixed-bed reactor. To determine the optimum temperature, the sample was gasified and evaluated at 700, 750, 800, and 850 °C. The next stage was to determine the optimum holding time, which was tested at 45, 50, 55, and 60 minutes after the optimal temperature had been determined. This process produced tar, bio-gas, and bio-char.

Five grammes of torrefied SCG were weighed and placed in the reactor. The reactor was then purged with N₂ gas for ten minutes prior to gasification. When the steam is ready, the N₂ gas valve is closed, allowing the steam to flow in during gasification. The gas bag was positioned to capture the biogas. Fig. 2 shows a schematic representation of the gasification reactor system.

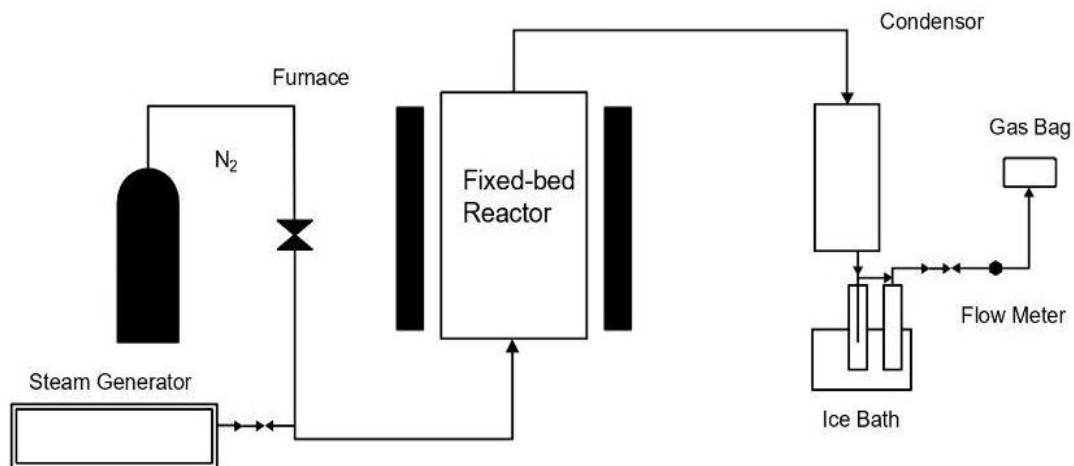


Fig. 2 Schematic diagram of gasification reactor system

The yields of biochar, tar, and biogas were determined using the mass balance of the input and output products. The yields of biochar, tar, and biogas were calculated using Eqn. (3) to Eqn. (5).

$$\% \text{ biochar yield} = \text{mass of char (g)} / \text{mass of sample (g)} \times 100 \quad (3)$$

$$\% \text{ tar yield} = \text{mass of tar (g)} / \text{mass of sample (g)} \times 100 \quad (4)$$

$$\% \text{ bio - gas yield} = 100 - (\% \text{ tar yield} + \% \text{ char yield}) \quad (5)$$

Tar collected under optimal gasification conditions was analysed using FTIR to determine its functional group. Infrared radiation in the range of 10,000 to 100 cm⁻¹ was passed through the tar sample, with absorbance spectra recorded between 4000 and 400 cm⁻¹. The resulting molecular fingerprints allowed for the identification of functional groups and hydrocarbon structures, providing insight into its fuel potential.

3. Results and Discussion

3.1 Proximate Analysis

3.1.1 Untreated SCG

The proximate analysis indicated that biomass is composed of moisture, volatile matter, ash, and fixed carbon, which are commonly reported as weight percentages. From the proximate analysis shown in Table 1, the untreated SCG has high moisture and volatile matter contents of 8.35% and 78.70%, respectively. This shows how much material will evaporate when the fuel is heated under controlled conditions.

It will inevitably affect the overall efficiency of the gasification process because elevated moisture and volatile matter content would require some of the energy produced during gasification to evaporate water and heat [7]. On the other hand, low moisture content and volatile matter content in biomass allow better use of available energy for gasification, minimising energy loss due to water evaporation [8]. This indicates that thermal pretreatment is essential for reducing the high volatile matter content in untreated SCG and improving the efficiency of the gasification process. As for the ash content, the untreated SCG contains 0.24% and 17.72% of fixed carbon.

