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Thermal decomposition of wheat straw pellets in a nitrogen environment: Characterization using thermogravimetric analyzer

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ABSTRACT

This experiment used a thermogravimetric (TG) analyzer within a nitrogen environment, investigating the thermal degradation patterns of wheat straw pellets (WSP) under temperatures ranging from 31 to 800 °C and varying heating rates (5, 10, and 20 °C/min). Two pellet types were considered: T₁ (100 % wheat straw) and T₂ (70 % wheat straw, 10 % sawdust, 10 % bentonite clay, and 10 % biochar). This study comprehensively analyzes WSP's thermal degradation, emphasizing model selection, composition effects, heating rate, and temperature. Results highlighted higher volatile matter content and calorific value in WSP. Model-free methods were applied to analyze TG/DTG profiles, revealing three distinct zones in WSP thermal decomposition: drying, devolatilization, and carbonization. Devolatilization, especially its 1st and 2nd steps, was extensively examined, with a significant mass loss (approx. 65 %) observed between 150 and 550 °C. Higher heating rates induced a shift in thermogravimetric profiles to elevated temperatures. Maximum mass loss rates during devolatilization ranged from 4.41 to 16.28 %/min for T₁ and 4.0–15.9 % for T₂ pellets. Temperature significantly influenced mass loss and reaction rates, whereas heating rates had a negligible impact. Thermodynamic properties indicated equilibrium reactions during pyrolysis for both T₁ and T₂ pellets. Additionally, increasing heating rates correlated with an upward trend in the reactivity index. The findings contribute valuable knowledge for optimizing biomass utilization in combustion and pyrolysis processes.

1. Introduction

Biomass is acknowledged as a clean, alternative, renewable, and sustainable form of bioenergy [1]. It is the fourth most significant energy source globally, following coal, oil, and natural gas [2]. Comprising approximately 14 % of the total global energy demand, biomass is a valuable energy reservoir for developing nations, addressing nearly 35 % of their energy needs [3]. The increasing availability of biomass fuels is considered for its carbon-neutral attributes, with the carbon released as CO₂ during combustion being offset through photosynthesis in plants [4]. The recent progress in conversion technologies makes biomass a growing potential biofuel choice [5]. Consequently, the extensive and abundant biomass reservoir is utilized to fulfill diverse energy needs, including power generation, heating, and the production of liquid fuels through thermal conversion.

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Using biomass as an energy source involves primarily two facets: thermochemical processes (such as direct combustion, gasification, and pyrolysis) and biochemical conversion processes [6]. Among these, pyrolysis stands out as one of the most intriguing thermochemical conversion methods, capturing increasing attention from researchers for its potential to produce gaseous, liquid, and solid fuels from biomass [7]. However, biomass pyrolysis is an intricate procedure encompassing various physical and chemical processes that entail numerous parallel and competing reactions [8]. Hence, to better comprehend the intricacies of the process and enhance pyrolysis performance, there is a need for kinetic studies of biomass. The analysis of the kinetics of the pyrolysis process is crucial for designing and developing models, as well as for scaling up biomass conversion systems through pyrolysis. Additionally, it plays a vital role in predicting reaction characteristics and optimizing the process to achieve the desired product distribution from pyrolysis [9].

The primary biomass sources for generating biofuels encompass agricultural residues, forest wastes, industrial by-products, and specialized energy crops [10]. Among them, wheat straw is a significant field crop residue, which can be a globally available resource with substantial carbon and volatile matter content suitable for use as a fuel feedstock. The global production of wheat in 2021/22 is estimated to be approximately 780 MMT (million metric tons), accounting for 1013 MMT (<https://www.statista.com/statistics/267268/production-of-wheat-worldwide-since-1990/>). In contrast, Australia alone produced 45.0 MMT of wheat residue as crop waste. Managing wheat straw at the field level often presents challenges, leading to on-field burning. However, given its renewable nature, wheat straw holds a prominent sustainable energy source for biofuel production despite its tendency to burn quickly due to a higher flash point. To address this issue, scientists have suggested adding additives to convert low-quality wheat straw into a higher-value solid fuel [11]. This research specially employed wheat straw (WS) pellets, adding different biomass-based additives to enhance their properties and value for biofuel production.

Many researchers have experimented with biomass pyrolysis [12–14]. The majority of these pyrolysis investigations were conducted on small-scale laboratory equipment, primarily focusing on feasibility studies and reactivity values about time and temperature. The scientist mentioned that the detailed pyrolysis experimental study is sometimes complicated, challenging, and costly [15]. As an alternative, the thermogravimetric analyser (TGA) is a frequently used tool for pyrolysis and combustion for extensive study [16–18]. Therefore, this study investigated wheat straw pellet pyrolysis decomposition behavior with thermogravimetric (TG) analysis. It is an excellent way to determine how solid fuels react to heat during pyrolysis and combustion [19].

Several studies have utilized the TGA approach to examine thermal decomposition patterns and kinetics in the pyrolysis of various samples, including biomass, coals, and their blends (Table 1). This research used the STA449F3 Jupiter with NETZSCH Proteus 8.0 software. Various methods have been developed to assess kinetic parameters using TGA results, falling into two categories: model-fitting and model-free (iso-conversational) processes. Previous research indicated that model fitting is suitable for analyzing thermokinetic parameters, leading this study to consider model-based methods.

Recently, there has been extensive global research focusing on the physicochemical characterization, pyrolysis decomposition, and kinetics of various biomass and waste materials. However, a notable gap exists in the literature concerning kinetic decomposition evaluation, with a particular scarcity of reports on wheat straw pellet (WSP) pyrolysis. Therefore, the impact of the wheat straw pellet and additives on kinetic studies still needs to be studied. In addition, in-depth studies on biomass blends with additives still need to be investigated, particularly in agricultural straws (wheat, rice, etc.). Furthermore, the kinetics of the co-pyrolysis process involving biomass-based materials are not well understood. Therefore, conducting thermogravimetric (TG) analysis on wheat straw pellets

Table 1

Recent review articles and reports have explored the thermogravimetric (TG) analysis of pyrolysis decomposition behavior in biomass.

Materials	Technology	Findings	Reference
Rice husk	Pyrolysis (TGA)	Thermal stability and thermal degradation process	[96]
Napier grass	Combustion and Pyrolysis (TGA)	Thermal characteristics	[97]
Corn brakes, wheat straw, and hazelnut shell	Pyrolysis (TGA-MS)	Gas product increase with heating rate	[98]
Sugarcane straw	Slow pyrolysis (TGA-FTIR)	Decomposition increases with the heating rate	[99]
Corn stover	Combustion and Pyrolysis (TGA)	TGA characteristics depend on heating rates and temperatures	[100]
Rice straw and pine sawdust	Combustion and Pyrolysis (TGA)	Pyrolysis kinetics characteristics depend on analysis models	[66]
Wheat straw and plastic	Combustion and Pyrolysis (TGA)	Synergistic effect on blending materials	[101]
Mustard straw	Pyrolysis (TGA)	Biochar, bio-oil, and hydrocarbon gases are influenced by temperature	[102]
Corn straw powder, poplar wood chip, and rice husk	Combustion and Pyrolysis (TGA)	Pyrolysis and combustion characteristics boost heating rates	[2]
Sewage sludge and wheat straw	Pyrolysis (TGA)	Mixing ratio has an impact on pyrolysis yield	[103]
Empty fruit bunch, rice husk, coconut pulp, sawdust, coconut shell, and sugar cane bagasse	Pyrolysis (TGA)	Heating value and thermal degradation rate rose with operating conditions such as temperature, time, and reactors	[104]
Biomass wastes and digests biomass wastes	Co-pyrolysis (TGA)	The blending process enhances the gas yield	[105]
Wheat straw	Pyrolysis (TGA-FTIR)	Pyrolysis or DTG changes with temperature	[106]
Wheat straw, rape straw, reed canary grass and switch grass	Pyrolysis (TGA)	Perennial grass has more attractive properties for the first pyrolysis	[107]

becomes crucial to elucidate pyrolysis properties, aiding in the design of reactors and transforming biomass into energy. Subsequently, these derived kinetic parameters serve as essential input data for Computational Fluid Dynamics (CFD) modeling, enabling the design and analysis of the energy conversion reactor, specifically a gasifier, for the pellets.

On the other hand, Sher et al. [20] investigated the thermal and kinetic analysis of various materials, including barley straw, waste wood, wheat straw, willow, miscanthus, and wood pellets. Their findings revealed a higher activation energy coupled with lower reactivity. Pešenjanski et al. [7] focused on the thermal degradation of wheat straw samples, indicating that heating rates significantly influence mass losses, while moisture content does not impact the process. Greenhalf et al. [21] carried out experimental studies on the thermal behavior and kinetics of wheat straw, switchgrass, miscanthus, willow, and beech wood, utilizing both thermogravimetric analysis (TGA) and a laboratory-scale continuous-fed bubbling fluidized reactor. Their observations indicated that wheat straw contains a higher bio-oil content with more water than perennial grass.

