

Fuel Characterisation of the Physicochemical, Thermal and Kinetic Properties of Corn Cob Biomass Wastes for Potential Energy Recovery

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Abstract

This study presents insights into the solid biofuel properties of corn cob biomass (CCB) wastes for sustainable energy recovery. The physicochemical, thermal, and kinetic properties of CCB were characterised through ultimate, proximate, heating value, and thermogravimetric (TGA) analyses. Results showed that CCB contains high carbon (41.88 wt.%), hydrogen (6.33 wt.%), volatile matter (68.21 wt.%), and higher heating (15.70 wt.%) values for potential energy recovery. However, the high ash (16.56 wt.%) content could pose bed agglomeration, fouling, and sintering problems during high-temperature conversion. Thermal analysis resulted in 55.84%–59.51% loss of mass and residual mass of 40.49%–44.17%. Kinetic analyses revealed that CCB is highly reactive as characterised by the average activation energy, $E_a = 134.58$ kJ/mol and pre-exponential factor, $k_0 = 2.53 \times 10^{08}$ /min. In conclusion, CCB is a potentially practical feedstock for sustainable energy recovery through thermochemical conversion.

Keywords: Biofuel characterisation; Physicochemical properties; Thermokinetics; Corn cobs.

1. Introduction

The valorisation of agricultural biomass wastes is gaining significant traction worldwide. Over the years, the quest to reduce the overdependence of global economies on fossil-based fuels such as petroleum and coal has prompted the transition to cleaner alternatives [1]. The utilization of agricultural biomass wastes for energy, chemicals, and fuels is considered carbon neutral with potential benefits for human health, safety, and the environment [2-3]. Researchers posit that the transition to clean energy technologies such as biomass will enhance the diversification of the world's energy mix and reduce the long term impacts of global warming and climate change [4-5]. Therefore, the potential of valorising various agricultural biomass waste streams such as wheat straw [6-7], soybean stalk/straw [8-9], oil palm wastes [10-11], rice

husks and other biomass into biofuels, bioenergy and biomaterials have been widely explored in the literature.

However, the valorisation of agricultural biomass wastes requires analyses of their intrinsic physical, chemical, thermal, kinetic and thermodynamic properties [12-13]. Hence, prospective agricultural biomass wastes are typically subjected to extensive characterisation and assessment through ultimate, proximate, and calorific analyses to determine the elemental (CHNSO-carbon, hydrogen, nitrogen, sulphur and oxygen contents in wt.%), chemical fuel properties (moisture, volatiles, ash, and fixed carbon), and higher heating value (HHV) [14]. The thermal and kinetic analyses of prospective agricultural biomass wastes are also examined through thermogravimetric analysis (TGA) [15]. TGA provides comprehensive data on the thermal behaviour, degradation mechanism, and temperature profile characteristics (TPCs) of prospective biomass feedstock [16-17]. Based on literature reviews, the valorisation of agricultural biomass wastes is typically accomplished by biochemical and thermochemical technologies such as digestion, fermentation, torrefaction, pyrolysis, gasification, and combustion [18-19]. However, lack of data on the outlined characteristics of potential feedstock biomass could severely hamper the design, development, and scale-up of future bioenergy and biofuel conversion systems.

Therefore, this study seeks to characterise the solid biofuel properties of corn cob biomass (CCB) wastes through physicochemical, thermal, and kinetic analysis for sustainable energy recovery. Previous studies in the literature have examined the biochemical conversion and pre-treatment of CCB through hydrolysis, saccharification, ultrasonic treatment, and extraction [20-22]. Other studies have examined the thermochemical conversion of CCB through gasification [23-24], and combustion [25-27]. Therefore, it is envisaged that this study will provide insights into the biofuel properties of CCB and present comprehensive data for sustainable energy recovery through pyrolysis.

2. Experimental

2.1. Physicochemical analysis

The physicochemical properties of CCB were elucidated through ultimate, proximate, and calorific analyses to determine its elemental, chemical fuel, and heating values. The ultimate analysis was carried out using an elemental analyser (Model: vario MACROCUBE, Germany) to determine the composition of carbon, hydrogen, nitrogen, and sulphur contents based on the ASTM D5291-16 technique. The proximate analysis was performed by thermogravimetric (TG) analysis to determine the moisture (M), volatile matter (VM), ash (AC) and fixed carbon (FC). The TG Analyser (Model: Shimadzu TG-50, Japan) was employed to determine the M and VM by heating a known mass of the sample at 105°C and 950°C in nitrogen gas (N₂) at a flow rate of 100 mL/min for a hold time of 11 minutes. The ash (A) was determined in an air atmosphere at 950°C. The calorific analysis was performed using an isoperibol oxygen bomb calorimeter (IKA C200, USA) located at the School of Mechanical Engineering, Universiti Teknologi Malaysia (UTM, Skudai Malaysia) to compute the higher heating value (HHV). Each test was performed at least twice to ensure accuracy and precision of the results presented in Table 1.