Moreover, a high moisture content in biomass will compensate for the syngas, thereby lowering the syngas' energy potential. Meanwhile, the low moisture content leads to a higher concentration of combustible gases in the syngas, improving its quality and potential applications, such as electricity generation or biofuel production [9]. As a result, untreated SCG with high moisture content must undergo thermal pretreatment to remove excess moisture before gasification.

Table 1 Proximate analysis of untreated SCG

Analysis	Untreated SCG
Proximate Analysis	(wt %)
Moisture Content	8.35
Volatile Matter Content	78.70
Ash Content	0.24
Fixed Carbon Content	12.71

3.1.2 Torrefied SCG

Fig. 3 illustrates the effects of torrefaction on the proximate components: moisture content, volatile matter, ash content, and fixed carbon of both untreated and torrefied SCG. Torrefaction is a thermal process carried out in an inert atmosphere, typically at 200-300 °C, that decomposes biomass components [10].

Upon torrefaction, the moisture content decreased significantly, reaching 0.74 wt.% at 250°C. An insignificant decrease to 0.71 wt.% was observed at 300 °C. This trend suggests that torrefaction effectively removes moisture, enhancing the material's storability and reactivity during gasification.

The volatile matter content decreased from 77.43 wt.% at 200°C to 76.32 wt.% at 250°C, likely due to the release of volatile substances. A slight decrease to 75.57 wt.% was observed at 300°C, indicating partial decomposition of volatiles and chemical changes in the biomass matrix. The ash content increased from 1.82 wt.% at 200°C to 1.84 wt.% at 250°C, then increased to 1.98 wt.% at 300°C, likely due to the concentration of inorganic residues after organic matter volatilisation [11].

The fixed carbon content slightly increased from 18.28 wt.% at 200°C to 21.10 wt.% at 250°C, then increased slightly to 21.74 wt.% at 300°C. This rise at 300°C may indicate enhanced decomposition of carbonaceous compounds. Thus, 250°C for 60 minutes was determined to be the optimal torrefaction condition, yielding a high moisture content (0.74 wt.%), high fixed carbon content (21.1 wt.%), and low volatile matter content (76.32 wt.%).

3.2 FTIR Analysis of Untreated SCG and Torrefied SCG

The chemical structure of both untreated and pretreated SCG biomass samples was evaluated using FTIR analysis to assess the impact of torrefaction conditions. The FTIR spectra of torrefied SCG in the mid-infrared region (4000-500 cm⁻¹) are presented in Fig. 4. The FTIR spectra of untreated SCG and torrefied SCG samples heated up to 250 °C are almost identical in shape due to the absence of significant changes in their chemical structure. However, at 300 °C for 60 minutes (severe treatment conditions), the changes in vibration intensity in the FTIR spectra are more pronounced than those observed at 200 °C and 250 °C. Various peaks were detected at different

wavenumbers between 3200-3400, 2980-2850, 1730-1750, 1649, 1470-1350, 1243, and 998-850 cm^{-1} , as indicated in Table 2.

