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Research article

Investigation of process parameters in the pyrolysis of sheep manure using a two-level factorial

Illa Rizianiza[1,2,*], Ahmad Indra Siswantara[1] and Eny Kusrini[3,4,5]

[1] Department of Mechanical Engineering, Universitas Indonesia, Depok 16424, Indonesia
[2] Department of Mechanical Engineering, Institut Teknologi Kalimantan, Balikpapan 76127, Indonesia
[3] Department of Chemical Engineering, Universitas Indonesia, Depok 16424, Indonesia
[4] Green Product and Fine Chemical Engineering Research Group, Universitas Indonesia, Depok 16424, Indonesia
[5] Tropical Renewable Energy Research Center, Universitas Indonesia, Depok 16424, Indonesia

* **Correspondence:** Email: illa.rizianiza@ui.ac.id; Tel: +628112703944.

Abstract: This study analyzed the effect of a slow pyrolysis process at a temperature range of 270–360 °C using a two-level factorial design. This pyrolysis process is limited to the process that occurs in the pyrolysis reactor, namely the decomposition process of raw materials into char and non-condensable gas. This study evaluated the effect of three process variables: fuel mass flow rate (0.006–0.008 kg/min), feedstock mass (2.5–4 kg), and residence time (120–180 min). The results showed that residence time had the most significant effect on pyrolysis products. Residence time contributes 30.1% to increasing char yield and correspondingly decreases non-condensable gas yield, indicating an inverse relationship. The feedstock mass and fuel mass flow rate show moderate effects, decreasing char yield by 16.1% and 6.2%, respectively, while inversely increasing non-condensable gas yield by the same magnitude. These findings underline the importance of residence time in determining char yield, especially in the slow pyrolysis process. This study also proves that the quantity of raw materials has an influence on char yield, although not as strong as the influence of residence time. The data revealed a clear trend, indicating how adjustments to the pyrolysis process can affect performance. With a longer residence time, it will produce a greater biochar than a non-condensable gas yield. Optimization of pyrolysis products must consider the balance between residence time, fuel heat rate, and mass of raw material load in order to obtain the optimal product as desired.

Keywords: biochar; pyrolysis; sheep manure; two-level factorial design

1. Introduction

As the global population grows, fossil fuel reserves are increasingly depleting due to excessive exploitation. The combustion of fossil fuels makes a large contribution to greenhouse gas emissions, such as carbon dioxide. This is one of the causes of global climate change [1], releasing harmful pollutants that degrade the environment and the quality of the air [2]. Energy demand continues to increase significantly, in line with population growth and improvements in human living standards [3]. To address this growing demand in the face of diminishing fossil fuel supplies, the transition to renewable energy sources is essential. Renewable energy plays a central role in sustainable energy systems. Its utilization can minimize adverse environmental impacts, reduce secondary waste generation, and support the goals of socially and economically sustainable development. According to a study by Holechek et al., achieving a complete transition from fossil fuels to renewable energy by 2050 requires the aggressive implementation of all eight recommended pathways—one of which is renewable energy development—alongside significant lifestyle changes in developed nations and strong international collaboration [4]. Furthermore, research by Yuping et al. demonstrated that increased use of renewable energy can mitigate climate risks, with findings validated through robust testing across various methodological adjustments, including changes in variables, sample sets, and analytical techniques [5].

A biomass type that can be used for renewable energy is animal manure. Animal manure is a significant contributor to greenhouse gas emissions, accounting for nearly 10% of total emissions, including CO_2, CH_4, and N_2O [6]. In Indonesia, several types of livestock are commonly raised, including cows, sheep, goats, and horses [7]. Sheep are often kept both on farms and in households. The largest population of sheep in Indonesia is in the West Java region, which accounts for 68.41% of the national sheep population [7]. Each sheep, weighing 20–40 kg, produces approximately 0.32–0.625 kg of manure daily, equivalent to 0.3 tons annually [8,9]. The potential of biomass renewable energy sources continues to increase along with the increase in livestock population [10]. Notably, sheep manure contains a higher fixed carbon content (13%) compared to other types of animal manure, indicating a higher calorific value and greater potential for energy conversion [11].

Some methods used in biomass energy conversion are combustion, gasification, and pyrolysis. In the combustion process, fuel can be completely oxidized and converted into heat. However, the efficiency of the combustion process is only 10%, and this process can cause environmental pollution [8]. In contrast, gasification involves the partial oxidation of biomass to produce fuel in the form of gas. Pyrolysis, on the other hand, is a thermal decomposition process that occurs in the absence or near-absence of oxygen [12]. A key advantage of pyrolysis is its flexibility, allowing the process to be tailored to produce specific desired products [13]. For example, slow pyrolysis is preferred to maximize biochar yields, whereas fast pyrolysis is used to increase bio-oil production [14]. The application of pyrolysis to animal waste is a promising solution to reduce greenhouse gas emissions and mitigate negative environmental impacts. In addition to its environmental benefits, biochar produced from animal manure exhibits versatile functionalities. Due to its porous structure and rich elemental composition, biochar can serve not only as a soil amendment to enhance fertility but also as an adsorbent, a medium for carbon sequestration, a catalyst, and even as material for energy storage, such as supercapacitors [15].

Moreover, biochar derived from the pyrolysis of animal manure possesses favorable fuel properties, making it a viable alternative energy source with significant utilization potential [16].

Currently, numerous studies have focused on the use of animal manure as a feedstock for both pyrolysis and co-pyrolysis processes [8,17–20]. These studies primarily focus on the quality of biochar under various operational conditions. Several factors that can influence the yield of pyrolysis products are temperature, heating rate, residence time, particle size, and reactor type [21]. These operational parameters play a crucial role in determining the overall performance of the pyrolysis process. However, pyrolysis technology has significant challenges due to the complexities of heat transfer and the heterogeneity of feedstocks, leading to inconsistent heating, which affects both the yield and quality of pyrolysis products [22].

Many studies have investigated the effect of feedstock type, chemical composition, and particle size on biochar yield in the pyrolysis process [23,24]. These studies have predominantly concentrated on optimizing thermal parameters such as temperature, heating rate, and residence time to enhance both the quantity and quality of biochar. However, the influence of feedstock quantity—defined as the total mass of biomass loaded into the reactor—on biochar yield has not been systematically addressed in the literature. This parameter is critically important, as increasing the amount of feedstock can significantly alter the heat transfer dynamics within the reactor [25]. In batch or fixed-bed pyrolysis systems, an excessive amount of feedstock can result in non-uniform heat distribution, where certain regions within the biomass bed may not attain the desired pyrolysis temperature. This condition can inhibit the carbonization process and generate biochar with inconsistent physical and chemical characteristics. In some cases, higher biochar yields may be observed due to unconverted or partially decomposed material, although such yields may not reflect optimal conversion efficiency or product quality [26]. Therefore, feedstock quantity not only affects the thermal regime but also plays a direct role in determining the actual biochar yield.

The study by Chen et al. [27] examined the optimization of pyrolysis conditions for herbal tea and *Salvia miltiorrhiza* residues. Their work emphasized the effects of temperature, heating rate, and pyrolysis strategies, employing artificial neural networks and response surface methodology to predict product yields. However, despite the comprehensive treatment of process variables, the study did not examine the role of feedstock quantity in influencing biochar production, thereby highlighting an important and underexplored aspect in pyrolysis research. In light of this gap, the present study seeks to systematically evaluate the effect of feedstock quantity on biochar yield under controlled pyrolysis conditions. By isolating this operational parameter and assessing its impact, the research aims to contribute new empirical evidence and practical insights that are critical for optimizing biochar production, particularly in the context of reactor scale-up and biomass throughput management.