Recently, Fonseca et al. [18] examined the challenges associated with determining kinetic parameters in the pyrolysis of high ash-content wheat straw. They employed model-fitting approaches and an isoconversional model to identify degradation kinetics, revealing a dependence on potassium content. Despite the wealth of research, the literature review highlights the scarcity of thermokinetic pyrolysis studies specifically focused on additive blends of wheat straw pellets and individual wheat straw pellets.

This study aims to assess the potential of pyrolysis decomposition from Wheat Straw Pellets (WSP) in transforming agricultural residues into valuable products for energy recovery. The proposed approach uses thermogravimetric analysis to analyze WSP's solid fuel characteristics comprehensively. This analysis is crucial for gaining insights into solid fuel combustion, which, in turn, contributes to the design, construction, and operation of large-scale industrial reactors [22].

The primary objectives of this pyrolysis study include (a) employing model-free methods to examine thermal decomposition techniques, (b) determining burning profile parameters, and (c) establishing quality indicators of thermal decomposition, such as reactivity and burning parameters. These objectives aim to enhance our understanding of the thermal behavior of WSP, providing valuable insights for optimizing energy recovery processes from agricultural residues.

Nomenclature

HHV	Higher Heating Value	M	Mass
CFD	Computational Fluid Dynamics	TG	Thermogravimetric
TA	Thermal Analysis	TGA	Thermogravimetric Analyser
DTG	Derivative Thermogravimetric	WS	Wheat Straw
A	Pre-exponential factor	WSP	Wheat Straw Pellet
E_a	Activation of energy	R	Universal gas constant
ΔG	Gibbs free energy	h	Planck constant
ΔH	Enthalpy/latent heat enthalpy	T_p	The peak temperature of devolatilization
ΔS	Entropy	T_f	Final Temperature of devolatilization
R_M	Reactivity index	DTG_{max}	Maximum weight loss rate of devolatilization
P_f	Pyrolysis factor	T_i	The initial temperature of devolatilization
R_a^{dev}	Average devolatilization rate	T_m	Maximum temperature at which maximum decomposition occurred

2. Materials and methods

2.1. Sample and sample preparation

In this study, two wheat straw pellets (T_1 : without additives and T_2 : with additives) were manufactured with different material combinations (weight proportions). The pellets were subsequently assessed using a thermogravimetric analyzer (TGA). The chemical analysis of the materials was done in the previous study following the standard prescribed techniques [23].

The pellet was a solid cylindrical fuel made from wheat straw (Table 2). However, in the case of TGA, it is necessary to grind the samples to enhance the surface area [24] and conversion efficiency [25]. Before this analysis, the samples were dried at 105 °C for 24 h in an oven. Subsequently, they were finely ground into a powder, with each particle attaining an average size of 1 mm (Fig. 1). To ensure uniform particle size, the crushed samples were sieved. A dummy test was also conducted at each heating rate to prevent systematic errors and provide baseline information.

To ensure precise experimental results, a consistent sample size (weight) for each treatment is essential [26]. They explicitly considered 50 mg as the initial weight for every run. However, it is essential to note that the TGA pan's sample holding capacity ranged

Table 2
Pellet physical characteristics.

Pellets	Average length, mm	Mean diameter, mm	Bulk density, Kg/m ³
T_1	22.0	8.21	244.79
T_2	37.0	8.13	607.40

Note.

T_1 : 100 % Wheat straw.

T_2 : 70 % Wheat straw, 10 % Bentonite clay, 10 % Sawdust (*Pinus Radiata*) and 10 % Biochar (coconut shell).

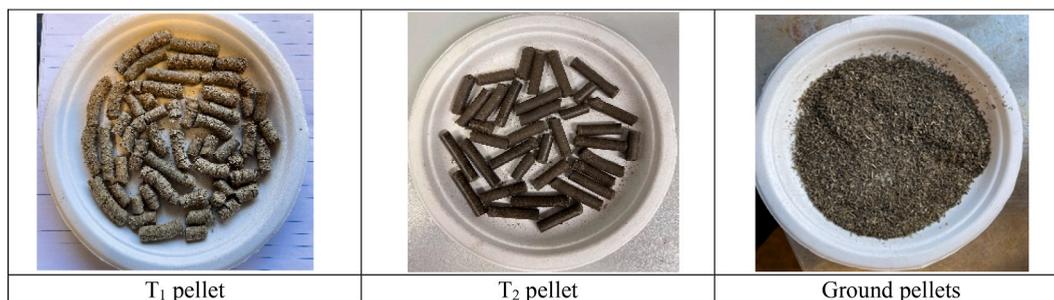


Fig. 1. Visual representation of the experimental materials.

from 8.75 to 9.75 mg. Specifically, they considered 50 mg as the initial weight for every run. However, it is essential to note that the TGA pan's sample holding capacity ranged from 8.75 to 9.75 mg.

2.2. Thermogravimetric analyser (TGA)

Investigating the thermal behavior and kinetics of carbonaceous materials and fuels is a commonly used technique in TGA [27]. This research utilized the STA 449F3 Jupiter TGA from Erich NETZSCH GmbH & Co. Holding KG, Germany, to observe and document samples' continual weight loss dynamics as temperature and time increased [28]. Throughout the experimentation, the pyrolysis of the feedstock in the control zone under a pressure of 0.1 MP, with Nitrogen serving as the carrier gas was maintained at a consistent flow rate of 50 ml/min. For the current investigation, the thermal behavior profile (TG/DTG) was derived by the kinetic software NETZSCH Proteus 8.

2.3. Data analysis and treatments

The reaction rate of the samples during pyrolysis was determined by analyzing the distribution of remaining weight (TG) and the derivative of weight loss (DTG) data. Additionally, the weight loss (TG curves) were used to estimate the degradation of pseudo-components such as hemicellulose, cellulose, and lignin, following the methodology [29]. In contrast, the degradation rate was evaluated based on DTG profiles [18]. A moving average trend line was applied to mitigate noise in the TGA data, a common strategy in examining TGA data [30,31].

The choice of heating rate depends on the specific goals of the research, as it directly influences the decomposition temperatures observed in TG/DTG profiles [32]. Different materials exhibit varying decomposition rates; for instance, woody biomass may require a faster rate for process optimization, whereas more recalcitrant biomass like straw may benefit from a slower rate to balance accuracy and practicality. In the case of wheat straw pellets, where a detailed understanding of the decomposition mechanism is desired, opting for a slower heating rate may be preferable. However, for thermo-kinetic analysis, researchers commonly utilize various heating rates ranging from 2.5, 5, 10, 15, 20, 30, 40, and 50 °C/min [33,34]. The study selected three distinct heating rates—5, 10, and 20 °C/min—to achieve experimentally high accuracy, gain a detailed understanding, and minimize thermal lag, ensuring robust and reliable results for further analysis and interpretation.

In addition, the research considered temperatures ranging from 31 to 800 °C. These heating rates and temperature ranges align with those frequently employed in various experiments to assess the kinetic properties of diverse biomasses [35,36].

2.4. Analyze the TG/DTG profiles

Non-isothermal and isothermal models serve as fundamental tools in analyzing the thermal properties of TG/DTG profiles [37]. Recent advancements have favored non-isothermal techniques due to their simplicity and reduced noise compared to isothermal methods [38]. Within the non-isothermal analysis, two primary approaches exist for estimating kinetic data: model-free and model-fitting [39]. Thermal decomposition data are often analyzed using kinetic models, including model-free and model-based methods [40]. Typically, TG and DTG profiles are utilized to synthesize thermo-kinetic properties gathered at constant heating rates and temperatures [41–43].

Model-free methods are advantageous for their ease of use [44] and ability to accurately identify multi-step processes in complex materials like wheat straw. However, it may not be ideal for single-step reactions [45,46]. Model-based methods, on the other hand, are more sophisticated and can dissect intricate chemical processes with multiple reaction steps [47]. This method is precious for analyzing complex reactions, which is the norm, as roughly 95 % of chemical reactions involve multiple stages [48].

The choice between these methods depends on the material under study. However, model-free analysis can be misleading for complex materials like lignocellulosic biomass (straw) [108], whose decomposition involves multi-step processes. Model-based methods prove more suitable for wheat straw, a biomass known for its complex decomposition [49]. Also, studies have shown that model-based methods are better suited for analyzing wheat straw's thermal degradation patterns, as seen in TG/DTG curves [50]. Therefore, this study focuses on utilizing the more appropriate model-based approach to investigate and contrast the thermal

decomposition patterns of wheat straw pellets.