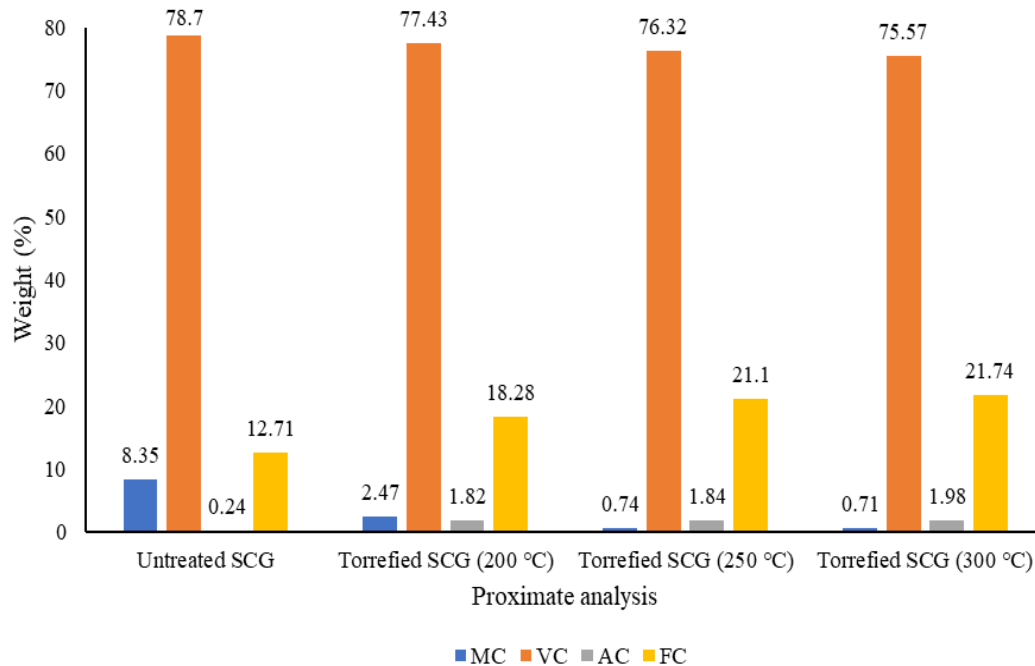


Fig. 3 Proximate analysis comparison between untreated SCG and pretreated SCG

Table 2 Summary of the functional group identified in untreated SCG [12]

Frequency (cm^{-1})	Functional Group
3200-3400	Hydroxyl compound
2980-2850	C-H stretch of an alkane molecule
1730-1750	Carbonyl groups
1649	C=C stretching bond of an alkene molecule
1470-1350	CH_2 and CH_3 deformation
1243	C-O stretch of alcohols and phenols
998-850	Aromatic phosphates

The peak at around 3200–3400 cm^{-1} in IR spectroscopy indicates a hydroxyl (O–H) stretching vibration, primarily associated with the lignocellulosic component (cellulose). As the torrefaction temperature increases, this peak diminishes and eventually disappears due to partial dehydration and carbohydrate decomposition under more severe torrefaction conditions. The 2980–2850 cm^{-1} peaks are attributed to the vibrations (stretching) of asymmetric and symmetric aliphatic groups (C–H), narrowing and disappearing in the torrefied samples. The band at around 1730–1750 cm^{-1} in both untreated SCG and torrefied SCG samples could be attributed to the carbonyl stretching (C=O) of aldehyde, ketone, ester, or carboxylic acid groups in hemicellulose.

The peak at 1649 cm^{-1} corresponds to unsaturated alkene groups, such as vinyl, vinylidene, or cis (C=C), while the peak at 1470–1350 cm^{-1} represents the alkane group, specifically (CH_2 , CH_3) deformation. At lower wavenumbers, which are below 1300 cm^{-1} , the alcohol and phenols group (C–O) at 1243 cm^{-1} is removed by the torrefaction process, whereas aromatic phosphates (P–O–C), which are generated between 995–850 cm^{-1} , are formed in return. Due to its low crystallinity and polymerisation, hemicellulose is the most reactive biopolymer in biomass. Consequently, hemicellulose undergoes significant decomposition during torrefaction, as indicated by the less intensified peaks at 300 °C for the respective torrefied samples.

Based on the spectrum provided, raising the temperature to mild levels (200 and 250 °C) retains the peaks. However, under the severe torrefaction condition (300 °C), significant changes in the spectra of the tested samples are observed. These changes are attributed to the release of oxygenated species, primarily from the breakdown of hemicellulose and limited disintegration of cellulose. Torrefaction reduces the hydroxyl (O–H) groups in SCG, thereby lowering its moisture content. This is beneficial because it requires less energy to evaporate water during gasification, making the process more efficient.

Additionally, changes in aliphatic hydrocarbons (C-H) indicate a decrease in volatile matter and an increase in fixed carbon, thereby improving the energy density of the feedstock. The formation and alteration of carbonyl groups (C=O) during torrefaction suggest the creation of new compounds, potentially boosting the yield of valuable syngas components such as hydrogen and carbon monoxide. The presence of C-N groups indicates the formation of nitrogen-containing compounds, which is crucial for managing nitrogen content in syngas, particularly for applications such as ammonia synthesis or the reduction of nitrogen oxides (NO_x) emissions.