Table 1. Physicochemical fuel properties of CCB

Analysis	Fuel property	Symbol (Unit)	Corn Cob Biomass
Ultimate	Carbon	C (wt.%)	41.88
	Hydrogen	H (wt.%)	6.33
	Nitrogen	N (wt.%)	0.79
	Sulphur	S (wt.%)	0.14
	Oxygen	O (wt.%)	50.86
Proximate	Moisture	M (wt.%)	9.44
	Volatile Matter	VM (wt.%)	68.21
	Ash	A (wt.%)	16.56
	Fixed Carbon	FC (wt.%)	5.79
Calorific	Higher Heating Value	HHV (MJ/kg)	15.70

2.2. Thermal analysis

The thermal properties of CCB were examined by thermogravimetric (TG) analysis based on the non-isothermal and multiple heating rate programs of the TG analyser (Model: Shimadzu TG-50, Japan). For each TGA experiment, approximately 13.5 mg of CCB (particle size below 250 μm) was weighed in an alumina crucible before heating from 25 $^{\circ}\text{C}$ to 700 $^{\circ}\text{C}$ based on the multiple heating rates of 10 $^{\circ}\text{C}/\text{min}$, 20 $^{\circ}\text{C}/\text{min}$, 30 $^{\circ}\text{C}/\text{min}$ under nitrogen (N_2) gas flow rates of 100 mL/min. The aim of the process was to simulate the non-isothermal thermal degradation of CCB under the non-oxidative conditions typical of pyrolysis.

On completion, the furnace of the TG analyser was cooled to room temperature using an automatic air blower. The TG data was then analysed using the thermal analysis software (Shimadzu TA-60WS) to determine the mass loss and derivative of the mass loss, which were plotted against temperature ($^{\circ}\text{C}$) to deduce the TG (%) and DTG (%/min) plots in Figures 1 and 2. Subsequently, the temperature degradation profiles for each heating rate during TGA were determined to understand the thermal behaviour and degradation mechanism. The temperature degradation profiles deduced from the TG plots in this study were; Onset temperature (T_o), Midpoint temperature (T_m), Endpoint temperature (T_e), Loss of Mass (L_M) and Residual Mass (R_M). However, the temperature degradation profiles deduced from DTG were; peak drying temperature (T_{DRY}), peak devolatilization temperature I (T_{DV1}) and peak devolatilization temperature II (T_{DV2}).

2.3. Kinetic analysis

The kinetic parameters for the non-oxidative thermal degradation of CCB were examined based on the Kissinger kinetic model (KKM). In principle, the governing equations of KKM are based on the peak decomposition temperatures derived from the DTG plots of multiple heating-rate thermal degradation of materials such as the CCB examined this study. The central concept is derived from the Arrhenius relation for thermally degrading carbon materials described as follows;

$$k(T) = k_o \exp\left(-\frac{E_a}{RT}\right) \quad (1)$$

From Eq. 1, the terms $k(T)$, k_o (/min), E_a (kJ/mol), and R (J/mol K) represent the temperature-based rate constant, pre-exponential factor, activation energy, and ideal gas constant, respectively. Hence, the thermal decomposition of CCB based on the multiple heating rates, non-oxidative, and non-isothermal decomposition could be described as;

$$\frac{d\alpha}{dT} = \frac{k_o}{\beta} \exp\left(-\frac{E_a}{RT}\right) f(\alpha) \quad (2)$$

From Eq. 2, the term β symbolises the heating rates (10, 20, and 30 $^{\circ}\text{C}/\text{min}$), whereas the reaction function for the CCB thermal decomposition model is described by $f(\alpha)$. Hence, the governing equations for the KKM can be subsequently deduced by separating and integrating the variables in Eq. 2 to derive the relation in Eq. 3;

$$\ln\left(\frac{\beta}{T_x^2}\right) = \ln\left(\frac{k_o R}{E_a}\right) - \ln\left(\frac{E_a}{RT_x}\right) \quad (3)$$

Consequently, the peak decomposition terms namely; peak drying temperature (T_{DRY}), peak devolatilization temperature I (T_{DV1}), and peak devolatilization temperature II (T_{DV2}) derived from the DTG plots were substituted for T_x in Eq. 3. The kinetic parameters; activation energy (E_a) and pre-exponential factor (k_o) for the drying (T_{DRY}) and devolatilization (T_{DV1} and T_{DV2}) of CCB during TGA were deduced.