Classically, research on the effect of operational conditions on pyrolysis was carried out using the one-variable-at-a-time (OVAT) approach. This method varies a single parameter while keeping others constant, thereby allowing only isolated effects to be observed. However, a key limitation of the OVAT approach is its inability to capture interaction effects between variables. In addition, this method does not account for how changes in one parameter may influence the system response when combined with variations in other parameters. As a result, the derived optimum for one variable may not reflect the true global optimum within the multivariable system [28]. To address these limitations, the present study adopts a design of experiment (DOE) approach, which systematically varies multiple process parameters across different levels to evaluate their individual and interactive effects on response variables [28,29]. DOE enables more comprehensive optimization and provides deeper insight into process behavior, offering recommendations for setting parameter combinations that yield optimal outputs. Specifically, a two-level factorial design was selected in this study to efficiently investigate the influence of critical operational parameters in the pyrolysis of sheep manure. Previous studies, such as that conducted by Al-Mrayat et al., have applied factorial designs to analyze the effects of residence time and heating rate on product yields during microwave pyrolysis of fecal sludge [28].

Few studies have systematically investigated feedstock quantity as a primary variable affecting biochar yield under controlled pyrolysis conditions. This parameter plays a critical role in heat transfer dynamics, particularly in fixed-bed or batch systems, and can significantly affect thermal decomposition behavior, especially during scale-up and high-throughput operations. Addressing this research gap, the present study focuses on the slow pyrolysis of sheep manure, with the aim of maximizing biochar yield through a factorial experimental design. By simultaneously evaluating feedstock mass, residence time, and fuel mass flow rate, this work not only isolates the effects of each parameter but also explores their interactions, offering empirical evidence and practical insights for optimizing biochar production in decentralized and low-cost biomass utilization systems. While recent studies have successfully implemented advanced infrared-assisted pyrolysis systems—particularly for synthetic or fossil-based feedstocks such as coal and PVC [30,31]—these approaches often demand high energy input and precise control [32], limiting their applicability in rural or small-scale contexts. In contrast, the current study employs waste heat from engine exhaust to drive slow pyrolysis, making it a more feasible, energy-efficient, and environmentally friendly alternative for agricultural waste valorization.

2. Materials and methods

2.1. Feedstock characterization

The physicochemical properties of biomass feedstock significantly influence the pyrolysis process and product distribution [33]. Ultimate and proximate analyses were conducted to determine key parameters, including moisture content, volatile matter, fixed carbon, ash, and elemental composition (C, H, N, O, S). A high ash content is a typical characteristic of animal manure, and biochar is the primary product obtained from its pyrolysis. Low temperature, slow heating rate, and long residence time play important roles in enhancing biochar yield during pyrolysis [34]. To evaluate the feedstock's quality—particularly regarding undesirable compounds, combustion stability, and thermal behavior—a comprehensive analysis is required. Table 1 presents a comparison of ultimate and proximate analysis results for different types of animal manure. Sheep manure exhibits a higher fixed carbon content (12.79%) compared to goat and cow manure, indicating a higher heating value (HHV) [35]. Table 2 shows variation in ultimate and proximate properties of sheep manure on both an as-received and dry basis, with fixed carbon content ranging from 12.79% to 17.1% across multiple studies.

The lignocellulosic composition of animal manure significantly influences the characteristics of pyrolysis products [36]. In particular, the cellulose and lignin content in biomass are critical parameters for assessing pyrolysis behavior. Biomass with high lignin content typically undergoes slower pyrolysis, whereas high cellulose content leads to a faster decomposition rate [37]. Compared to plant-based biomass, animal manure generally contains lower levels of lignin. For instance, cattle manure consists of approximately 60 wt% cellulose, 15 wt% hemicellulose, and 9 wt% lignin [22,38]. Animal manure contains cellulose, hemicellulose, lignin, protein, lipid, and fat, as well as several other nutritional contents [39]. Among different animal manures, sheep manure exhibits lower cellulose content than cow and goat manure but has the highest crude protein content [40]. During the pyrolysis process, these compounds will decompose at different temperatures [41]. Hemicellulose and cellulose will decompose at low temperatures. Lignin decomposition occurs in a wide temperature range, from 100 to 900 °C. This shows that lignin is more resistant to heat and decomposes gradually during pyrolysis at various temperatures. Meanwhile, hemicellulose decomposes in the range of 220–315 °C, and cellulose will decompose into simpler compounds at temperatures of 315–400 °C [39]. In terms of energy potential, sheep manure also has a higher HHV (16.02) than other animal manure, such as poultry (13.66) and pig manure (14.87) [42].

Table 1. Comparison of ultimate and proximate parameters in animal manure [35].

Parameter	Sheep manure	Goat manure	Cattle manure
Proximate (dry basis)			
Moisture (%)	8.00	7.57	7.90
Volatile matter (%)	59.98	63.50	52.67
Fixed carbon (%)	12.79	10.50	9.32
Ash (%)	19.23	18.43	30.11
Ultimate (dry basis)			
Carbon (%)	51.33	38.74	40.77
Hydrogen (%)	6.45	5.65	5.14
Nitrogen (%)	2.65	2.11	2.79
Oxygen (%)	38.81	53.49	50.07
Sulphur (%)	0.76	0.01	0.61
Chlorine (%)	0.00	0.00	0.61

Table 2. Comparison of sheep manure properties.

Properties	As-received basis [43]	As-received basis [35]	Dry basis [44]	Dry basis [45]
Ultimate				
C (%)	49.0 ± 0.5	51.33	34.33	33.97
H (%)	6.3 ± 0.1	6.45	4.96	4.27
N (%)	3.3 ± 0.0	2.65	2.36	3.37
O (%)	41.3	38.81	41.96	25.48
S (%)	-	0.76	0.31	0.58
Cl (%)	-	0.61	-	-
Proximate				
Moisture (%)	8.3 ± 0.5	8	-	-
Volatile matter (%)	58.6 ± 0.7	59.98	68.61	52.85
Fix carbon (%)	16.8 ± 0.3	12.79	15.31	14.83
Ash (%)	24.6 ± 0.6	19.23	16.08	32.32
HHV	21.1 MJ/kg	-	-	12.28 MJ/kg
LHV	-	-	14.72 MJ/kg	11.71 MJ/kg

2.2. Experimental setup

The pyrolysis experiments were conducted using a fixed-bed batch reactor constructed from refractory materials to withstand high temperatures. Fixed-bed reactors are widely employed due to their simplicity, wide temperature distribution, ability to melt ash, and capacity to produce clean gas with low tar content and high carbon efficiency [46]. To enhance thermal insulation and maintain temperature stability, the reactor was wrapped with a 2.5 cm thick ceramic fiber, which provides resistance up to 1260 °C and minimizes heat loss to the surroundings. Prior to pyrolysis, the sheep manure feedstock was oven-dried at 105 °C for 24 h to reduce moisture content and improve conversion efficiency. The dried feedstock, with a particle size range of 0.005–0.007 m, was then loaded into the reactor through a top loading port prior to the start of each experiment.