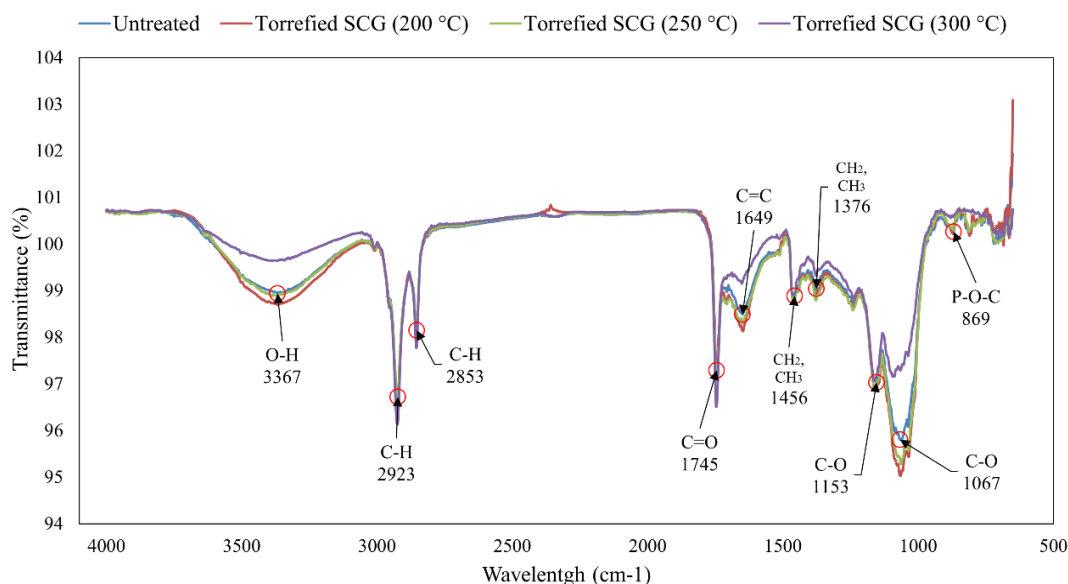


Fig. 4 FTIR spectra comparison between untreated SCG and pretreated SCG

3.3 Effect of Gasification Temperature on Product Yield

Fig. 5 illustrates the yields of bio-char, tar, and bio-gas from biomass treated at various temperatures during gasification. The bio-char yield shows a clear inverse correlation with temperature. At 700 °C, the bio-char yield is 27.75%, gradually decreasing to 19.31% at 850 °C, indicating the complex thermal decomposition mechanisms at play during gasification. At 700 °C, a significant portion of the biomass undergoes pyrolysis, forming biochar as a solid carbonaceous residue. As the temperature increases, the thermal breakdown of biomass intensifies, promoting more extensive gasification reactions that convert organic matter into gaseous products like syngas and tar [13]. This shift towards higher-temperature gasification results in a lower biochar yield, as more biomass is converted into gaseous components rather than solid char.

The tar yield exhibits greater temperature variability, whereas the biochar yield decreases with increasing temperature. For instance, at 750 °C, the tar yield peaks at 24.54%, indicating the highest tar production. This implies that specific temperatures can promote the formation of tar compounds during gasification processes. However, at 850 °C, the tar yield sharply declines to 10.31%, indicating a significant reduction in the formation of liquid by-products at higher temperatures. The rapid decrease in tar production at higher temperatures indicates a shift in the gasification reactions, favouring the formation of gaseous products over liquid tar compounds [14]. The variation in tar production may be due to the intricate nature of gasification reactions, which are influenced by factors like temperature, residence time, and biomass composition.

The biogas yield shows a consistent upward trend as temperature increases. Specifically, the data show that at 700°C, the biogas yield is 51.4%. However, as the temperature increases, the biogas yield steadily rises, reaching 70.38% at 850°C. It has been observed that higher temperatures increase gasification and decomposition of biomass, resulting in greater production of gaseous products such as methane, carbon dioxide, and hydrogen. This increase in biogas yield at elevated temperatures emphasises the importance of temperature control in optimising gas production during biomass gasification. By carefully managing the operating temperature in the gasifier, it is possible to shift the product distribution towards higher biogas yields, benefiting applications such as energy generation or fuel production [15].