2.5. Thermodynamic parameters estimation

The thermodynamic parameters are essential in gaining insights into biomass pyrolysis, aiding reactor design, and facilitating scale-up efforts [46]. Calculating these parameters is pivotal for exploring the feasibility of the pyrolysis process and conducting energy assessments. The enthalpy (ΔH), entropy change (ΔS) and gibbs free energy (ΔG) stand out as commonly used thermodynamic parameters. Enthalpy (ΔH) specifically quantifies the energy difference between raw biomass and the activated complex, revealing the amount of heat evolved or absorbed during a process [51]. In addition, entropy change (ΔS) is a comprehensive characteristic used to describe a system's disorder. In contrast, Gibbs free energy (ΔG) characterizes the spontaneity of the process. To determine ΔH , ΔS , and ΔG from kinetic parameters involves specific measures [35,36].

$$\Delta H = E_a - RT \quad (1)$$

$$\Delta G = E_a + RT_p \ln \left(\frac{K_\beta T}{hA} \right) \quad (2)$$

$$\Delta S = \frac{\Delta H - \Delta G}{T_m} \quad (3)$$

Where: K_β = Boltzman constant (1.38×10^{-23}) ($\text{m}^2 \cdot \text{kg} / \text{sec}^2 \cdot \text{K}$)

h = Planck constant (6.626×10^{-34}) ($\text{m}^2 \cdot \text{kg} / \text{sec}$)

T_p = Maximum temperature at which maximum decomposition occurred (K)

R = Universal gas constant (8.3145) ($\text{J} / \text{mole} \cdot \text{K}$)

A = Activation energy (kJ/mol)

E_a = Pre-exponential factor

For the calculation of ΔH , ΔS , and ΔG , it is necessary to obtain data on the pre-exponential factor (E_a) and activation energy (A) data, which were taken from *NETZSCH with Proteus 8.0* software at a particular conversion for $20^\circ\text{C}/\text{min}$. The thermodynamic parameters were computed based on the highest temperature at which the maximum decomposition occurred, as determined from the DTG profile [52,53].

2.6. Reactivity and pyrolysis characteristics

Determining temperature ranges is essential for analyzing reactivity and pyrolysis characteristics, which can significantly impact the derived values [20]. These parameters influence the residence time and furnace temperature, and their values primarily depend upon the chemical composition of the biomass and the combustion environment [54]. The thermogravimetric profiles (TG/DTG) used to evaluate the thermal stability of the biomass were employed to characterize pyrolysis, including reactivity and the pyrolysis factor. In practice, both the reactivity index and the pyrolysis factor play significant roles in understanding the behavior of biomass during thermal processes like pyrolysis or combustion.

The reactivity index is a thermometer for the material, indicating its propensity to react at varying heating rates. Higher values suggest a greater tendency to combust, making it ideal for controlled burning or efficient energy generation. Conversion biomass-to-energy or biofuel production processes by examining how this index fluctuates with factors like heating rate or biomass composition. The reactivity index at the peak temperature (R_M), while the thermogravimetric profiles (TG/DTG) used to evaluate the thermal stability of the biomass were employed to characterize pyrolysis, including reactivity and the pyrolysis factor. The reactivity index at the peak temperature by Ghetti et al. [55]:

$$R_M = \frac{DTG_{max}}{T_p} \quad (4)$$

Where.

DTG_{max} = Maximum weight loss rate of devolatilization (%/min);

T_p = Peak temperature of devolatilization ($^\circ\text{C}$)

On the other hand, the pyrolysis factor focuses on an essential aspect of pyrolysis: the release of volatile compounds. It measures how effectively these volatile components are liberated from the biomass. Studying how this factor changes with heating rate provides valuable insights into how pyrolysis responds to different temperature conditions, which is essential for designing and predicting the performance of pyrolysis systems. Optimizing the biomass materials and process parameters based on the pyrolysis factor can achieve efficient pyrolysis and enhance the yield of desired products like bio-oil or biochar. The calculation of the pyrolysis factor (P_f) was performed following the methodology outlined in Ghetti et al. [55]:

$$P_F = \frac{DTG_{max} * R_a^{dev}}{T_i^{2*} T_f} \quad (5)$$

Where.

R_a^{dev} = Average devolatilization rate (%/min);
 T_i = Initial devolatilization temperature (°C);
 T_f = Final devolatilization temperature (°C).

In summary, while the reactivity index offers a broad overview of combustion behavior, the pyrolysis factor delves into the specifics of volatile release. Together, these two metrics are indispensable for comprehending and improving any biomass thermal process.

3. Results and discussions

3.1. Properties of wheat straw pellets (WSP)

The fuel characteristics of biomass are primarily determined through the examination of proximate, ultimate analysis, and calorific values. Table 3 displays the analysis results of wheat straw pellets (WSP). Notably, the moisture content in the WSP sample is 6.20 % for T₁ and 3.5 % for T₂. The moisture content in biomass varies significantly, depending on the biomass type and storage methods applied [56]. It is specified that, for pyrolysis, the moisture content in biomass should be below 10 %. Biomass with high moisture content requires additional heat for reduction, potentially negatively impacting the pyrolysis process [57].

The results indicate that WSP exhibits a high volatile matter content (75.61 % for T₁), making it suitable for pyrolysis. Biomass with elevated volatile content is easily devolatilized and more reactive than biomass with low volatile content, leading to lower fixed carbon [58]. The current WSP sample displays a moderate ash content (11.875 % for T₂ and 7.09 % for T₁). The fixed carbon content is 11.10 % for T₁ and 31.60 % for T₂. The ultimate analysis results for T₂ WSP reveal carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O) content of 45.87 %, 6.30 %, 0.72 %, 0.21 %, and 46.90 %, respectively. The presence of sulfur (S) and nitrogen (N) in biomass contributes to the emission of sulfur oxides (SOx) and nitrogen oxides (NOx), causing environmental pollution. The low N and S content in the current wood sawdust implies that this biomass is an attractive source for energy production through pyrolysis with minimal environmental pollution, providing a sustainable solution for energy needs.

The chemical and elemental composition of WSP needs to be considered to understand the behavior of thermal degradation [59]. Literature suggests that biomass cellulose, hemicellulose, lignin, and extractives are essential for conversion processes [60]. Generally, the biomass's cellulose, hemicellulose, and lignin concentrations range from 42–49 %, 16–23 %, and 21–39 %, respectively [61]. The experimental findings indicate that the lignocellulosic composition of WSP consists of cellulose (30.0–41.30 %), hemicellulose (22.40–23.30 %), and lignin (7.0–10.60 %). This composition, with a higher concentration of hemicellulose and cellulose, contributes to a greater bio-oil yield during pyrolysis, ensuring the efficiency of the process [62]. The experimental results demonstrate that the Higher Heating Value (HHV) for T₁ and T₂ is 17.02 and 19.06 MJ/kg, respectively, further confirming the potential of WSP for energy production.

3.2. Analysis of TG/DTG profiles

The biomass pyrolysis thermal profiles are typically divided into three phases with various reaction mechanisms and decomposition activities [63]. Moreover, Jeguirim and Trouvé [64] categorized the TG and DTG curves into active and passive phases. The passive zone (I and III) includes the carbonization and drying zones, whereas the active phase (II) provides devolatilization [65]. In this research, categorizing the TG and DTG profile into three phases (refer to Table 4) is consistent with the study Liu et al. [2] findings.

-TG/DTG profile for wheat straw pellet without additives (T1)

Table 3
Properties of wheat straw pellets.

Pellets	Proximate analysis, %				Ultimate analysis, %					HHV ^d MJ/kg	Dry wt, %		
	MC	VM	FC	Ash	C	H	N	S	O ^a		Hemicellulose	Cellulose	Lignin
T ₁	6.20	75.61	11.10	7.09	44.32	4.90	0.56	0.11	50.11	17.02	23.30	30.00	10.60
T ₂	3.50	53.03	31.60	11.87	45.87	6.30	0.72	0.21	46.90	19.06	22.40	41.30	7.00

Note: MC = Moisture content; VM = Volatile matters; FC = Fixed carbon; C = Carbon.

H = Hydrogen; N = Nitrogen; S = Sulfur; O = Oxygen; * by difference.

aas received, dry basis as denoted in the table.

bOn dry basis except for moisture, which is on a received basis.

^a Calculated by the difference.

Table 4
Pyrolysis decomposition phases/stages.