The system was operated under oxygen-limited (anaerobic) conditions to ensure true pyrolytic decomposition. The heating process was initiated using exhaust gas from an internal combustion engine as the primary heat source, supplemented by an auxiliary furnace to maintain the desired temperature profile when needed. The pyrolysis reactor was indirectly heated by the exhaust gas from a genset running on LPG fuel. This approach was motivated by the low thermal efficiency of conventional internal combustion engines. According to previous studies, spark-ignition engines typically operate with a brake thermal efficiency of about 30%–36%, while diesel engines reach up to 40%–47% [47]. This indicates that a substantial portion of input energy is lost as exhaust heat, which may be feasibly redirected for thermal applications such as biomass pyrolysis.

The reactor temperature was increased from ambient temperature to the target pyrolysis temperature at an average heating rate of 2 °C/min. Once the target was reached, the temperature was held constant (isothermal conditions) for either 120 or 180 min, depending on the experimental run. Temperature data were recorded continuously using a data acquisition system. Based on prior thermogravimetric analysis (TGA) of sheep manure [45], the primary devolatilization occurred between 250 and 450 °C, with the most intense thermal degradation near 450 °C. Therefore, pyrolysis was conducted within this temperature range to ensure optimal conversion.

Upon completion of the isothermal period, the reactor was allowed to cool naturally. The solid residue (biochar) was collected and weighed to determine the char yield. The pyrolysis gas was directed upward to a multi-stage condenser, allowing the condensation of bio-oil while non-condensable gases were separated and released. A schematic of the pyrolysis system, including flow pathways and heating components, is shown in Figure 1.

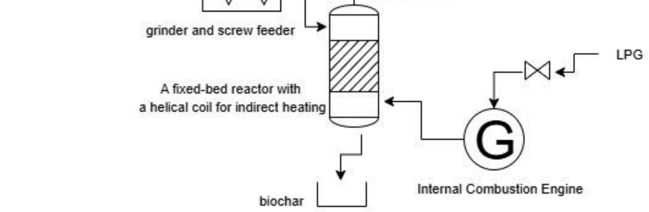

Figure 1. Process flow diagram of the pyrolysis system.

2.3. Experimental design

In the slow pyrolysis of sheep manure, a multivariate experimental design was employed to evaluate the effects of key process variables. Previous studies on biomass pyrolysis, such as rice straw, have demonstrated that residence time and temperature significantly affect product yield, where higher temperatures and longer residence times often lead to lower biochar yield due to enhanced devolatilization and secondary reactions [48]. Sheep manure was selected as the feedstock in this study due to its local abundance, high fixed carbon content, and favorable thermal decomposition characteristics. A full factorial design (2^3) with two replications was adopted to investigate the effects of three independent variables: fuel mass flow rate (0.006–0.008 kg/min), feedstock mass (2.5–4 kg), and residence time (120–180 min). The specific levels of each parameter are summarized in Table 3.

The temperature range was determined based on TGA, which identified a critical devolatilization window of 250–450 °C for sheep manure, with a maximum degradation peak near 350–400 °C (Figures 1 and 2, peaks c and d) C [45]. The subrange of 270–360 °C was selected to target the low-to-medium temperature regime of slow pyrolysis, which favors biochar formation by limiting secondary cracking and gasification reactions that predominate at higher temperatures (>400 °C) [49,50]. This range ensures sufficient thermal decomposition of hemicellulose (220–315 °C) and cellulose (315–400 °C), while preserving fixed carbon content (13% in sheep manure) and minimizing excessive volatilization. Meanwhile, the feedstock mass range was determined based on the thermal capacity and geometry of the fixed-bed reactor, which govern heat penetration and retention within the bed. A range of 2.5–4.0 kg was selected to ensure an adequate reaction scale while minimizing thermal gradients that could compromise product uniformity. Feedstock masses below 2.5 kg were found to produce insufficient char due to limited thermal interaction, whereas masses exceeding 4.0 kg led to uneven heating. In a fixed-bed reactor, the feedstock itself acts as the thermal medium, absorbing and redistributing heat across the bed. Therefore, selecting an appropriate feedstock mass is critical to ensure effective heat transfer and uniform pyrolysis throughout the reactor volume [51].

The residence time was varied between 120 and 180 min to explore its influence on carbonization. This range was selected based on both theoretical foundations and preliminary experimental observations. Slow and intermediate pyrolysis processes, with residence times ranging from several minutes to hours or even days, are generally considered optimal for maximizing biochar production, typically yielding 25%–35% of the feedstock mass. This relationship between residence time and biochar yield has been widely acknowledged in the literature, as summarized by [52], who cite [53] in their comprehensive review of pyrolysis mechanisms. Prolonged thermal exposure under oxygen-limited conditions promotes secondary reactions such as tar cracking, carbon condensation, and the stabilization of fixed carbon structures, which collectively enhance biochar formation and quality. In prior preliminary trials, residence times shorter than 120 min resulted in incomplete carbonization, with a noticeable reduction in char yield and the presence of volatile-rich residues. Conversely, extending the residence time beyond 180 min led to diminishing returns in yield, likely due to secondary degradation of already-formed char and increased energy consumption without significant performance gains. Thus, the selected range of 120–180 min represents an optimal window within the slow pyrolysis regime, balancing thermal input with carbon conversion efficiency. Furthermore, the fuel mass flow rate (0.006–0.008 kg/min) was adjusted to control the rate of heat input, maintaining an average heating rate of approximately 2 °C/min. This heating rate falls within the typical slow pyrolysis regime of 1.5–5 °C/min, as reported by Ouerhani et al. [54], and is consistent with previous reactor-scale studies that demonstrate optimal biochar formation under these conditions.

Sheep manure was chosen as the feedstock due to its abundant availability and high renewable energy potential. A full factorial design (2^3) with two replications was employed to assess the effects of three independent variables—fuel mass flow rate, feedstock mass, and residence time (see Table 3)—on two response variables: char yield and pyrolysis gas yield. The complete experimental matrix and results are detailed in Table 4. All experiments were conducted in triplicate under identical conditions to ensure reproducibility and statistical reliability; reported values are means of three independent runs, with standard deviations (SD) indicating experimental variability. Error bars representing SD are shown in Figure 2. Gas yield (%) was estimated by difference, calculated as 100% minus the experimentally determined biochar yield, based on the assumption that condensable liquids were minimal under the slow pyrolysis conditions applied.

Table 3. Levels of process parameters.

Parameters	Low level (−1)	High level (+1)
Feed rate of fuel	0.006 kg/min	0.008 kg/min
Mass of feed rate	2.5 kg	4 kg
Residence time	120 min	180 min

Table 4. Experimental runs and responses.