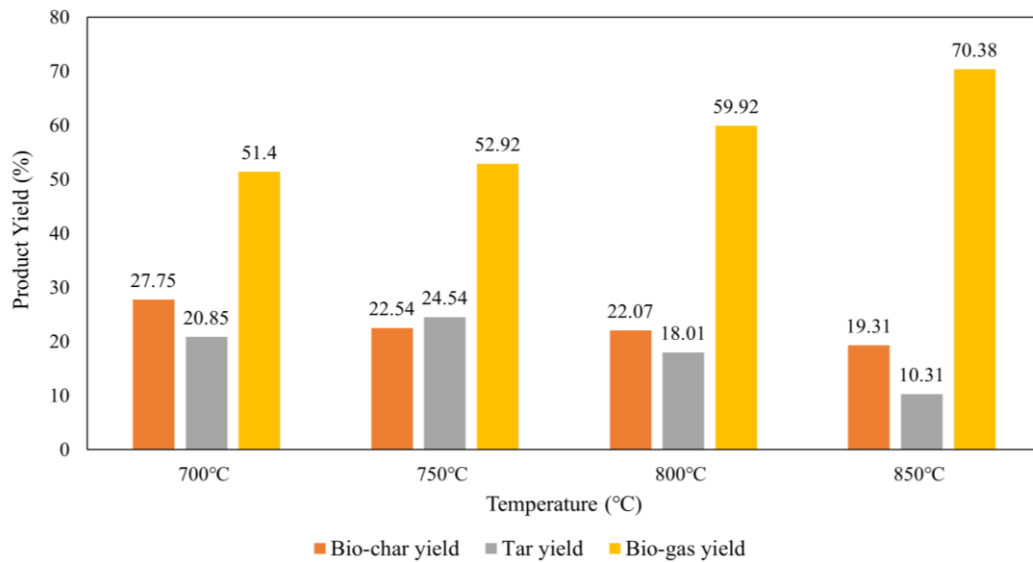


Fig. 5 Effect of temperature on the product yield of pretreated SCG

3.4 Effect of Gasification Time on Product Yield

Fig. 6 shows the yields of biochar, tar, and biogas obtained over different time intervals during gasification. Time significantly influences the distribution of these products, as evidenced by variations in yields across different intervals. There was a slight decrease in bio-char yield from 13.13% at 45 minutes to 12.84% at 50 minutes, followed by an increase at 55 minutes and a sharp decline to 7.47% at 60 minutes. Variations in the gasification process indicate fluctuations in the extent of carbon conversion and char formation. Factors such as temperature variations and residence time can significantly influence these fluctuations, ultimately affecting the efficiency of biochar production and the overall gasification process [6].