3. Results and discussion

3.1. Physicochemical properties

The physicochemical properties of potential biomass are crucial to determining its potential for bioenergy recovery [28]. Numerous studies have employed various techniques to examine the solid bioenergy and biofuel properties of different biomass feedstock [29]. In this study, the physicochemical properties of corn cob biomass (CCB) are examined based on ultimate,

proximate, and calorific analyses. Table 1 shows the elemental composition, chemical fuel properties, and higher heating value of CCB in as received (a.r.) basis.

The results indicate that CCB contains carbon, hydrogen, nitrogen, sulphur, oxygen, along with moisture, volatile matter, ash, fixed carbon, and higher heating value in various proportions. The high carbon and hydrogen content observed in CCB indicates it has good potential for energy recovery, despite the high oxygen content. Typically, high oxygen content could result in over oxidation of biomass feedstock during thermal conversion producing high ash and mineral matter along with low heating value flue gases [19]. However, the low moisture content (< 10%) indicates CCB does not require additional treatment or conditioning such as drying, which is an energy-intensive and costly process during biomass energy recovery [30]. The volatile matter was also found to be sufficiently high (> 65%), which indicates CCB has high condensable and non-condensable fractions, which are crucial to enhancing product yield and distribution during biomass conversion. Furthermore, high VM ensures good ignitability and thermal conversion particularly for gasification and combustion [31]. The ash content was considerably high (> 5%), whereas the fixed carbon was within the acceptable limits [13]. The high ash content of CCB could present operational and technical challenges due to bed materials agglomeration, fouling, or sintering during thermochemical conversion [32-33]. Lastly, the heating value of CCB (HHV = 15.70 MJ/kg) was found to be within the range from 14 MJ/kg to 20 MJ/kg typically reported for biomass [34]. Based on its physicochemical properties CCB is a potentially good feedstock for energy recovery through thermochemical conversion.

3.2. Thermal properties

The thermal properties of CCB were analysed by thermogravimetric analysis (TGA) to determine its thermal behaviour, decomposition mechanism, and temperature degradation profiles. The TG and derivative TG plots for the non-oxidative, non-isothermal, and multiple heating rate thermal decompositions of CCB are shown in Figures 1 and 2. The TG plots for CCB are characteristic of thermally decomposing carbonaceous materials with progressive loss of mass observed during TGA. The plots indicate that the non-isothermal incremental change in temperatures from 25°C to 700°C along with the variation of the heating rates from 10°C/min to 30°C/min significantly influenced the thermal decomposition of CCB during TGA.

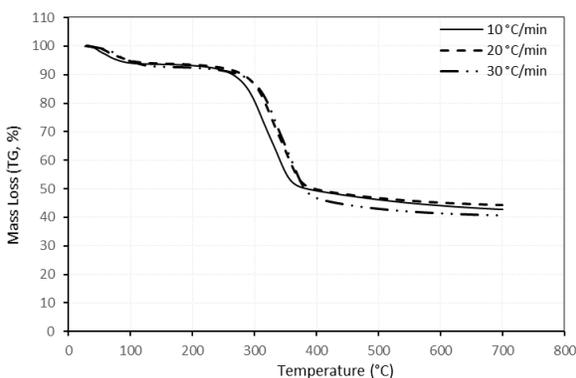


Figure 1. TG plots for thermal decomposition of CCB

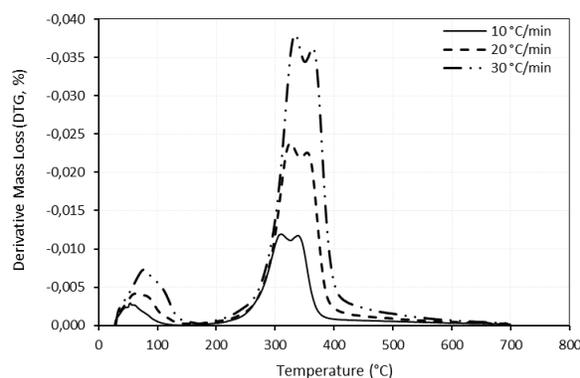


Figure 2. DTG plots for thermal decomposition of CCB

As observed, the TG plots shifted from right to left with increasing heating rate during TGA. According to numerous authors in the literature [30, 35-36], the shifts can be ascribed to the thermal-time lag that causes delayed degradation of biomass decomposition and transforms the degradation temperature profiles. Hence, the extent of the shifts on the TG plots and the thermal properties of CCB were examined as presented in Table 2.