Std	Run	Factor 1 A: Mass flow rate of fuel (kg/min)	Factor 2 B: Mass of feedstock (kg)	Factor 3 C: Residence time (min)	Response 1 Char yield (%)	Response 2 Pyrolysis gas yield (%)
14	1	1	−1	1	61.57 ± 0.60	38.37 ± 0.55
4	2	1	1	−1	19.50 ± 0.50	80.50 ± 0.78
11	3	−1	1	−1	21.50 ± 0.50	78.50 ± 0.87
6	4	1	−1	1	64.00 ± 0.50	36.03 ± 0.84
5	5	−1	−1	1	72.03 ± 0.25	28.03 ± 0.15
8	6	1	1	1	37.53 ± 0.64	62.50 ± 0.50
9	7	−1	−1	−1	30.03 ± 1.79	70.00 ± 1.00
1	8	−1	−1	−1	32.00 ± 0.87	68.00 ± 0.44
15	9	−1	1	1	48.23 ± 0.42	51.80 ± 0.20
10	10	1	−1	−1	29.23 ± 0.68	70.80 ± 0.20
16	11	1	1	1	39.00 ± 0.92	61.03 ± 0.25
7	12	−1	1	1	50.50 ± 0.50	49.53 ± 0.50
3	13	−1	1	−1	23.03 ± 0.25	76.97 ± 0.93
12	14	1	1	−1	20.00 ± 0.87	80.00 ± 0.89
13	15	−1	−1	1	71.20 ± 0.80	28.83 ± 0.25
2	16	1	−1	−1	27.97 ± 1.00	72.00 ± 0.79

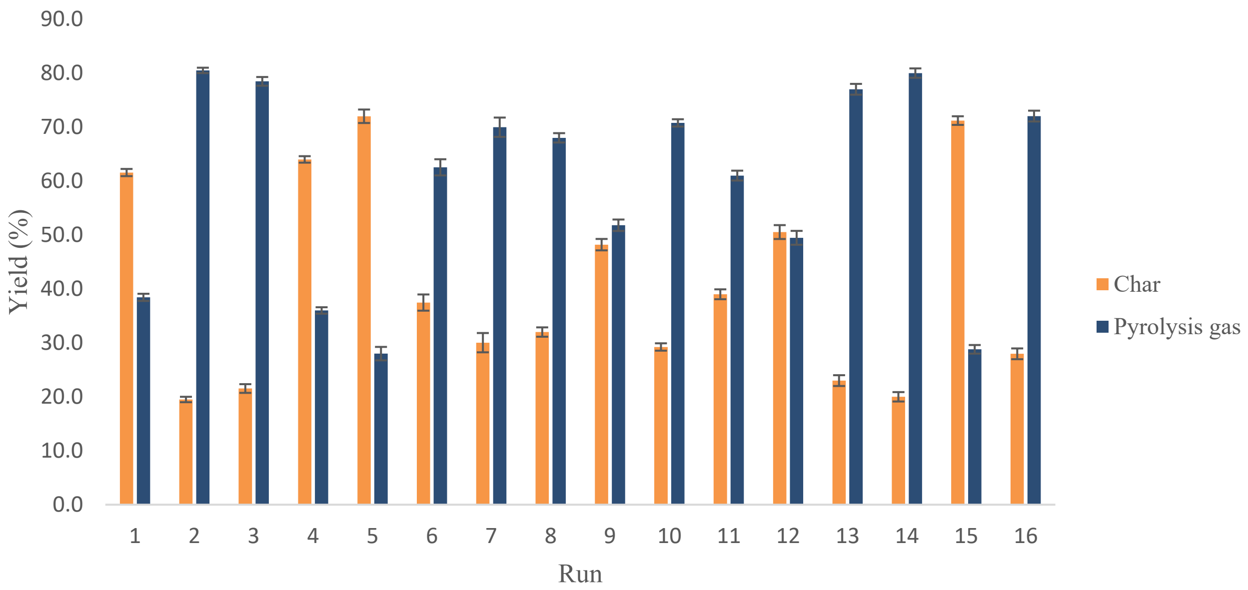

Figure 2. Char and pyrolysis gas yields with error bars for each experimental run.

2.4. Statistical analysis

The accuracy of the predictive model was evaluated using the mean absolute error (MAE) and coefficient of determination (R^2) [55]. MAE quantifies the average magnitude of the errors between predicted and observed values. Meanwhile, R^2 indicates the proportion of the variance in the observed data that is explained by the model. An R^2 value closer to 1 suggests better predictive capability and model fit. MAE and R^2 were calculated using Eqs 1 and 2, respectively [55]:

$$MAE = \frac{1}{n}\sum(|Y_i - \widehat{Y_i}|) \quad (1)$$

$$R^2 = 1 - \frac{\sum_{i=1}^{n}(Y_i - \widehat{Y_i})^2}{\sum_{i=1}^{n}(Y_i - \bar{Y})^2} \quad (2)$$

where $\widehat{Y_i}$ is the predicted yield, $\bar{Y}$ is the mean, and n is sample size.

3. Results and discussion

3.1. Effects of parameters on pyrolysis product yields

Evaluation of the effects of fuel mass flow rate, feedstock mass, and residence time has been analyzed effectively using a full factorial design. Figure 3 shows a main effects plot, illustrating the individual impact of each process parameter on char yield. By varying each process parameter, changes in the char yield value can be identified, allowing the identification of parameters with the most significant influence. Meanwhile, Figure 4 displays a main effects plot for pyrolysis gas yield. The magnitude of influence can be visually interpreted from the slope of the lines in each plot. Among the three process parameters investigated, residence time and feedstock mass exhibited a statistically significant influence on both char and pyrolysis gas yields. This observation is attributed to their critical roles in governing thermal exposure and reaction kinetics during the pyrolysis process. Extended residence time facilitates prolonged thermal interaction between the biomass and heat source, thereby enhancing the extent of devolatilization and enabling secondary reactions such as tar cracking and secondary char formation [56]. These conditions result in a higher conversion of volatiles into solid carbonaceous residues, thereby increasing the char yield while simultaneously reducing the yield of pyrolysis gas [57]. This trend aligns with previous findings, which demonstrate that longer residence times enhance carbonization and promote the formation of stable char structures [58,59]. Similarly, variations in feedstock mass directly affect the heat transfer dynamics within the reactor. An increased biomass load introduces thermal resistance, potentially leading to temperature gradients and non-uniform heat distribution, which in turn influence the rate and completeness of the pyrolytic reactions. Studies by Mohan et al. (2006) [49] and Bridgwater (2012) [50] have emphasized that greater feedstock mass may hinder effective heat penetration, thereby altering the balance between gas and char production. Accordingly, precise regulation of both residence time and feedstock mass is essential to achieve optimal product distribution and improve the overall efficiency of the pyrolysis process. According to Figure 3, the plot corresponding to the mass flow rate of fuel exhibited a relatively flat slope, indicating a minor or negligible influence on char yield within the studied range. Specifically, increasing the residence time from 120 to 180 min substantially increased the char yield from 25.4% to 55.5%, while reducing pyrolysis gas yield from 74.6% to 44.5%. This observation is consistent with the mechanism of secondary char formation, where prolonged heating allows further solid residue accumulation while volatiles are minimized. Figures 3 and 4 provide a preliminary understanding of the individual influence of fuel mass flow rate, feedstock mass, and residence time on char yield. However, the red warning messages above each plot indicate that each factor also plays a significant role in interaction effects. These warnings do not suggest errors in the model but serve as a reminder that the factors are interdependent and their effects on the response variable (char yield) are conditional. These interaction effects highlight the nonlinear and synergistic nature of the pyrolysis process, where multiple factors collectively determine the final yield and quality of char. Therefore, a full-factorial analysis that accounts for these interactions, as employed in this study, provides a more reliable optimization framework than one that considers main effects alone.