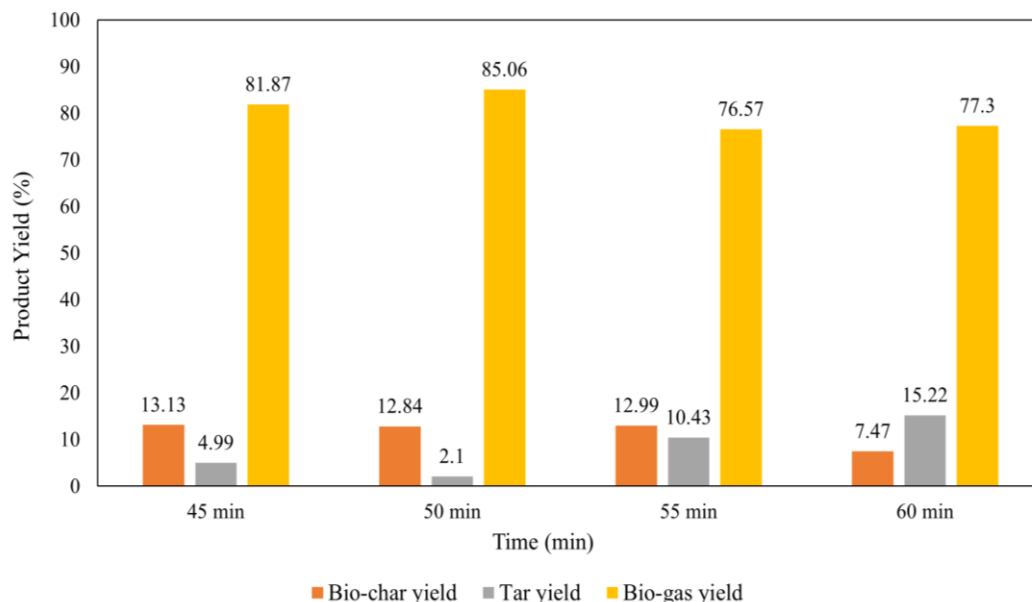


Fig. 6 Effect of time on the product yield of pretreated SCG

The amount of tar produced varies significantly over time. The tar yield drops from 4.99% at 45 minutes to 2.1% at 50 minutes and then sharply increases to 10.43% at 55 minutes and 15.22% at 60 minutes. These indicate dynamic changes in the composition and distribution of liquid by-products during gasification. Factors such as temperature profiles and reaction kinetics can play a crucial role in influencing these variations in tar yield over different time durations [6]. The interaction of these factors can affect the formation and evolution of tar compounds, thereby altering tar production.

The biogas yield shows a clear pattern over time, peaking at 85.06% at 50 minutes, then slightly decreasing to 76.57% at 55 minutes and 77.3% at 60 minutes. This trend indicates the significant impact of time on

gasification and biomass decomposition, resulting in varying yields of gaseous products such as methane, carbon dioxide, and hydrogen. The distinct peak and subsequent decreases in biogas yield highlight the time-dependent nature of gas production during gasification, underscoring the importance of timing control in optimising biogas yields and overall gasification efficiency.

Therefore, based on the findings, the optimum time for gasifying SCG is 50 minutes, as it yields the highest gas yield and the lowest tar yield. This duration enables efficient gasification and decomposition of the biomass, yielding a higher proportion of gaseous products, including methane, carbon dioxide, and hydrogen. Maximising biogas production is a primary objective of gasification processes for energy generation and biofuel production.

3.5 FTIR Analysis of Biochar from Optimum Gasification

Fig. 7 shows the FTIR results for the biochar under optimum gasification parameters. Table 3 lists different peaks at wavenumbers 2921, 2854, 1749, 1576, 1091, and 876 cm^{-1} . The peak at 2921 cm^{-1} corresponds to the asymmetric stretching vibrations of alkane groups (C-H). The peak at 2854 cm^{-1} also represents the symmetric stretching vibrations of C-H bonds, further suggesting the presence of hydrocarbon chains. A strong peak at 1749-1576 cm^{-1} indicates carbonyl groups (C=O).