Phase	Zone Name	Degradation component	Activities
I (passive)	Drying	Free moisture and a limited amount of volatile matters	<ul style="list-style-type: none"> Removal of moisture from below 110 °C
II (active)	Devolatilization	Hemicellulose and cellulose	<ul style="list-style-type: none"> Removal of volatiles and adsorption/absorption of oxygen Mass loss in temperature zone 110–350 °C Produces primary gasses
III (passive)	Carbonization	Cellulose, lignin, and extractives	<ul style="list-style-type: none"> Burnout lignin and produces secondary gases Temperature greater than 350 °C Produces carbon-enriched char/ahs

The first zone (I) of the TG-DTG curve was the dehydration/drying stage, where the temperatures ranged from 31 to 161 °C for the T₁ pellet (Fig. 2(a)). In this passive stage, heating the sample resulted in a certain amount of weight loss due to moisture evaporating. Also, observing Fig. 2(b), modest fluctuation occurred in the DTG curves. Xiao et al. [66] supported these results using the ligno-cellulosic biomass for kinetic pyrolysis investigation. In this phase, the unbound moisture was released between 31 and 100 °C, while bounded water and extractives were removed from 100 to 161 °C.

The second zone (II) is devolatilization, where biomass experiences depolymerization and experiences a "glass transition." Devolatilization zone is the core part of the pyrolysis, and temperatures range from 161 to 556 °C, while the volatiles were released rapidly, and a significant amount of weight loss occurred (Fig. 2(a)). Alternatively, the DTG profiles presented a primary peak along with several side peaks, where major volatile components of WSP were present (Fig. 2(b)). The various peaks depend on the biomass relative to hemicellulose and cellulose concentration [67]. According to Mishra and Mohanty [68], the prominent peak is attributed to

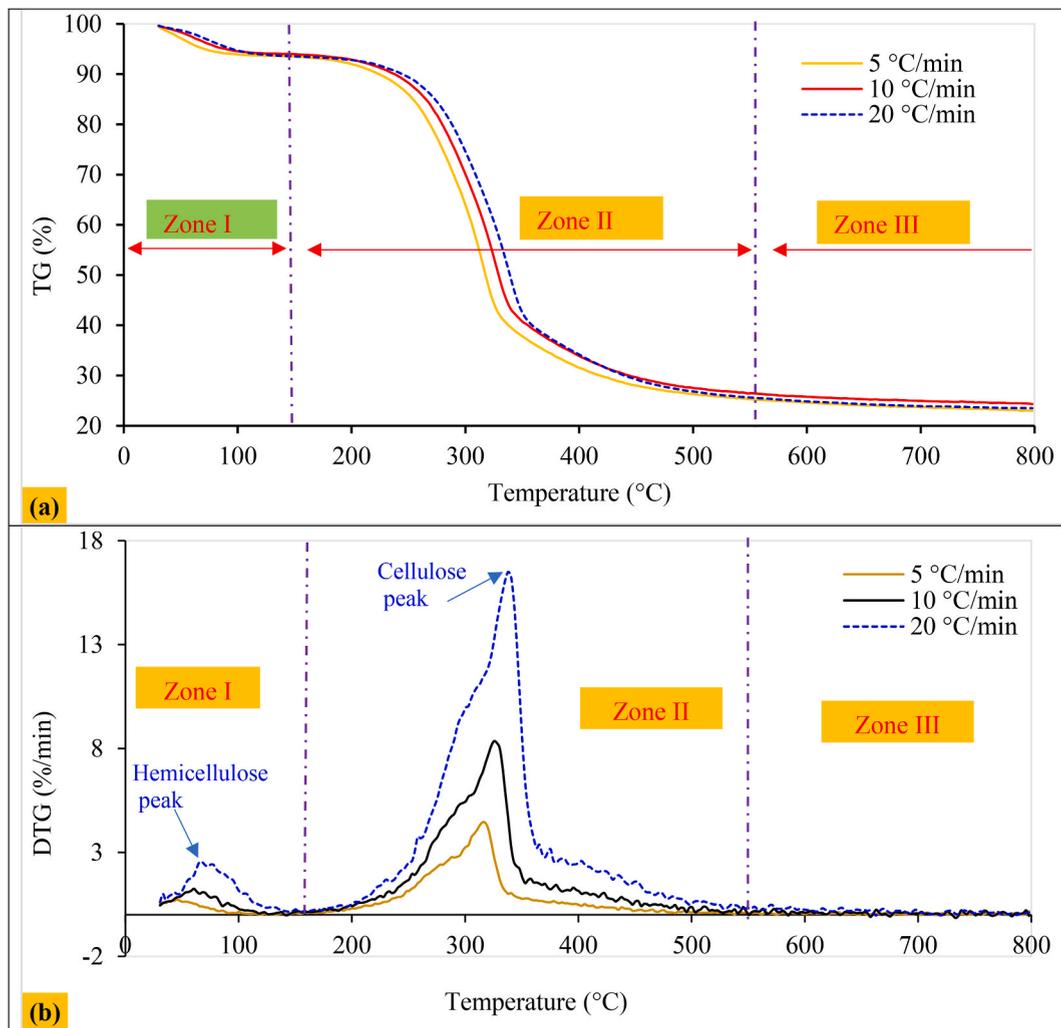


Fig. 2. Thermal decomposition profile of T₁ pellets under N₂ atmosphere: (a) TG curve and (b) DTG curves.

cellulose pyrolysis, while the side peak is associated with hemicellulose pyrolysis.

The last stage (III) was carbonization, where temperatures range from 553 to 800 °C for the T₁ treatment (Fig. 2(a)). In this stage, the remaining hemicelluloses and cellulose nearly solidified into char, and the lignin pyrolysis started [2]. The slow pyrolysis in the third stage resulted in a smaller weight loss amplitude than in other stages. A comparable pattern has been documented by Singh et al. [69]. In Fig. 2(b), the DTG profiles were relatively flat tended, resulting in a very nominal reaction rate. This observation aligns with the findings of Gani and Naruse [70].

- TG - DTG curves for a wheat straw pellet with additives (T2)

Fig. 3(a) shows the TG curves for T₂ pellets, where the WSP decomposition process was allocated into three stages.

The initial stage, called the drying phase, initiates at 31 °C and concludes at 185 °C. This phase can be subdivided into two distinct segments. The initial segment involves expelling free moisture (6 %) from the biomass within the temperature range of 33–100 °C. The subsequent segment within this phase spans the temperature range of 100–185 °C, and the mass reduction during this period is 2 %. This reduction is likely attributed to removing bound moisture and extremely lightweight volatile components from the biomass. This passive zone is also depicted by the subtle peak in the DTG curve, where the maximum reaction rate was 2.33 %/min for a heating rate of 20 °C/min (Fig. 3(b)).

The TG curve (zone II) illustrates the primary degradation of WSP's major components taking place between 185 and 496 °C (Fig. 3(a)). In this second zone (II), a dynamic phase, cellulose primarily undergoes breakdown, leading to maximum mass loss and main devolatilization. This stage can be further divided into two subsections regarding mass loss. The initial subsection occurs within the