In contrast, an increase in the fuel mass flow rate is directly associated with higher reactor temperatures. Increasing the mass flow rate of fuel (LPG) shows a positive correlation with the increase in reactor temperature. This can be explained by the thermodynamic principle that the total energy entering the system increases in proportion to the product of the mass flow rate of the fuel and its calorific value. When more fuel enters the combustion chamber of an internal combustion engine, the oxidation reaction becomes more intense and results in a higher combustion temperature. The internal combustion engine can only convert a portion of this energy into mechanical energy to generate electricity, while the remaining energy is released as heat through the exhaust gas. Consequently, the exhaust gas temperature increases and causes the rate of heat transfer to the reactor through the helical tube to increase, thus increasing the internal temperature of the reactor. Furthermore, the increase in the amount of combustion products also increases the rate of heat convection. This finding supports the observation that increasing the LPG supply directly impacts the reactor temperature increase. As a result, the char yield decreased from 43.55% to 37.35%, while the gas yield increased by approximately 6.2%. This trend reflects the intensified thermal cracking of heavier organic compounds at elevated temperatures, leading to the formation of more non-condensable gaseous products [60]. At higher thermal input levels, primary pyrolysis vapors undergo secondary cracking reactions that fragment larger molecules into lighter volatiles [61]. This process enhances gas yield while simultaneously diminishing the formation of solid carbonaceous residues [49,50]. Moreover, elevated temperatures may disrupt the structural integrity of intermediate char, contributing to the observed decrease in its yield.

In addition, increasing the mass of feedstock resulted in a notable decline in char yield by 14.54%, while simultaneously enhancing gas yield from 55.1% to 66.04%. This behavior can be attributed to the limitations in heat transfer efficiency within larger biomass loads in fixed-bed configurations. Greater biomass volume introduces increased thermal resistance and can create temperature gradients within the reactor, leading to uneven pyrolysis [62]. While the outer layers may reach the target pyrolysis temperature and release volatiles efficiently, the inner core may remain under-pyrolyzed, thereby reducing overall char formation [58,59]. Nonetheless, the larger amount of feedstock also contributes to greater volatile release, which explains the corresponding increase in gas-phase product formation. These findings emphasize the critical role of thermal control and reactor loading in optimizing the yield distribution of pyrolysis products. The thermal evaluation reveals that temperature, although not explicitly controlled as a parameter, plays an indirect but significant role mediated by fuel flow rate and reactor loading. To support this, future work may incorporate temperature distribution inside the reactor and thermogravimetric analysis (TGA) of the feedstock to determine thermal stability regions.

Compared to other studies, the char yields obtained in this work under comparable pyrolysis temperatures (350–450 °C) fall within the expected range for manure-based biomass, which typically ranges from 30% to 55% depending on feedstock composition and operating conditions. However, the distinctive aspect of this study lies in the utilization of engine exhaust heat, transferred through a helical coil embedded within the reactor, to drive the pyrolysis process. This passive heat recovery method not only eliminates the need for external electric or fossil fuel–based heating but also enhances thermal efficiency by directly coupling exhaust heat from combustion engines with the pyrolytic conversion of biomass. The integration of a helical heat exchanger offers an extended heat transfer surface area and promotes uniform temperature distribution along the reactor length, which is critical for ensuring consistent pyrolysis reactions and product yield stability [63]. Furthermore, this approach aligns with the principles of circular economy and sustainable energy systems by valorizing agricultural and animal manure streams while utilizing otherwise wasted thermal energy [64]. As such, the reactor configuration proposed in this study presents a promising step toward developing decentralized, low-cost, and energy-autonomous pyrolysis technologies suitable for rural or off-grid communities with limited access to conventional energy infrastructure.

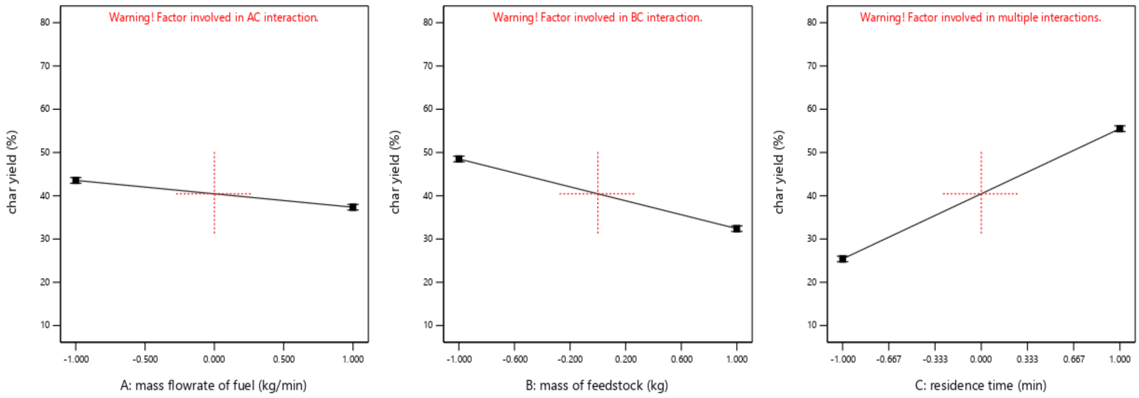

Figure 3. Main effect of process parameters on char yield.

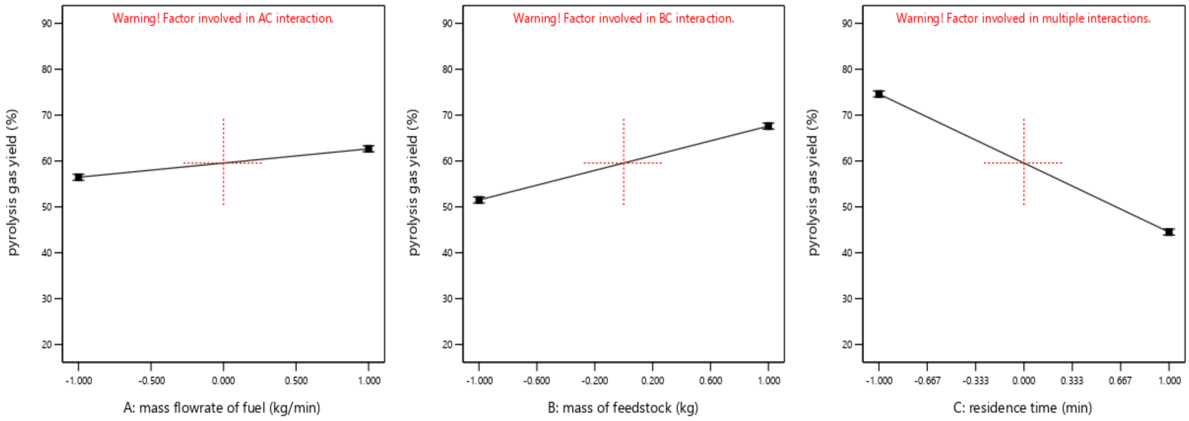

Figure 4. Main effect of process parameters on pyrolysis gas yield.

The influence of key process parameters, namely fuel mass flow rate, feedstock mass, and residence time, reveals contrasting effects on char and pyrolysis gas yields as shown in Table 5. An increase in fuel mass flow rate resulted in a reduction in char yield (–6.2%) and a simultaneous increase in gas yield (+6.2%). This phenomenon can be attributed to elevated reactor temperatures associated with higher fuel combustion rates, which accelerate devolatilization and facilitate secondary cracking reactions [65]. These thermal pathways favor the conversion of solid intermediates into volatile compounds, thereby enhancing gas-phase product formation while reducing char yield [66]. The mass of feedstock also exhibited a notable negative effect on char yield (–16.1%) and a positive effect on gas yield (+16.1%). Larger feedstock volumes may introduce greater thermal inertia within the reactor system. This increased thermal mass can lead to uneven heat distribution, particularly when heating durations are fixed. As a result, the completion of the carbonization process may be hindered. Incomplete carbonization can promote the release of additional volatile compounds, thereby affecting the overall yield and composition of pyrolysis products [67].