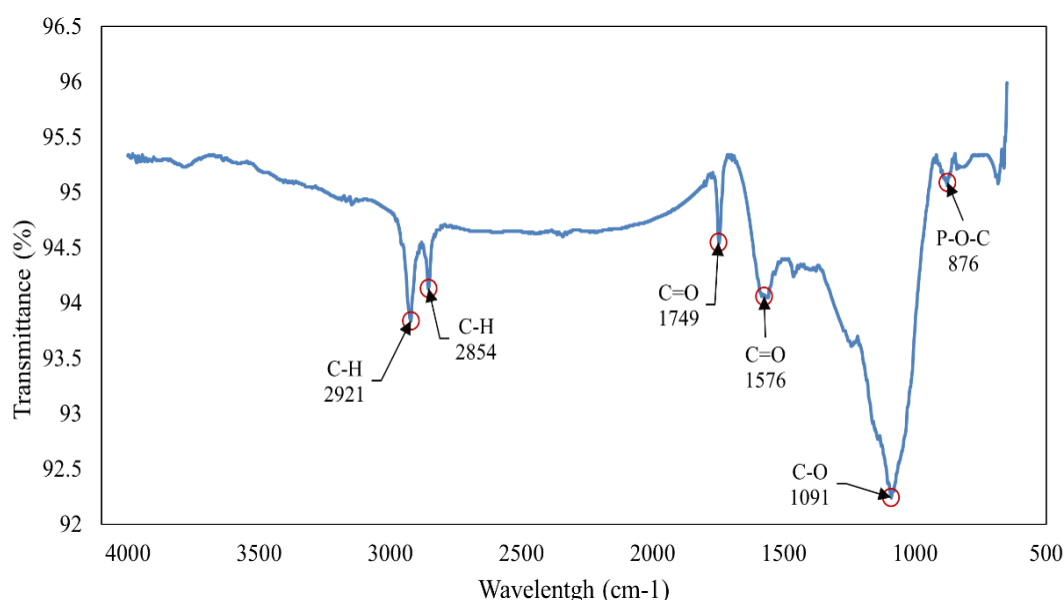


Fig. 7 FTIR spectra of bio-char at optimum gasification parameters

Table 3 Summary of the functional group of bio-char identified in optimum gasification parameters [12]

Frequency (cm^{-1})	Functional Group
2921 and 2854	C-H stretch of an alkane molecule
1749 and 1576	Carbonyl groups, C=O
1091	C-O stretch of alcohols and phenols
876	Aromatic phosphates

Carbonyl groups are characteristic of various functional groups, including aldehydes, ketones, esters, and carboxylic acids. This peak suggests the sample contains one or more of these groups, providing insight into its chemical structure. The peak at 1091 cm^{-1} corresponds to the C-O stretching vibrations, typically observed in alcohols, ethers, carboxylic acids, and esters. This indicates that the sample may contain one or more oxygenated functional groups. Lastly, at peak 876 cm^{-1} , aromatic phosphates (P-O-C) are generated.

By identifying the functional groups present in the optimised char, the feedstock's quality can be better understood. This information allows for the selection of the most suitable biomass types and treatment conditions for efficient gasification. Knowledge of specific functional groups helps predict how the char will behave before further use. For instance, oxygenated functional groups (C=O) can enhance reactivity, while hydrocarbons (C-H) contribute to the energy content. The presence of P-O-C bonds suggests potential catalytic activity within the char. This could improve the gasification process by increasing reaction rates and enhancing the overall efficiency of syngas production [16]. These functional groups confirm that the bio-char contains both oxygenated and hydrocarbon compounds, contributing to its structural integrity and potential as a high-energy solid fuel.

3.6 FTIR Analysis of Tar from Optimum Gasification

Fig. 8 shows the FTIR results for the tar at the optimum gasification parameters, and Table 3 summarises the functional groups. The peaks at 2921 cm⁻¹ and 2851 cm⁻¹ correspond to symmetric C-H stretching vibrations characteristic of alkanes, suggesting the presence of hydrocarbon chains in the sample. Furthermore, the strong peak at 1705 cm⁻¹ indicates the presence of carbonyl groups (C=O), characteristic of compounds like aldehydes, ketones, esters, and carboxylic acids. This suggests that the sample contains one or more of these functional groups. Similarly, the peak at 1463 cm⁻¹ signifies the asymmetric stretching vibrations of methyl (CH₃) and methylene (CH₂) groups, commonly found in aliphatic hydrocarbons, indicating their presence. The peak at 1273 cm⁻¹ suggests the presence of C-O bonds, indicating the presence of alcohols and phenols in the sample. Finally, the peak at 746 cm⁻¹ corresponds to the bending vibrations of C-Cl bonds, which are prevalent in various organic compounds.

The spectrum provides clear evidence of diverse functional groups in the sample, aiding in identifying the chemical structure of the compound or mixture under analysis. The presence of these functional groups indicates that the sample may be an organic compound containing aliphatic hydrocarbons, carbonyl groups, ether or ester linkages, and potentially chlorinated compounds.

Moreover, understanding the functional groups present can help in predicting the chemical behaviour, reactivity, and potential applications of the material [17]. For example, the presence of carbonyl and C-O groups may hint at reactivity in condensation reactions or susceptibility to hydrolysis. Aliphatic groups, based on the functional group, consist of straight carbon chains (C-H), making them ideal for fuel production [18], [19]. The generated tar shows promise as a biofuel substitute, but further refinement is needed for commercial use. Therefore, identifying and understanding the functional groups can enhance process efficiency and provide better characterisation of feedstock materials.