Table 2. TG-Degradation temperatures for CCB

Heating Rate (°C/min)	Onset temperature (T_o , °C)	Midpoint temperature (T_m , °C)	Endpoint temperature (T_e , °C)	Loss of mass (L_M , %)	Residual mass (R_M , %)
10	262.15	317.22	372.72	57.23	42.77
20	278.42	331.71	385.81	55.84	44.17
30	287.70	338.90	395.24	59.51	40.49

As observed, the TG based degradation temperature profiles; onset (T_o), midpoint (T_m), and endpoint (T_e) temperatures for CCB increased with increasing heating rates from 10°C/min to 30°C/min during TGA. The onset (T_o) temperatures increased from 262.15°C to 287.70°C indicating the ignition of CCB increased by 25.55°C with an increase in the heating rates. Likewise, the midpoint (T_m) temperatures increased from 317.22°C to 338.90°C signifying the rise in the heating rates resulted in a 21.68°C increase in the T_m . Lastly, the endpoint (T_e) temperatures also increased from 372.72°C to 395.24°C or by 22.52°C with rising heating rates from 10°C/min to 30°C/min during TGA. Hence, it can be reasonably surmised that the variation in the heating rates exerted a higher influence on the onset (T_o), compared to the endpoint (T_e), and lastly midpoint (T_m) temperatures in decreasing order. On the other hand, the loss of mass was in the range of 55.84% to 59.51%, whereas the residual mass was from 40.49% to 44.17%. On average the onset temperature (T_o), midpoint temperature (T_m), and endpoint temperature (T_e), loss of mass (L_M) and residual mass (R_M) for CCB degradation are; 276.09 °C, 329.28 °C, 384.59 °C, 57.52% and 42.48%.

The analysis of the derivative TG or DTG plots was examined to understand the decomposition mechanism of CCB during TGA. Figure 2 shows the DTG plots for CCB at different heating rates based on non-isothermal heating under non-oxidative conditions.

The DTG plots for each heating rate displayed two sets of endothermic peaks. The first set of small-sized peaks can be observed from 25 °C to 150°C, which signifies the loss of mass due to surface-bound moisture (drying) and low molecular weight (partial devolatilization). However, the second set or peaks can be observed between 150°C and 425°C, which signifies the mass loss due to holocellulose and lignin degradation during TGA [37-39]. Furthermore, the mass loss in this range is typically ascribed to the loss of volatile matter, hence the term devolatilization. Further analysis of the devolatilization peaks revealed the presence of two humps denoted as peak devolatilization temperatures I (T_{DV1}) and II (T_{DV2}) for the heating rates. Table 3 presents the characteristic peak temperatures for drying and devolatilization at T_{DV1} and T_{DV2} .

Table 3. DTG-Degradation temperatures for CCB

Heating Rate (°C/min)	Peak drying temperature (T_{DRY})	Peak devolatilization temperature I (T_{DV1})	Peak devolatilization temperature II (T_{DV2})
10	54.53	310.90	339.45
20	62.59	326.31	353.87
30	77.66	333.58	363.49

Similarly, the findings indicate that higher heating rates shifted the degradation parameters to higher temperatures during TGA. From Table 3, it can be observed that peak drying temperature (T_{DRY}) increased by 23.13°C from 54.53°C to 77.66°C with the increase in heating rate from 10°C/min to 30°C/min. Likewise, the peak devolatilization temperature I (T_{DV1}) increased but by 22.68°C, whereas the peak devolatilization temperature II (T_{DV2}) was by 24.04°C from 310.90°C and 339.45°C, respectively. Typically, the mass loss at the peak devolatilization temperatures I (T_{DV1}) and II (T_{DV2}) are attributed to the thermal degradation of hemicellulose and cellulose during TGA [38-39].

3.3. Kinetic properties

The Kissinger kinetic model (KKM) was employed to examine the kinetic parameters; activation energy (E_a) and pre-exponential factor (k_o) for CCB based on the drying (T_{DRY}) and devolatilization (T_{DV1} and T_{DV2}) stages during TGA. Based on Eq 1-3, the terms $\ln\left(\frac{\beta}{T_x^2}\right)$ were plotted against $\left(\frac{1}{T_x}\right)$, whereby the values of E_a and k_o were determined from the slope and intercept of the plots, respectively. Therefore, the kinetic plots for the drying and devolatilization stages (T_{DV1} and T_{DV2}) are presented in Figures 3-5.