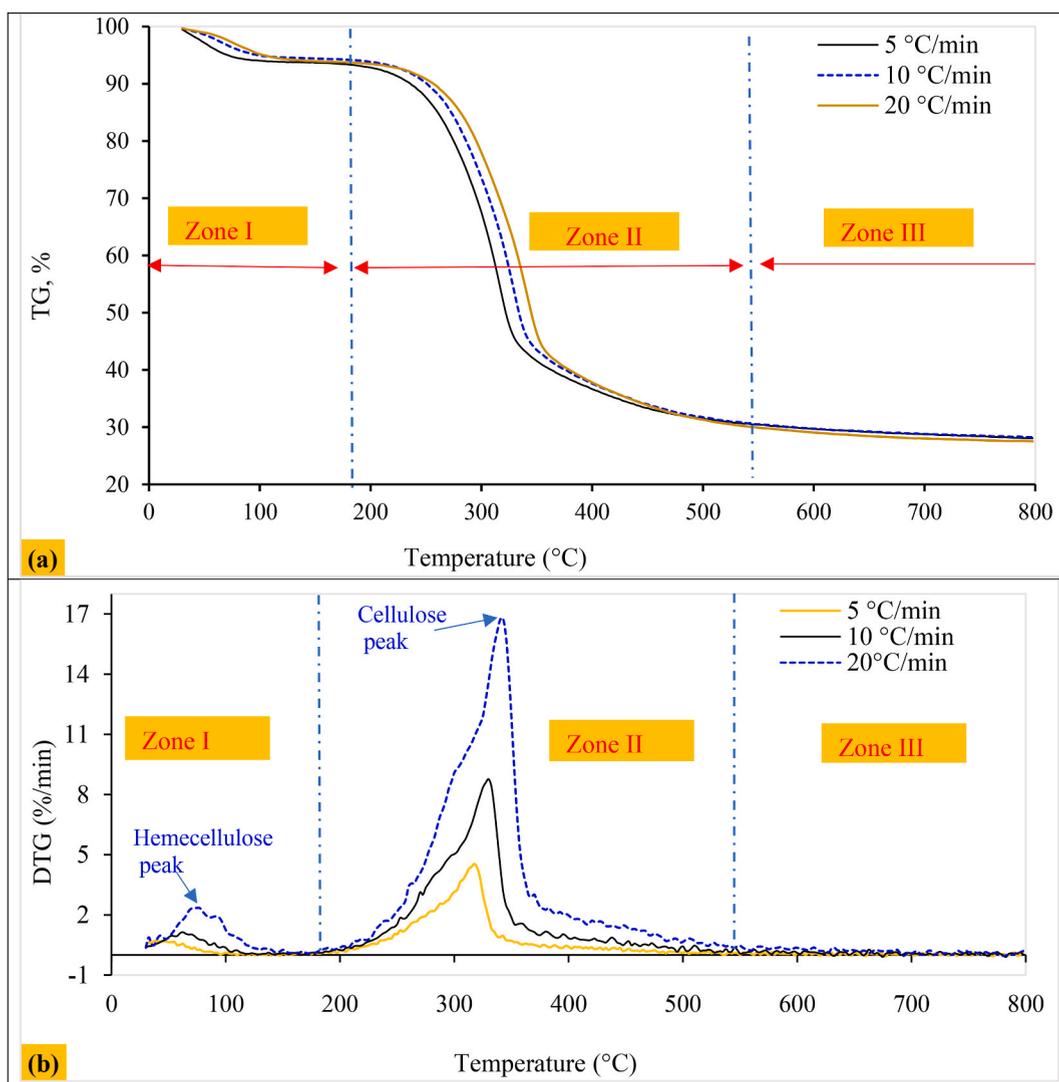


Fig. 3. Thermal decomposition profile of T₂ pellets under a nitrogen (N₂) atmosphere: (a) TG curves and (b) DTG curves.

185–341 °C temperature range, resulting in a mass loss of 44.14 %. Within this range, the DTG curve indicates a peak mass loss rate of 16.78 %/min at 341 °C due to the degradation of hemicelluloses and cellulose, along with the removal of volatile matter during a heating rate of 20 °C/min (Fig. 3(b)). These findings align with the research of Masnadi et al. [71], who investigated switchgrass pyrolysis characteristics and found devolatilization temperatures reaching 600 °C.

Furthermore, the second subsection occurs within the temperature range of 341–496 °C, resulting in a mass loss of 21.79 %. The DTG curve in this range indicates a mass loss rate of 0.29 mg/min at 496 °C, signifying the degradation of cellulose and some lignin. The overall mass loss during this stage amounts to approximately 66 %. This stage is commonly referred to as the active pyrolysis stage.

The third phase, termed the passive zone, commences at 496 °C and extends to 800 °C. For T₂ pellets, the mass loss during this stage is a mere 3.52 %, and minimal fluctuation is observed in the DTG curve throughout this period (Fig. 3(a)). This stage is likely attributed to the degradation of the remaining lignin and is commonly called the passive pyrolysis stage. Huang et al. [72] investigated the kinetic parameters of seven biomass types, including rice straw, rice hulls, corn leaves, coffee hulls, bamboo leaves, sugarcane bagasse, and sugarcane peel. They concluded that most of the biomass exhibited similar decomposition characteristics. The current study aligns with these findings. Examining the TG curve reveals that the degradation of T₂ is nearly complete at 600 °C. Ultimately, the residue remaining at 800 °C accounts for about 23.5 %, resembling the ash content in biomass.

Conversely, the TG/DTG curve levels off after the peak reaction, corresponding to the pyrolysis and combustion of lignin. This pattern aligns with the findings of other researchers [27,73]. In the existing literature, the thermal decomposition behavior of hemicellulose, cellulose, and lignin suggests degradation temperatures ranging from 220 to 315 °C, 315–400 °C, and 500–900 °C, respectively [109]. It is worth mentioning that the TG/DTA curves of the current biomass demonstrate a comparable trend to those of other biomass sources [74].

3.3. Effect of temperature on the pyrolysis behavior

The decomposition of biomass components is contingent on temperature and heating rates, influencing the pyrolysis behavior [75]. Analysis of the TG curve (Figs. 4 and 5) reveals that the mass loss of WSP is temperature-dependent, resulting in weight loss increasing in temperature. Both pellets found rapid weight loss at approximate temperatures between 160 and 560 °C (zone II), referred to as the active pyrolysis zone [76]. Conversely, the weight loss in the passive pyrolysis zones (I and III) was more moderate [77].

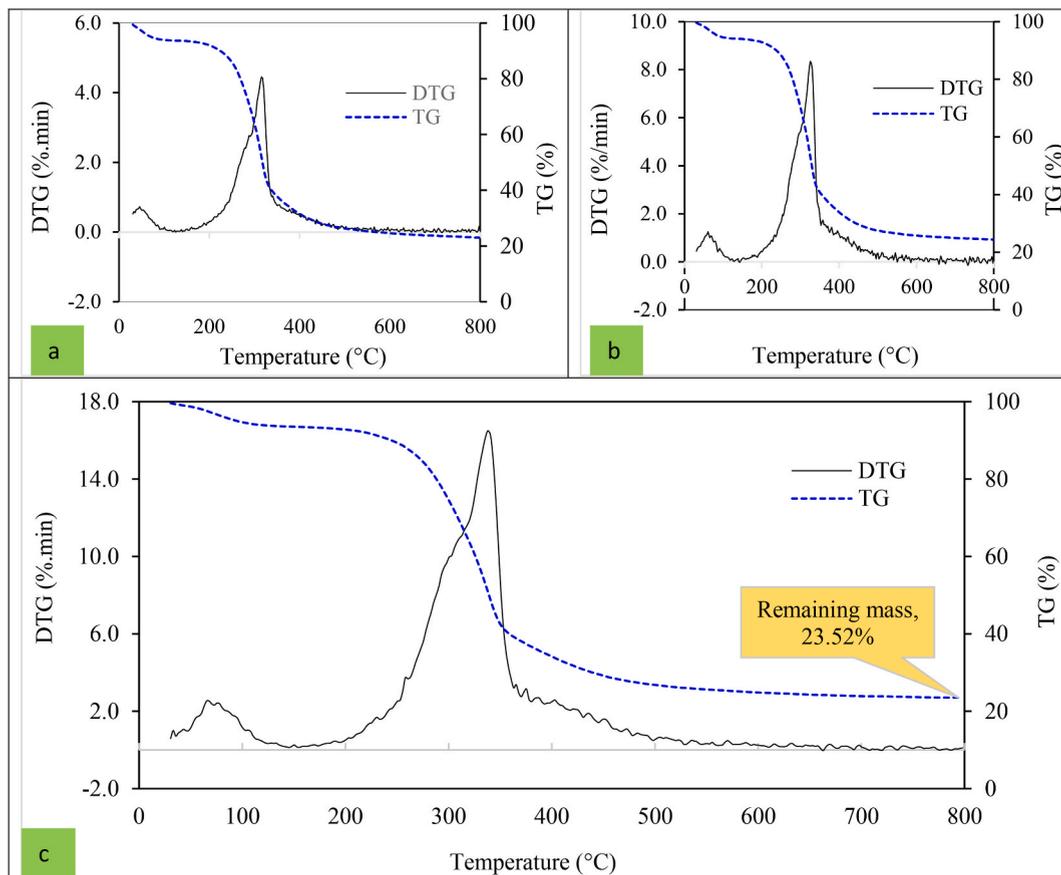


Fig. 4. TG and DTG curves for the pyrolysis of T₁ pellets at a constant heating rate: (a) 5 °C/min; (b) 10 °C/min, and (c) 20 °C/min.

