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Carbon dioxide-assisted Torrefaction of Maize Cobs by Thermogravimetry: Product Yield and Energy Recovery Potentials

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Abstract – The objective of this study is to examine the potential product yields and energy recovery of maize cobs (MC) through carbon dioxide-assisted torrefaction using thermogravimetric analysis (TGA). The CO_2-assisted torrefaction of MC was performed from 240 °C to 300 °C (Δ 30 °C) for the residence time of 30 minutes based on the selected non-isothermal/isothermal heating programme of the TGA. Furthermore, the physicochemical, microstructure and mineral characteristics of MC were examined. The results showed that the CO_2-torrefaction of MC resulted in mass loss (ML) ranging from 18.45% to 55.17%, which resulted in the mass yield (MY) ranging from 81.55% to 44.83%. The HHV of the solid product was in the range from 22.55 MJ/kg to 26 MJ/kg, which indicates the CO_2-torrefaction process enhanced the energy content of MC by 40% – 60%. In conclusion, the findings showed that the CO_2 torrefaction is a practical, sustainable, and cost-effective approach for the valorisation of MC into a clean solid biofuel for enhanced energy recovery.

Keywords: Carbon dioxide, Torrefaction, Maize Cobs, Thermogravimetry, Energy Recovery

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Introduction

The transition from fossil-based fuels to cleaner energy sources is considered the panacea to the twin scourges of global warming and climate change that currently afflicts humanity [1]. Biomass is considered a carbon-neutral source of renewable and sustainable energy for the future [2]. Furthermore, analysts posit that the development, adoption and integration of biomass energy technologies could provide affordable and clean energy for the world and provide a framework for the action against climate change [3]. Given the energy and environmental benefits, numerous researchers around the globe have sort to examine and highlight the potentials of various biomass such as agricultural and forestry wastes as candidate feedstock for biobased energy recovery and utilisation [4]. However, current strategies for disposing and managing agroforestry wastes consist of open-pit dumping, landfilling or combustion that pose grave risks to human health, safety and the environment [5, 6]. Hence, it is envisioned that agroforestry waste valorisation could solve waste management and disposal problems and provide accessible, affordable, and clean energy.

The annual cultivation of Indian corn otherwise called maize (*Zea mays L.*) around the world yields significant amounts of stover, which comprises the leaves, stems and cob based wastes [7, 8]. Typically, these wastes account for over 50% of the weight of the plant, and as such present significant disposal and management challenges [9]. Presently, maize stover is utilised as organic manure, mulching material, livestock feeds, and boiler fuel [10]. Other strategies include landfilling, dumping or open-air burning, which are inefficient, unsustainable, and costly approaches to addressing the problems of corn stover [7]. Consequently, large fractions of these wastes remain underutilised against the backdrop of growing energy demand, particularly in developing countries. Furthermore, the inefficiency of the outlined challenges typically results in the emission of large quantities of greenhouse gases (GHG) such as carbon dioxide (CO_2), which are hazardous to humans and the environment.

In contrast, the lignocellulosic nature of the corn stover related wastes such as maize cobs (MC) present opportunities for effective utilisation as renewable raw materials to produce clean energy [11, 12]. One promising approach for the valorisation of MC is the process of torrefaction into solid biochar and biofuels [13, 14]. Torrefaction is described as a pre-treatment and valorisation process in which low-value biomass or carbonaceous feedstocks are heated at low temperatures (200 to 300) °C, low pressures, and short residence times (10 to 60) minutes [15, 16]. During the process, the raw material properties such as grindability, hydrophobicity, energy content, and energy density are enhanced [17, 18]. However, the application of the process to biomass feedstock, which typically has variable properties requires proper pre-examination. Thermogravimetric analysis (TGA) is a versatile technique used to examine the thermal degradation behaviour, temperature profiles, and waste potential properties of potential biomass-based raw materials for energy recovery [19]. Numerous studies have employed TGA to examine the thermal behaviour and energy recovery potential of biomass feedstock through torrefaction [20, 21], pyrolysis [22, 23], and gasification [24, 25] in the literature. TGA is typically performed under either oxidative (air/oxygen) or non-oxidative (nitrogen, carbon dioxide) conditions to simulate combustion, pyrolysis, or torrefaction.

Therefore, the objective of this study is to investigate the potential product yield and energy recovery of MC from carbon dioxide-assisted torrefaction through thermogravimetric analysis (TGA). The CO_2-assisted torrefaction of MC is performed from 240°C to 300°C (Δ30°C) for the residence time of 30 minutes based on the non-isothermal/isothermal heating programme of the TGA. Furthermore, the study examines the physicochemical, microstructure, mineral, and thermal fuel characteristics of MC for potential energy recovery and other applications.

I. Experimental

This section of the paper presents the materials and methods employed to examine the physical, chemical, microstructure, and mineral characteristics of MC. The procedure for the carbon dioxide (CO_2) assisted TGA torrefaction of MC along with the yield and distribution of products are presented in detail.