Table 5. Percentage effect of process parameters on response parameters.

Parameters	Standardized effect		
	Effect of mass flow rate of fuel	Effect of mass of feedstock	Effect of residence time
Char yield (%)	−6.2	−16.1	30.1
Pyrolysis gas yield (%)	6.2	16.1	−30.1

In contrast, residence time demonstrated the most significant influence, with a strong positive effect on char yield (+30.1%) and an equally strong negative effect on gas yield (–30.1%). Extended residence time allows more complete thermal degradation of organic matter, stabilizing carbon within the solid phase and reducing the availability of volatiles for gas formation. Furthermore, extended residence time may enhance the occurrence of condensation or recombination reactions involving volatile intermediates, leading to their incorporation into the solid product as secondary char [68]. Understanding these parameter dynamics is essential for optimizing the pyrolysis process based on target product yields and highlights the importance of integrated experimental design when developing low-energy and resource-efficient biochar systems.

The optimization of pyrolysis process parameters plays a crucial role in maximizing the yield and quality of its primary products, particularly char and pyrolysis gas [69]. Several operational variables, including feedstock mass, residence time, and the mass flow rate of fuel, have been identified as critical factors influencing the thermal decomposition behavior of biomass [26]. Understanding how these parameters interact is essential for designing efficient pyrolysis systems that ensure complete conversion and minimize energy losses. The effect of each parameter on product yield and key interactions among them are presented in Figures 5–8 and Table 5. Figure 5 illustrates the interaction between feedstock mass and char yield, indicating an inverse relationship: as the feedstock mass increases, the char yield tends to decrease. For instance, when the mass flow rate of fuel and residence time were kept constant at 0.008 kg/min and 180 min, respectively, a feedstock mass of 2.5 kg resulted in a char yield of 61.6%, whereas increasing the feedstock mass to 4 kg reduced the yield significantly to 39%. This decline can be attributed to the larger volume of material requiring uniform heating. As the quantity of feedstock increases, heat distribution within the pyrolysis reactor becomes more challenging, potentially leading to incomplete thermal decomposition in portions of the feedstock. Consequently, the conversion efficiency from biomass to char is reduced. Additionally, higher reactor temperatures play an important role in facilitating the dehydration and decomposition of biomass [70]. However, a large mass of feedstock can limit the effective residence time at these temperatures. As a result, the pyrolysis process may remain incomplete, leading to a reduced char yield [13]. In terms of residence time, longer durations such as 180 min tend to enhance char yield compared to shorter periods like 120 min. However, the positive effect of extended residence time is counteracted when the feedstock mass is also increased, as the thermal demand becomes harder to satisfy. The data reveal a clear interaction: at higher residence times, the negative impact of increasing feedstock mass on char yield becomes more pronounced. Therefore, optimizing feedstock mass relative to the heating duration is essential to achieving high char yields in the pyrolysis process.

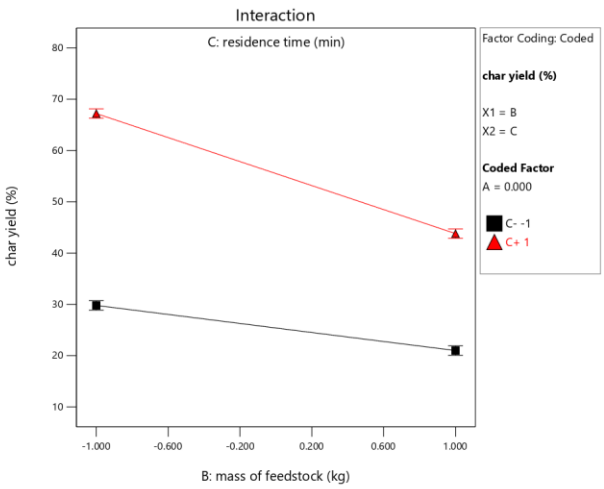

Figure 5. Interaction plot of the mass of feedstock on the char yield (%).

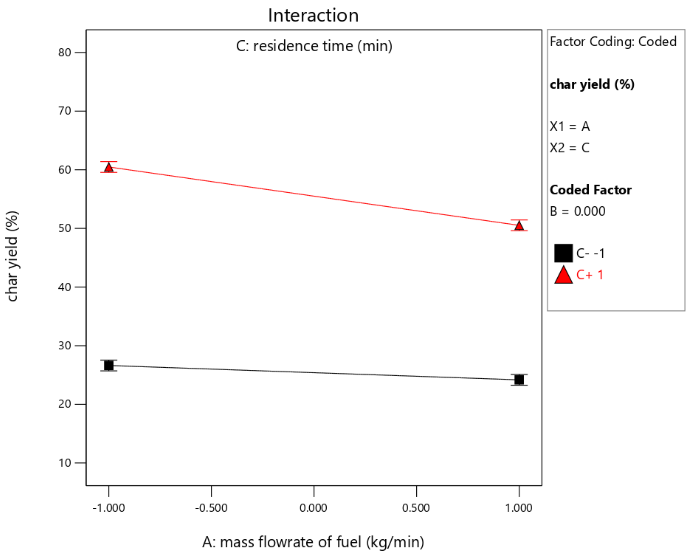

Figure 6. Interaction plot of the mass flow rate of fuel on the char yield (%).

To evaluate the relevance of the char yields obtained in this study, a comparison with previous manure pyrolysis studies was conducted, as summarized in Table 6. Sheep manure produced the highest char yield (72.03% ± 0.25%) when pyrolyzed at a relatively low temperature of 360 °C for an extended residence time of 180 min. This suggests that long residence time under moderate thermal conditions enhances carbon retention, likely due to the progressive devolatilization and stabilization of fixed carbon. Cow manure with a char yield of 62.17% at a lower temperature of 300 °C and 60 min of residence time also demonstrates high carbon conversion efficiency. This indicates that lower pyrolysis temperatures can still produce biochar. In contrast, goat manure yields only 44.5% ± 0.29% char despite undergoing pyrolysis at a higher temperature (400 °C), but with a short residence time of 30 min. This lower yield may result from accelerated volatilization at higher temperatures without enough time for carbon restructuring into char. Similarly, dairy cattle manure exhibits a moderate char yield of 49.1% at a wide temperature range of 400–550 °C, though the residence time is not specified.

Table 6. Comparison of char yields from different manure feedstock.

Feedstock	Residence time (min)	Temperature (°C)	Char yield (%)	Reference
Sheep manure	180	360	72.03 ± 0.25	This study
Goat manure	30	400	44.5 ± 0.29	[71]
Dairy cattle manure	-	400–550	49.1	[18]
Cow manure	60	300	62.17	[72]

Following the analysis of the interaction between feedstock mass and residence time, it is also crucial to examine how the fuel mass flow rate affects char yield under similar pyrolysis conditions. According to Figure 6, there is an inverse relationship between the mass flow rate of fuel and the resulting char yield. As the fuel mass flow rate increases, the pyrolysis reaction tends to proceed more rapidly due to the higher amount of heat energy being introduced into the reactor. This rapid temperature rise accelerates the reaction rate but may also shorten the effective residence time of the feedstock at elevated temperatures [73]. Consequently, the thermal decomposition process may become incomplete, leading to a lower char yield. Therefore, controlling the fuel mass flow rate is essential to maintain stable reactor temperatures and to ensure sufficient residence time for optimal pyrolysis performance and maximum char production. Furthermore, the figure illustrates a significant interaction between the mass flow rate of fuel and residence time. A longer residence time generally results in a higher char yield compared to a shorter residence time [74]. However, the detrimental effect of increasing the fuel mass flow rate on char yield becomes more pronounced at longer residence times. In both short and long residence time scenarios, increasing the fuel flow rate tends to reduce char yield, though the decline is noticeably steeper when the residence time is longer. This emphasizes the importance of carefully balancing fuel input and reaction duration to achieve efficient biomass conversion.