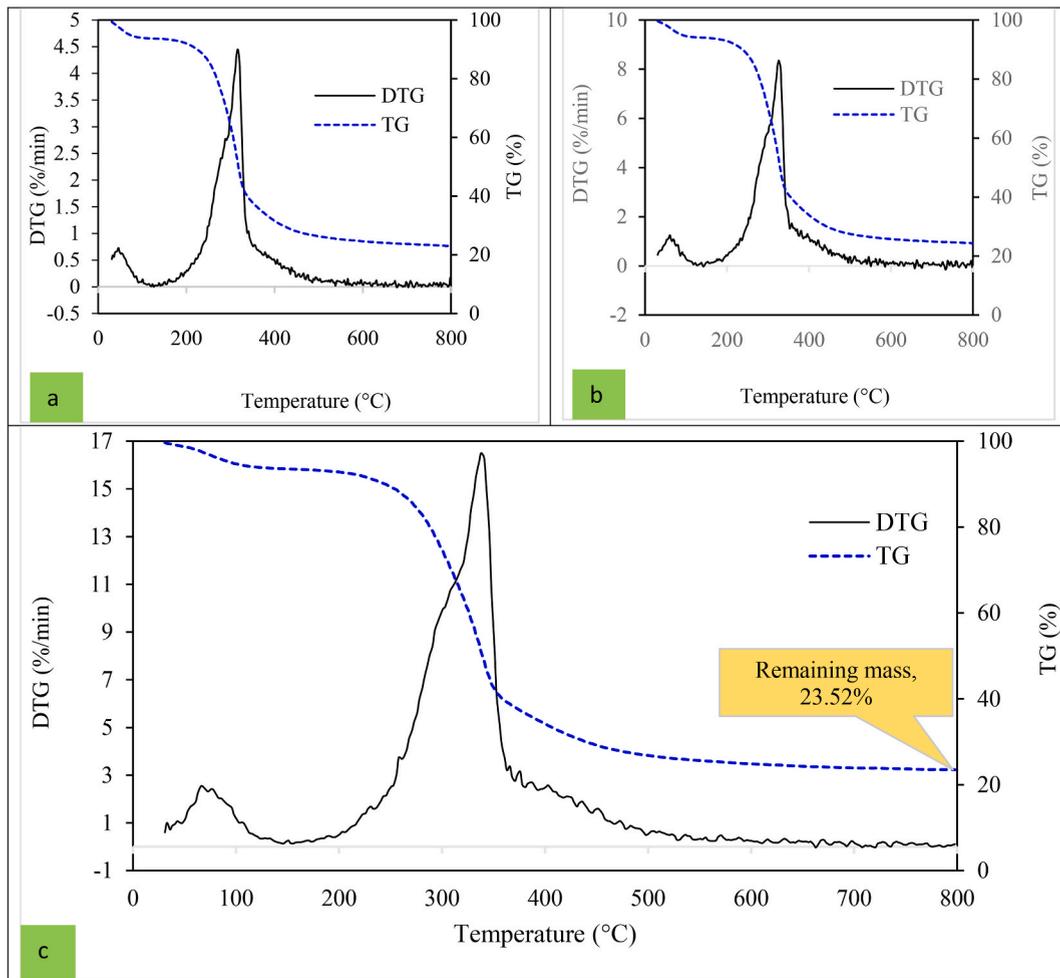


Fig. 5. TG and DTG profile for T₂ pellets pyrolysis at constant heating rate (a) 5 °C/min; (b) 10 °C/min and (c) 20 °C/min.

Due to the temperature application, the mass losses and reactions happened continuously. The temperature variation of different decomposition zones of I, II and III was 31~161 °C, 161~556 °C, and 556~800 °C, respectively, in the case of the T₁ treatment at all heating rates (Table 5). In addition, the mass losses of the T₁ pellet were 6.52, 68.07, and 2.05 % for the decomposition zone of I, II, and III, accordingly for a heating rate of 20 %/min (Table 6). The DTG profile fluctuation represents the component variation of the sample [78]. Considering that the DTG profile had identical peaks and several side peaks for all types of pellets with each heating rate, that

Table 5
Thermal decomposition temperature of the pellets.

Heating rate (°C/min)	Decomposition process temperature (°C)				
	Zone I (Drying)		Zone II (Devolatilization)		Zone III (Carbonization)
	Temperature range	Maximum temperature	1st step	2nd step	
T ₁ pallet					
20	31–161	68	161–341	341–556	556–800
10	31–161	66	161–332	332–556	556–800
5	31–161	61	161–318	318–556	556–800
T ₂ pallet					
20	31–185	78	185–341	341–496	496–800
10	31–186	53	185–331	331–496	496–800
5	31–187	60	185–321	321–496	496–800

Note.

Zone I = Mass losses due to the moisture evaporation.

Zone II = Mass losses devolatilization (i.e., volatile released and then burned, hemicellulose and cellulose).

Zone III = Mass losses carbonization (decompose of cellulose and char).

could be an elemental variation [79]. Fig. 4(c) shows that the most effective reaction rate was 16.28 %/min at 341 °C for a 20 °C/min heating rate. Therefore, the mass loss, decomposition zone and reaction rates varied with the temperature variation, which agrees with previous researchers [80,81].

In addition, for T₂, the weight loss was 7.05, 65.93, and 3.52 % in variation of temperatures 31–185 °C, 185–496 °C, and 496–800 °C, accordingly (Table 5). However, average weight loss (approximately 3.0 %) was negligible after 496 °C for both pellets regardless of heating rates, indicating that very little volatile stuff was present, while fast mass loss occurred between 158 and 553 °C. These findings are consistent with the literature of Singh et al. [69]; they used the garlic husk for the TGA investigation.

Alternatively, the highest peak represents the rapid maximum reaction rate (8.27 %/min), which happened approximately between the temperature of about 200–375 °C for a 10 %/min heating rate (Fig. 5(b)). These values align with findings from other studies that utilized agricultural residues, including castor residue, maize cob, linseed stalks, and rice straw [82]. In addition, the decomposition rate was slow in the first and third phases (passive pyrolysis zone). In contrast, the peak mass loss in the active zone for both pellets means temperatures influence pyrolysis rates. The results agreed with the other research [60] using rice straw, pine sawdust, and phoenix tree leave biomass.

3.4. Effect of heating rate on pyrolysis kinetics

The significance of the heating rate in the pyrolysis process is emphasized, as it affects the temperature ranges of the pyrolysis process, DTG peak temperatures, maximum decomposition rate, and final residual mass after the pyrolysis process [110]. Higher heating rates provide more thermal energy to overcome the temperature gradient within the biomass, resulting in enhanced conversion [83]. The impact of the heating rate at 5, 10, and 20 °C/min on the pyrolysis of WSP (T₁ and T₂) is shown in Tables 5 and 6. The results indicate that changes in heating rates from 5 to 20 °C/min did not affect temperature variation in the subdivision of TG-DTG profiles. However, the increase in heating rate shifted the peak temperature, leading to mass loss and maximum reaction rate changes, irrespective of treatment and heating rates. Asadieraghi and Daud [84] mention that rising heating speed accelerated the decomposition rate, which agrees with the current study.

Table 5 shows the T₁ pellets' pyrolysis behavior. The drying (zone I) temperatures were the same (from 31 to 151 °C) regardless of heating rates, but the peak reaction rate temperature (T_{peak}) was 68, 66, and 61 °C for 20, 10, and 5 °C/min heating rates, respectively. These findings were similar to those of Mishra and Mohanty [68], who observed an increased DTG peak temperature with a rise in heating rate. The maximum reaction rates (DTG_{max}) were 16.28, 8.55, and 4.41 %/min for heating rates of 20, 10, and 5 °C/min, respectively. The maximum reaction rates were 16.78, 8.38, and 4.34 %/min for 20, 10, and 5 °C/min heating rates. The findings align with Kirubakaran et al. [85], who suggested that a lower heating rate facilitates better heat transfer in biomass, reducing the remaining mass after pyrolysis.

Table 6 presents the pyrolysis behavior of T₂ pellets based on heating rates (5, 10, and 20 °C/min). In the initial zone (I), the mass loss was 6.13, 5.65, and 6.16 % for heating rates of 20, 10, and 5 °C/min, respectively. The maximum reaction rates were 16.78, 8.38, and 4.34 %/min for heating rates of 20, 10, and 5 °C/min. Similarly, the peak temperature varied as the heating rate increased from 5 to 20 °C/min. Conversely, the remaining mass was 23.52, 27.53, and 23.58 % for 20, 10, and 5 °C/min heating rates. Overall, the heating rates influence peak temperature, mass loss, and reaction rates, corroborated by the findings of Haykiri-Acma et al. [86] for rapeseed. These results align with other research, such as Singh et al. [69], who explored the pyrolytic behavior of banana leaves using TGA.

3.5. Effect of additive addition in the thermal pyrolysis of wheat straw pellets

Fig. 6 illustrates the TG profile of T₁ and T₂ pellets during pyrolysis. From the initial temperature up to approximately 200 °C, the TG curves for T₁ and T₂ exhibited considerable overlap. After 200 °C, it was seen that the mass loss of the T₂ pellet was high, but there

Table 6
Thermogravimetric (TG) data on weight change during decomposition of the pellets.

Heating rate (°C/min)	Weight loss (%)				Zone III (Carbonization)	Remaining weight, %
	Zone I (Drying)	Zone II (Devolatilization)				
		1st step	2nd step	Total loss		
T ₁ pellet						
20	6.52	44.67	23.26	68.07	2.05	23.5
10	5.97	45.13	22.57	67.66	2.02	24.33
5	6.55	46.74	21.59	67.41	2.14	22.98
T ₂ pellet						
20	7.05	44.14	21.79	65.93	3.52	23.5
10	6.93	49.06	16.37	65.43	3.31	24.33
5	7.74	52.77	12.9	65.67	3.61	22.98

Note.