1.1. Physicochemical Analysis.

The physicochemical properties of maize cobs (MC) were examined by ultimate, proximate, and calorific analyses to determine its elemental, chemical fuel, and higher heating values. The ultimate analysis was performed through elemental (CHNS) analysis using the Vario Macrocube (Germany) apparatus to determine the carbon (C), hydrogen (H), nitrogen (N), and sulphur (S) contents. Thermogravimetric analysis (TGA) was employed to determine the proximate properties, namely; moisture (M), volatile matter (VM), ash (AC) and fixed carbon (FC), based on the detailed experimental procedure presented in the literature [26]. The higher heating value was conducted through bomb calorimetry using the IKA C200 (USA) isoperibolic bomb calorimeter under oxygen (oxidative) thermal conditions.

1.2. Microstructure Analysis

The microstructure of the maize cobs (MC) was examined through scanning electron microscopy (SEM) JEOL JSM IT-300 LV (Japan). For each test, the pulverised MC sample was spray coated on carbon epoxy tape pre-placed on grain mounts. Next, the sample grain mounts were sputter-coated with gold (Au) using the automatic thin-film sputter coater (Quorum 150 RS, United Kingdom) to improve the image quality and inhibit the charging effect during SEM analysis. The sample was then scanned in a vacuum to acquire the surface micrographs at a magnification of ×1000 based on the point ID and mapping method.

1.3. Mineral Analysis

The bulk chemical or mineralogical properties of the maize cobs (MC) was examined by electron dispersive X-ray (EDX) analysis. For the test, the SEM microscope (JEOL JSM IT-300 LV, Japan) was used to map the selected zone on the surface of the sample. Next, the SEM micrograph was scanned to quantitatively compute the composition of the constituent elements based on charge balance.

1.4. CO_2 TGA Torrefaction

The carbon dioxide (CO_2) assisted torrefaction of MC was carried out through thermogravimetric (TGA)

Table 1

Physicochemical Fuel Properties of MC

Analysis	**Fuel property**	**Symbol (Unit)**	**MC (db)**
Ultimate	Carbon	C (wt.%)	46.25
	Hydrogen	H (wt.%)	6.99
	Nitrogen	N (wt.%)	0.87
	Sulphur	S (wt.%)	0.15
	Oxygen	O (wt.%)	45.74
Proximate	Moisture	M (wt.%)	9.44*
	Volatile Matter	VM (wt.%)	75.32
	Fixed Carbon	FC (wt.%)	18.29
	Ash	AC (wt.%)	6.39
Calorific	Higher Heating Value	HHV (MJ/kg)	17.34

analysis. The process was performed through combined non-isothermal and isothermal heating programs based on the procedures of the Shimadzu TG-50 (Japan) TG analyser. For each run, approximately 11.5 mg of sample was weighed in an alumina crucible before transferring to the sample chamber of the TG furnace. Next, the furnace was purged with CO_2 to remove any air/oxidative gases and create an anoxic environment for the torrefaction process. The sample was heated from room temperature to the selected torrefaction temperature (T = 240°C, 270°C, or 300°C) at the heated rate of 20°C/min for the non-isothermal stage of the heating process. Once the torrefaction temperature was achieved, the process was switched to isothermal heating mode and held for 30 minutes to perform the CO_2 torrefaction. On completion, the TG furnace was cooled to room temperature using an automatic air blower, whereas the resulting TG data were retrieved and analysed using the thermal analysis software (Shimadzu TA-60WS) to examine the performance of the process based on the mass loss (ML) and the thermogravimetric (TG, %) and derivative thermogravimetric (DTG, %/min) plots as a function of the torrefaction time (min).

1.5. Product and Energy Yield Analysis

The product and energy yield analysis of the CO_2 torrefaction process was examined based on the mass loss (ML) and residual mass (RM) of the TGA. The RM and ML were computed from the TG plots using the analysis feature of the Shimadzu thermal analysis software (version TA-60WS). Subsequently, the torrefaction parameters; higher heating value (*HHV*), mass yield (*MY*), and energy yield (*EY*) were computed from the mass loss (*ML*) using equations 1-4 [27], whereas the energy density (*ED*) was computed from the *MY* [16];

$$HHV\left(\frac{MJ}{kg}\right) = 19.85 + 9.35ML(\%) \qquad (1)$$

$$MY(\%) = 100 - ML(\%) \qquad (2)$$

$$EY(\%) = 1 - 0.06ML(\%) \qquad (3)$$

$$ED = \left(\frac{EY}{MY}\right). \qquad (4)$$

II. Results and Discussion

2.1. Physical and Chemical Analysis

Table 1 presents the physicochemical properties of MC examined based on ultimate, proximate, and calorific analyses computed and reported on an air-dry basis. The objective was to elucidate the elemental composition, chemical fuel properties, and higher heating value of MC. The energy recovery potential, suitability, and environmentally friendliness of biomass feedstocks can be determined from their physicochemical properties [28, 29]. The results show that the chemical structure of MC has high contents of carbon, hydrogen, and oxygen (which are combustible elements) but relatively low contents of the pollutant elements such as nitrogen and sulphur as computed on a dry basis. Likewise, the proximate properties reveal high volatiles (> 75%) and fixed carbon (> 15%) contents, whereas the moisture and ash exist in low concentrations. The low moisture (< 10%) suggests thermochemical energy recovery could result in an efficient conversion process due to the relatively dry nature of MC, which eliminates the high energy requirement for biomass drying [28].