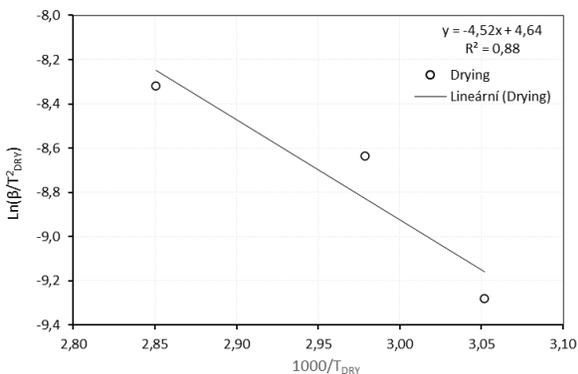


Figure 3. Kissinger kinetic plots for drying of CCB

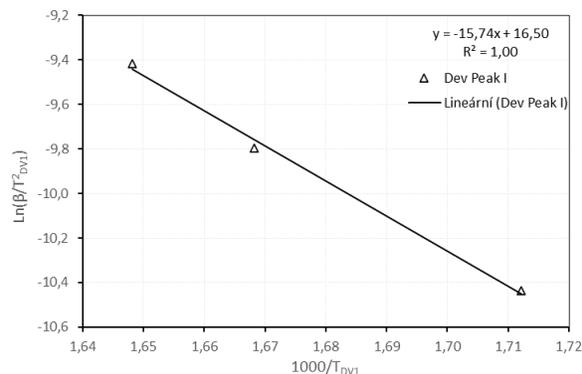


Figure 4. Kissinger kinetic plots for CCB devolatilization at T_{DV1}

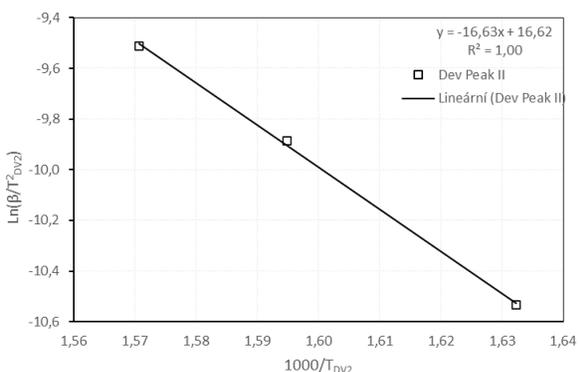


Figure 5. Kissinger kinetic plots for CCB devolatilization at T_{DV2}

Based on the slope and intercept of the plots in Figures 3-5, the kinetic parameters for the thermal degradation of CCB under non-oxidative and non-isothermal conditions were examined. Therefore, the activation energy required for drying CCB was computed as $E_a = 37.61$ kJ/mol, with the pre-exponential factor, $k_o = 4.71 \times 10^{02}$ /min. For the devolatilization at the peak decomposition temperature 1 (T_{DV1}), the computed activation energy was $E_a = 130.89$ kJ/mol with the pre-exponential factor, $k_o = 2.32 \times 10^{08}$ /min. Lastly, for devolatilization at the peak

decomposition temperature 2 (T_{DV2}), the activation energy was computed as $E_a = 138.27$ kJ/mol with the pre-exponential factor, $k_o = 2.75 \times 10^{08}$ /min.

On average the E_a and k_o for the devolatilization process was 134.58 kJ/mol and 2.53×10^{08} /min. In comparison, the kinetic parameters of CCB are lower than oil palm empty fruit bunch pellets (144.30 – 146.63 kJ/mol) [28], cornstalk (206.40 kJ/mol), oak (236.20 kJ/mol), and sawdust (232.60 kJ/mol) [40] as reported in the literature. Hence, the kinetic parameters indicate that CCB is highly reactive despite the mass loss (55.84% – 59.51%) and residual mass (40.49% – 44.17%) during TGA.

4. Conclusion

The study examined the physicochemical, thermal, and kinetic properties of corn cob biomass (CCB) wastes as potential solid biofuel for sustainable energy recovery. The characterisation analyses revealed that CCB has the requisite fuel properties such as high carbon, hydrogen, volatile matter, and higher heating values for energy recovery through thermochemical conversion. However, the high ash content could pose potential problems such as bed materials agglomeration, fouling, and sintering during high-temperature conversion. The thermal and kinetic analyses revealed that CCB is highly reactive with the average values of $E_a =$

134.58 kJ/mol and $k_o = 2.53 \times 10^{08}$ /min deduced for the devolatilization process and the mass loss (55.84% – 59.51%) and residual mass (40.49% – 44.17%) during TGA. Overall, the findings indicate that CCB is a potentially viable feedstock for sustainable energy recovery through thermochemical conversion.

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