Zone I = Mass losses due to the moisture evaporation.

Zone II = Mass losses devolatilization (i.e., volatile released and then burned, hemicellulose and cellulose).

Zone III = Mass losses carbonization (decompose of cellulose and char).

was no significant difference for all heating rates. Interestingly, the three heating rates followed the same trend, and the mass loss difference was about 5 %, which remained consistent with the original material (wheat straw pellet) properties. Therefore, the pyrolysis performance was not considerably affected by the additive blending. A similar observation was noted in the investigation conducted by [87], where they utilized rice husk, wheat straw, and a blend of both in pellet form.

The DTG curve illustrates the reaction rate, which varied due to volatile and fixed carbon content differences. For all heating rates, the DTG curves had identical peaks that might be changed in an elemental variation (Fig. 7). The small peak represents the slow decomposition rate as well as slow pyrolysis, while the highest peak represents the flash pyrolysis; the conversion rate accelerated when the temperature increased. Yu et al. [111] state that cellulose is primarily a concentrated product of biomass, indicating high reactivity of biomass. Also, moisture, hemicellulose and lignin decomposed slowly, which signifies slow pyrolysis and a lower reaction rate [88]. According to previous research through Link, Yrjas, and Hupa [112], ash content substantially impacts the conversion rate and makes it slower.

Interestingly, the conversion rates of T1 and T2 nearly overlapped, possibly due to the similar ash content. However, the conversion rate was slightly higher in T₂ than in the T₁ pellet. The high conversion rates for T₂ were probably a result of effective heat transfer facilitated by the additive (biochar) in the blended material, along with chemical interactions. Overall, additives mixed into wheat straw did not boost the conversion rate and could not indicate a synergistic occurrence.

3.6. Thermodynamic analysis

Thermodynamic parameters, including Enthalpy (ΔH), Gibbs free energy (ΔG), and Entropy (ΔS), were determined utilizing the activation energy values obtained from the kinetics model-based approach due to its precision in activation energy calculations. These parameters are crucial in designing, scaling, and optimizing the pyrolysis reactor and associated parameters [89]. The values for E_a and $\ln A$ were obtained from the NETZSCH program at 20 °C/min heating rates for determining the Entropy, Enthalpy, and Gibbs free energy, as depicted in Table 7.

Enthalpy (ΔH) is associated with the energy transfer among products and reactants in a thermochemical process, representing the heat absorbed or released under constant pressure [90]. In the context of T₂ samples, the activation energy (E_a) and enthalpy (ΔH)

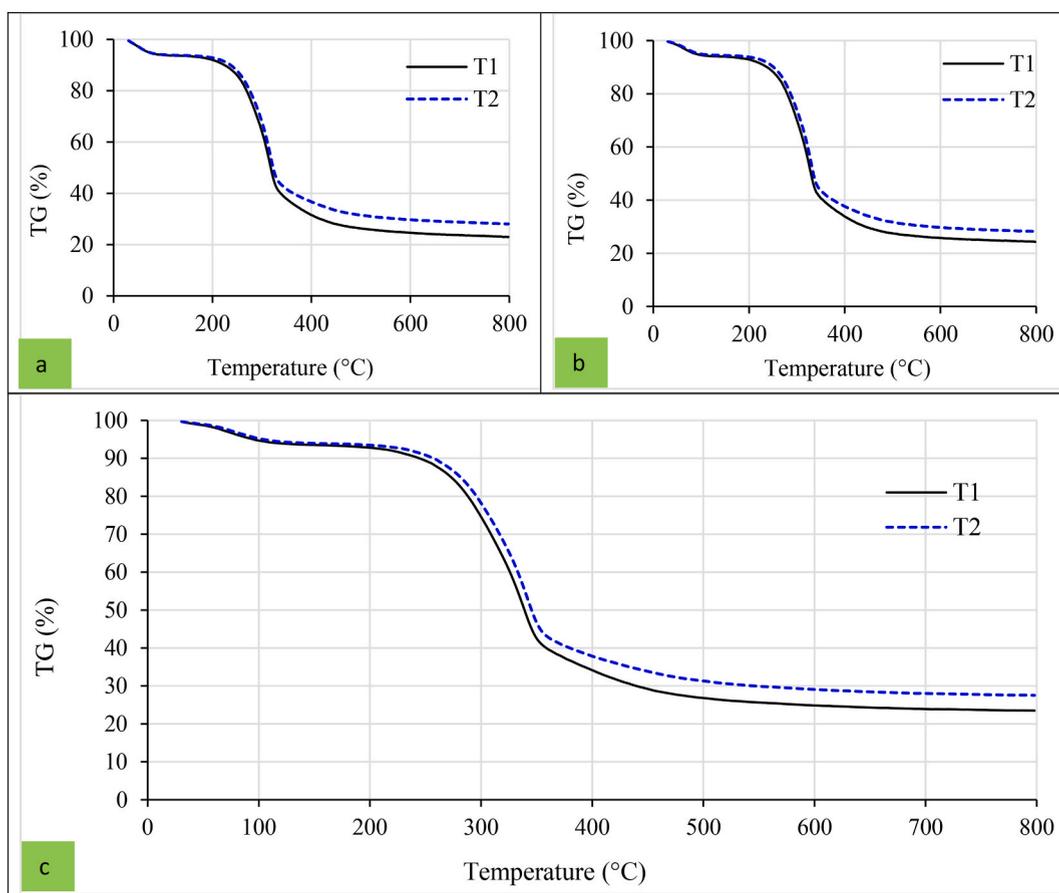


Fig. 6. Comparison of T₁ and T₂ pellet performance using DTG curves during pyrolysis at a consistent heating rate: (a) 5 °C/min; (b) 10 °C/min and (c) 20 °C/min.

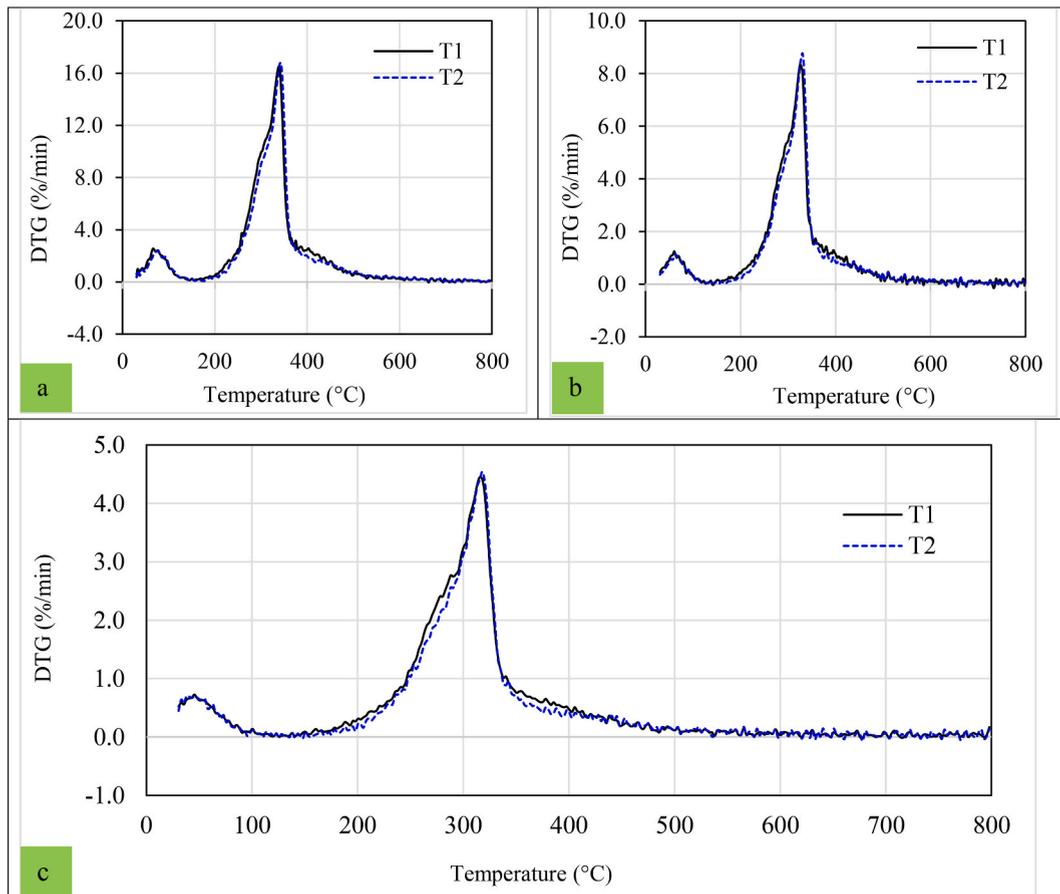


Fig. 7. Comparison of DTG curves during pyrolysis at a constant heating rate (a) 5 °C/min, (b) 10 °C/min, and (c) 20 °C/min for pellets T₁ and T₂.

demonstrate a low energy barrier, signifying the feasibility of the reaction under the specified parameters and a rapid initiation of chemical reactions. This correlation with low energy barrier outcomes aligns with findings from other published studies [35,56]. The ΔH values were 32.0–222.0 kJ/mol for T₂, alternatively 40~353.0 kJ/mol for the T₁ pellet. All positive values of ΔH in the thermal degradation of wheat straw pellets suggest an endothermic process. The low ΔH values facilitate the formation of the activated complex, indicating that the products can be easily generated with minimal energy input [91].