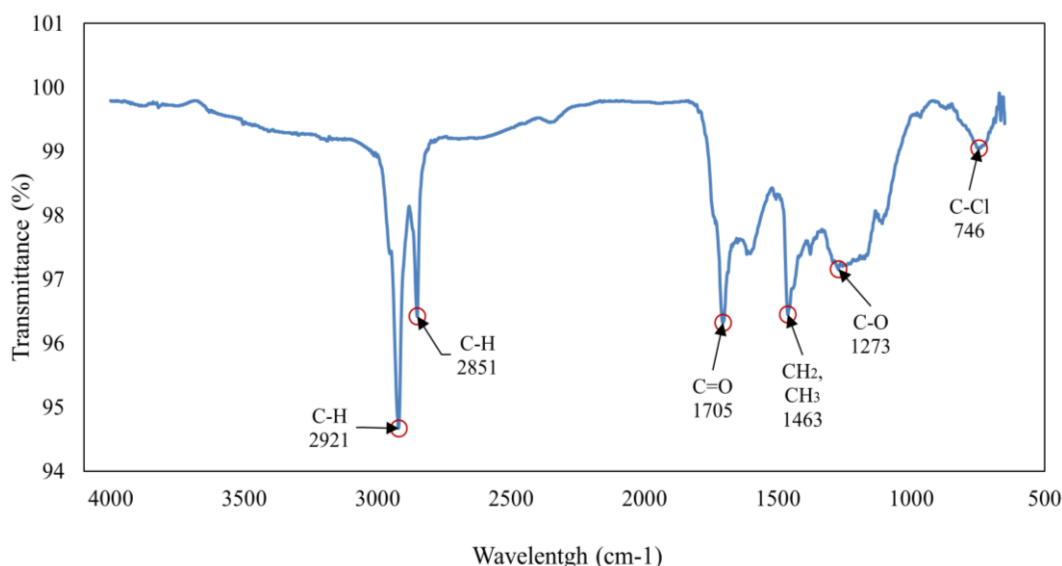


Fig. 8 FTIR spectra of tar at optimum gasification parameters

Table 3 Summary of the functional group of tar identified in optimum gasification parameters [12]

Frequency (cm ⁻¹)	Functional Group
2921 and 2851	C-H stretch of an alkane molecule
1705	Carbonyl groups, C=O
1463	CH ₂ and CH ₃ deformation
1273	C-O stretch of alcohols and phenols
746	Alkyl halides

3.7 Conclusion

This study successfully investigated the potential of torrefied SCG as a gasification feedstock. The pretreatment process, particularly torrefaction, significantly enhanced the physical and chemical properties of the biomass, making it more suitable for thermal conversion. Gasification experiments conducted in a fixed-bed reactor identified optimal conditions at 850 °C and 50 minutes, with a biogas yield of 70.38%. These conditions facilitated

efficient carbon conversion and decomposition, leading to increased production of valuable gases such as methane, carbon dioxide, and hydrogen; key components for renewable energy and biofuel applications. The tar obtained showed promise as a biofuel alternative due to its hydrocarbon-rich composition. Although further purification is necessary for practical and commercial applications. Overall, the results demonstrate that torrefied SCG is a viable and sustainable feedstock for thermochemical conversion via gasification into clean energy products.

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Conflict of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

Author Contribution

*The authors confirm contribution to the paper as follows: **Study conception and design:** Razi Ahmad, Anis Syahira Abdul Hamid, Norhafezah Kasmuri; **Data collection:** Anis Syahira Abdul Hamid; **Analysis and interpretation of results:** Razi Ahmad, Anis Syahira Abdul Hamid, Sharifah Nursyimi Azlina Syed Ismail, Jareerat Sakultrat; **Draft manuscript preparation:** Razi Ahmad, Anis Syahira Abdul Hamid. All authors reviewed the results and approved the final version of the manuscript.*

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