In addition to char yield, understanding the dynamics of pyrolysis gas yield is essential for evaluating the overall performance of the pyrolysis process. Figure 7 illustrates the interaction between these two variables on gas yield. Generally, a longer residence time results in a higher gas yield compared to a shorter one. This trend is clearly demonstrated by the graph, where the black line representing the longer residence time consistently lies above the red line across all feedstock mass values. Increasing the feedstock mass leads to a higher pyrolysis gas yield at both short and long residence times; however, the effect is more pronounced at shorter residence times. This phenomenon can be attributed to the accelerated pyrolysis reactions under shorter residence times, which promote the release of volatile gases before the feedstock begins to convert significantly into char [73]. In contrast, at longer residence times, the feedstock has more time to undergo complete devolatilization, leading to a greater release of gaseous products in the early stages of pyrolysis [75]. Nevertheless, if the residence time is too prolonged, most volatiles may have already been released early, reducing the gas yield in later stages. Additionally, this decline can be attributed to secondary reactions, during which the initial pyrolysis gases decompose further or react to form heavier products such as tar or even revert to char [75], ultimately reducing the total pyrolysis gas yield.

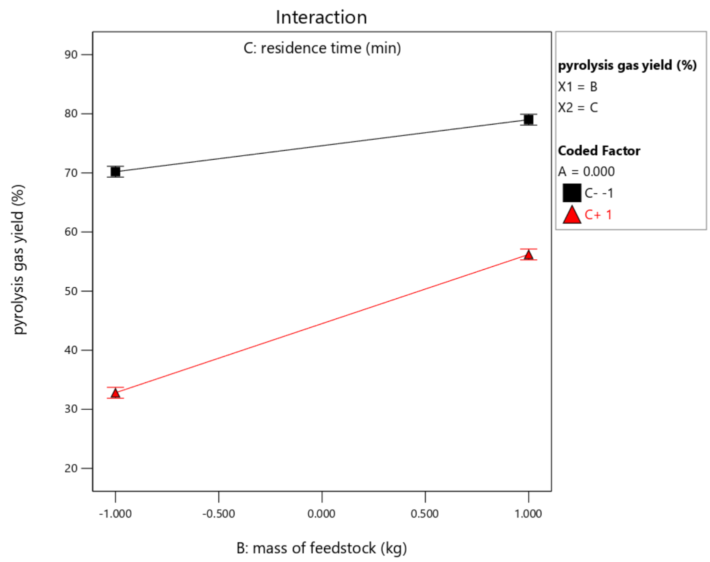

Figure 7. Interaction plot of feedstock mass on pyrolysis gas yield (%).

Following the analysis of how feedstock mass and residence time influence pyrolysis gas yield, it is also essential to examine the role of fuel mass flow rate in this context. Figure 8 illustrates the interaction between the mass flow rate of fuel and residence time on pyrolysis gas production. At both residence times of 120 and 180 min, the gas yield increases as the fuel mass flow rate increases, as evidenced by the upward trends of the red and black lines, respectively. This indicates that higher fuel flow rates deliver more heat energy into the system, increasing the reactor temperature and accelerating the pyrolysis process. Elevated temperatures are known to enhance the rate and extent of both primary and secondary pyrolysis reactions, resulting in more efficient conversion of biomass into gaseous products.

However, residence time also plays a critical role in determining the product yield. Shorter residence times tend to produce higher gas yields, which can be attributed to the rapid expulsion of volatiles before they can undergo secondary reactions [76]. Prolonged residence time may promote secondary reactions such as cracking, re-polymerization, and condensation of volatiles into tar or even char, thereby reducing the gas fraction [77]. This observation aligns with findings by Tripathi et al. (2016), who reported that at elevated temperatures (above 500 °C), shorter residence times are more favorable for maximizing syngas production during pyrolysis of lignocellulosic biomass [78]. Similarly, Mohammed et al. (2011) found that rapid heating coupled with short residence time enhanced gas yield due to limited secondary tar formation [79]. In contrast, longer residence times may be beneficial for char production, as seen in earlier sections of this study, but they appear to be less effective in maximizing gaseous products. Therefore, for applications prioritizing gas yield, such as syngas generation for energy or synthesis purposes, operating under higher fuel flow rates and shorter residence durations appears to be the most favorable strategy. This underlines the importance of fine-tuning thermal conditions within the reactor to steer the product yield toward desired outputs, whether gas, liquid, or solid.

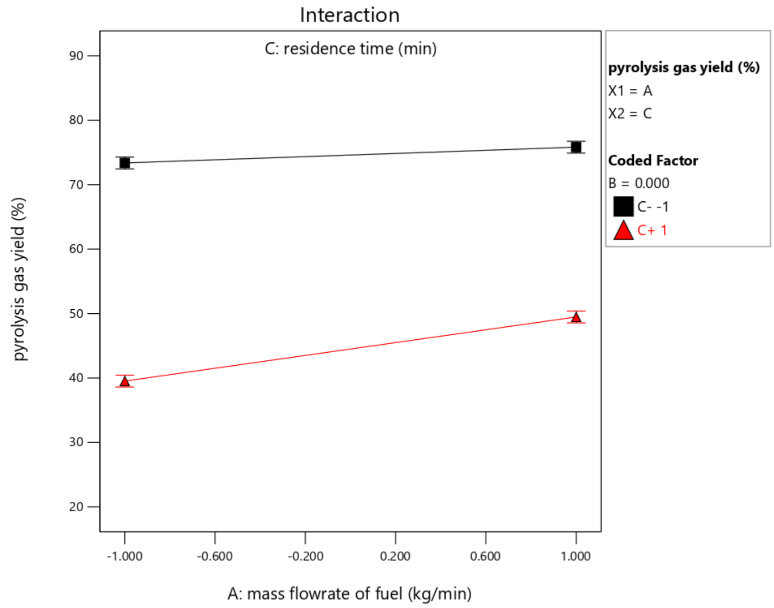

Figure 8. The interaction plot of the mass of the flow rate on the pyrolysis gas yield (%).