Gibbs free energy (ΔG) plays a crucial role in designing, scaling, and optimizing the pyrolysis reactor and related parameters [46]. The ΔG for the reaction zone (I ~ III) presented increasing trends in all pellets at 20 °C/min heating rate, which ranged between around 167.0 and 339.0 kJ/mol, which was near to the observation of Dhyani et al. [36].

In the case of T₁, the entropy (ΔS) exhibited negative values for reaction zones I and II but turned favorable for zone III, signaling an inconsistent reaction pattern. The heightened ΔS in T₁ implies a more robust reactivity during these stages, facilitating a quicker reaction rate for activation energy production. Conversely, for T₂, all entropy values were negative, indicating that the disorder in the

Table 7
Thermodynamic parameter for pyrolysis of WSP at 20 °C/min heating rate.

Items	Treatment					
	T ₁			T ₂		
	Zone I	Zone II	Zone III	Zone I	Zone II	Zone III
T _p , K	591.15	606.15	614.15	591.15	603.15	616.15
E _a , kJ/mol	45.015	136.038	358.110	37.298	132.868	227.105
A, (1/sec)	1.272E+2	3.910E+4	5.445E+14	38.359	2.449E+4	2.940E+9
ΔH , kJ/mol	40.100	130.998	353.004	32.383	127.853	221.982
ΔG , kJ/mol	169.347	234.782	338.955	167.522	233.445	270.042
ΔS , kJ/mol.K	-0.219	-0.171	0.023	-0.229	-0.175	-0.078

Note: determined by model – based e by model-based methods in the previous study [50].

generated products from bond dissociations was lower than in the initial blend samples. The negative (low) entropy (ΔS) value suggests that the pyrolysis of WSP biomass undergoes fewer physical and chemical changes, approaching thermodynamic equilibrium. The introduction of blends (T_2) significantly impacted the kinetic triplets compared to the T_1 sample, and the blending of feedstocks induced alterations in the thermodynamic parameters. A similar trend was observed by [92].

3.7. Characteristics of reactivity

The evaluation of biomass reactivity entailed the examination of several indicators, encompassing initial and final devolatilization temperatures (T_i , T_f); average devolatilization rate (R_a^{dev}); peak temperature (T_p); maximum decomposition rate (DTG_{max}); reactivity index (R_M); pyrolysis factor (P_f). The results for these indicators, considering three heating rates and two pellet types in two stages of the devolatilization process, are delineated in Table 8.

In the initial phase, the average devolatilization rate (R_a^{dev}) at T_2 was higher than at T_1 , indicating accelerated decomposition at T_1 attributable to lower temperatures and heightened reactivity. The elevation of the heating rate from 5 °C/min to 20 °C/min resulted in an increased peak temperature (T_p) and the corresponding maximum mass loss rate (DTG_{max}), likely stemming from reduced residence time and insufficient heat transfer to the particle center, thereby slowing down thermal degradation. This observed trend aligns with previous findings concerning wheat straw and corn cobs [93].

The reactivity index (R_M) serves as a key metric for assessing the thermal stability of biomass. In the case of the T_1 pellet, which comprises solely wheat straw (100 %) without any binding materials, its lower bulk density renders it susceptible to easier burn, especially when subjected to a higher heating rate, such as an increase from 5 °C/min to 20 °C/min. Conversely, the T_2 pellet, made from a composition of 70 % wheat straw combined with binding materials (10 % Bentonite clay, 10 % Sawdust, and 10 % Biochar) exhibits higher bulk density. Consequently, the T_2 pellet demonstrates excellent stability, though it manifests an inconsistent reactivity index when subjected to the same heating rate escalation from 5 °C/min to 20 °C/min.

The pyrolysis factor (P_f), is essential in assessing fuel behavior during volatile removal, and exhibits an increase with higher heating rates and reactivity, as evidenced by its values. Zhang et al. [94] have extensively documented interdependence among reactivity features. The determination of the pyrolysis factor (P_f), depends on various factors including biomass type, heating rate, residence time, devolatilization rate, and temperatures, among others. As outlined in Table 2, the T_1 pellet is of poorer quality than the T_2 pellet in terms of manufacturing materials composition and bulk density. The T_1 pellet consistently increases with higher heating rates, as shown by the escalation from 5 °C/min to 20 °C/min. Conversely, the T_2 pellet deviates from this trend in the first step of burning, although it aligns with the rising pattern in the second step following a heating rate increase. Overall, the P_f values are notably higher in the first step compared to the second step across all heating rates. The findings imply that an escalation in heating rate corresponds to an augmentation in reactivity. Similar findings of a direct correlation between reactivity and heating rate were observed for various residues [95].

4. Conclusion and outlook

In this research, the pyrolytic characteristics of wheat straw pellets (WSP) were examined using thermogravimetric analyses (TGA) conducted in a nitrogen atmosphere across a temperature range of 31–800 °C, employing varying heating rates (5, 10, and 20 °C/min). Two WSP types were examined: T_1 (100 % wheat straw) and T_2 (70 % wheat straw, 10 % sawdust, 10 % bentonite clay, and 10 % biochar). The research revealed valuable insights.

- Higher volatile matter content and calorific value in WSP compared to raw materials.
- There are three distinct degradation zones: drying, devolatilization (the most significant mass loss—65 % between 150 and 550 °C), and carbonization.
- Devolatilization analysis: extensive examination, particularly the first two steps responsible for the significant mass loss.
- Heating rate influence: shift in thermogravimetric profiles to higher temperatures with faster heating.
- Temperature vs. Heating Rate: Temperature significantly impacted mass loss and reaction rates, while heating rate had a minor effect.
- Thermodynamic properties: suggested equilibrium reactions during pyrolysis for T_1 and T_2 pellets.
- Reactivity index: The reactivity index (R_M) increased with the progressive rise in heating rate from 5 °C/min to 20 °C/min in the T_1 pellet. Conversely, the reactivity index ($R_{cap R sub cap M}$) observed in the T_2 pellets appeared inconsistent under similar conditions and might be more stable.

These findings provide a deeper understanding of WSP thermal behavior and the influence of composition and heating conditions. This knowledge is essential for optimizing the utilization of wheat straw biomass in combustion and pyrolysis processes for energy production and resource recovery.

Future research directions could involve.

- Applying the model-based methods to predict thermal behavior under different conditions.
- Investigating the synergistic effects of additives in T_2 pellets on decomposition.
- Exploring the potential for using the results for designing and optimizing biomass conversion technologies.

Table 8

Indicators of the decomposition reactivity of the pellets.

Heating rate (°C/min)	Temperature (°C)			Weight loss rate (%/min)				R_M (%/min.°C)	Pyrolysis factor (P_f) [*] 10 ⁷	
	T_i	T_f	T_p	1st step	2nd step	R_a^{dev}	DTG_{max}		1st step	2nd step
T₂ pellets										
20	185	550	343	0.26	0.37	0.315	16.78	0.0489	2.3177	3.2982
10	185	550	333	0.17	0.25	0.21	8.77	0.0133	0.4001	0.5884
5	185	550	320	0.12	0.09	0.105	4.43	0.0274	0.5591	0.4193
T₁ pellets										
20	161	556	341	0.19	0.28	0.235	16.28	0.0477	2.1463	3.1629
10	161	556	332	0.14	0.12	0.13	8.02	0.0242	0.7791	0.6678
5	161	556	318	0.1	0	0.05	4.11	0.0129	0.2851	0.0

Note: DTG_{max} = Maximum weight loss rate of devolatilization; T_i = Initial temperature of devolatilization.

T_f = Final Temperature of devolatilization; T_p = Peak temperature of devolatilization.

RM = Reactivity index; R_a^{dev} = Average devolatilization rate.

P_f = Pyrolysis factor.

CRedit authorship contribution statement

Bidhan Nath: Conceptualization, Formal analysis, Investigation, Validation, Writing – original draft. **Guangnan Chen:** Resources, Supervision, Writing – review & editing. **Les Bowtell:** Project administration, Supervision, Writing – review & editing. **Elizabeth Graham:** Visualization, Formal analysis, Validation.

Institutional review board statement

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Data availability statement

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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