3.2. Prediction of product yields

Based on the experimental design analysis, a first-order regression model was developed to describe the relationship between process parameters and the yield of pyrolysis products. This mathematical model represents the yield of char and pyrolysis gas as functions of three primary process variables: fuel mass flow rate (A), feedstock mass (B), and residence time (C), including their significant interaction effects. The regression equations are expressed in uncoded units, meaning that the actual physical values of each factor were used rather than normalized or standardized values. The derived models are shown in Eqs (3) and (4):

$$Y_{char} = -122.93 + 6275 * A + 13.6 * B + 1.47 * C - 62.5 * AC - 0.16 * BC \quad (3)$$

$$Y_{pyro.gas} = 222.95 - 6275 * A - 13.6 * B - 1.47 * C + 62.5 * AC + 0.16 * BC \quad (4)$$

These equations indicate that both main effects and interaction terms play a significant role in determining pyrolysis product yields. For instance, the negative coefficients of the interaction terms AC and BC in Eq (3) suggest that the combined effect of fuel flow rate and residence time, as well as that of feedstock mass and residence time, significantly reduces char yield under certain conditions. Such interaction effects are commonly reported in pyrolysis modeling studies, where the thermal behavior of the system becomes more complex due to nonlinear heat transfer and reaction kinetics at different scales and loadings [50]. To validate the predictive capability of the developed regression model, an additional set of experiments was conducted under varied operating conditions, as detailed in Table 7. The results of these validation experiments were compared with the model predictions to assess the model's accuracy. This step is crucial to ensure the robustness of the model, as recommended in previous works focusing on the statistical modeling of pyrolysis processes [80]. The close agreement between predicted and experimental values demonstrates that the model is capable of reliably simulating the behavior of the system, and therefore, can be used to support process optimization and scale-up efforts.

Table 7. Experiments for model validation.

No	Factor 1 A: Mass flow rate of fuel (kg/min)	Factor 2 B: Mass of feedstock (kg)	Factor 3 C: Residence time (min)	Response 1 Char yield (%)	Response 2 Pyrolysis gas yield (%)
1	0.009	2.5	120	27.35	72.65
2	0.008	2.5	180	62.22	37.78
3	0.007	2.5	150	48.5	51.5
4	0.009	3.2	120	23.24	76.76
5	0.008	3.2	180	51.3	48.7
6	0.007	3.2	150	40.99	59.01

To evaluate the reliability and predictive performance of the mathematical model developed in this study, a comparison between experimental results and model predictions was conducted. Such validation is essential in pyrolysis research to ensure that the model not only captures the trends observed in the experimental data but also provides accurate predictions across the range of tested process parameters. A reliable model can serve as a valuable tool for optimizing pyrolysis conditions without the need for extensive trial-and-error experimentation. Therefore, both visual and quantitative assessments were employed to determine the model's accuracy in predicting char and gas yields. Figure 9 illustrates the comparison between predicted values generated by the mathematical model and the actual experimental values for both char yield and pyrolysis gas yield. This visual comparison serves as a preliminary validation of the model’s predictive performance. A close alignment between predicted and observed data points suggests a well-fitted model, while noticeable deviations indicate areas requiring further refinement. To quantitatively assess the accuracy of the model, MAE was calculated using Eq (1), which provides a direct and interpretable measure of the average magnitude of prediction error, regardless of its direction. The MAE is a commonly adopted metric in model evaluation due to its simplicity and effectiveness in measuring prediction performance [55]. In this study, MAE was determined to be 2.8, based on a data range spanning from 23.24 to 72.65. This corresponds to an average deviation of approximately 5.67% relative to the total data range. This indicates a satisfactory level of predictive accuracy. Furthermore, the model yielded an R^2 of 0.94, demonstrating that 94% of the variance in the experimental data can be accounted for by the model. This high R^2 value reflects a strong correlation between the predicted and observed outcomes, reinforcing the model’s reliability and robustness in estimating char and gas yields across diverse process conditions.

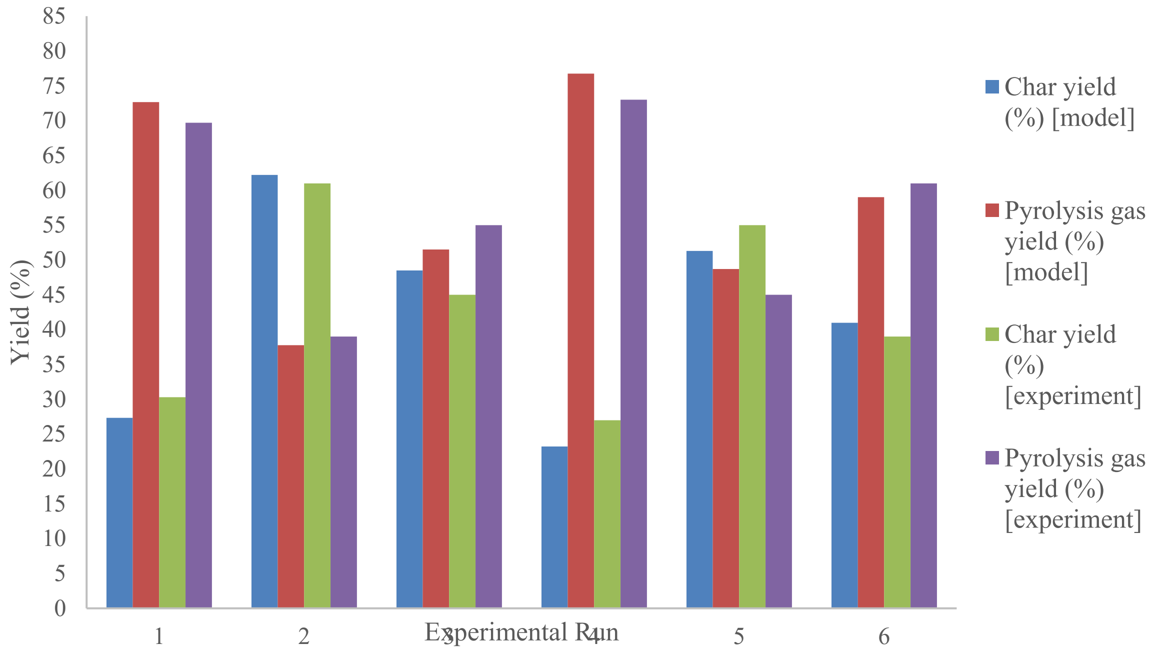

Figure 9. Comparison of model data with experimental data.

4. Conclusions

This study demonstrates the potential utilization of sheep manure as a sustainable feedstock for pyrolysis in a fixed-bed reactor, which is heated using waste heat from an internal combustion engine exhaust gas through a helical heat exchanger. A two-level factorial experimental design was employed to investigate the effects of three key process parameters: fuel mass flow rate, feedstock mass, and residence time. The findings indicate that all three parameters significantly affect the product yield distribution between char and pyrolysis gas. Among them, residence time is the most dominant factor, contributing 30.1% to the increase in char yield and inversely affecting pyrolysis gas yield. Specifically, increasing residence time from 120 to 180 min significantly enhanced char yield (from 25.4% to 55.5%) while reducing pyrolysis gas yield (from 74.6% to 44.5%). Increasing the fuel mass flow rate, which correlates with higher reactor temperatures, decreased char yield (from 43.55% to 37.35%) but slightly increased gas yield by 6.2%. Meanwhile, a higher feedstock mass reduced char yield by 14.54% but increased gas yield from 55.1% to 66.04%. The developed first-order regression model successfully captured the main effects and interactions of the parameters, with a high R^2 value of 0.94 and an MAE of 2.8, indicating excellent predictive accuracy. From a practical standpoint, this research supports the valorization of livestock waste into valuable pyrolysis products and demonstrates the feasibility of using engine waste heat, paving the way for more integrated and energy-efficient bioresource utilization systems.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

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Conflict of interest

The authors declare no conflicts of interest.

Author contributions

Conceptualization, methodology: Illa Rizianiza; supervision: Ahamd Indra Siswantara; review and editing: Eny Kusrini. All authors have read and agreed to the published version of the manuscript.

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