The low ash content eliminates the propensity for bed material agglomeration, sintering or fouling during thermochemical conversion in biomass reactors such as gasifiers and boilers [30, 31]. Lastly, the higher heating value of MC (17.34 MJ/kg) indicates that its calorific value is within the range of 14 MJ/kg – 22 MJ/kg [32, 33] typically required for bioenergy utilisation through thermochemical conversion. Overall, it can be reasonably surmised that MC contains the requisite elements and energy content required for energy recovery through torrefaction, pyrolysis, gasification, or combustion.

2.2. Microstructure Analysis

Fig. 1 shows the SEM micrograph of MC examined through scanning electron microscopy at the gnification of ×1000. Based on the analysis of the rograph, MC has a rough or coarse surface, but compact structure characterised by a layered arrangement of fibres. Hence, the microstructure of MC consists of fibres with an average diameter of 100 µm, which indicates the presence of microfibrils. The surface roughness or coarseness observed in the SEM micrograph of MC could indicate high surface area and crystallinity [34], whereas the compact structure could provide insights into the porosity [35]. Based on its rough surface, high surface area but low porosity, MC is potentially suitable for non-oxidative thermal conversion processes such as torrefaction and pyrolysis for high yield biochar production.

2.3. Mineral Analysis

Fig. 2 shows the EDX spectra of MC determined from the selected SEM micrograph. Based on the findings, MC contains numerous elements, namely; carbon, oxygen, potassium, chlorine, calcium, magnesium, silicon, and aluminium in various quantities as shown in the spectra. The major elements (indicated by composition ≥ 1 wt.%) detected include carbon, oxygen, and potassium. Carbon and oxygen are the key building blocks of lignocellulosic biomass and hence their detection during EDX may be ascribed to the holocellulose, lignin and extractives components in the chemical structure of MC [35, 36]. However, the trace elements (indicated by composition ≤ 1 wt.%) detected, include chlorine, calcium, magnesium, silicon, and aluminium. The presence of chlorine along with oxides of magnesium and calcium could pose technical problems arising from potential reactor fouling during thermal conversion. However, the findings also indicate that the ratio of Si to Al is unity, which indicates MC could be utilised for producing low silica zeolites with ratios of 1 to 2 and high hydrophilicity [37].

2.4. CO_2 TGA Torrefaction Analysis

Fig. 3 and 4 shows the TG and DTG plots for the CO_2 Torrefaction of MC, whereas Fig. 5 shows the temperature profile plots for the process at the selected conditions. The TG plots show that the increase in temperature from 240

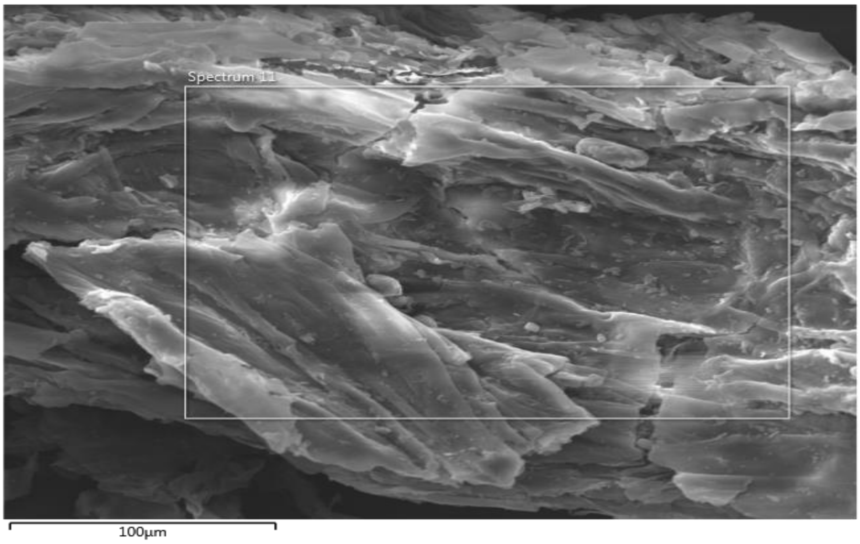

Fig. 1: SEM Micrograph of Maize Cob (MC).

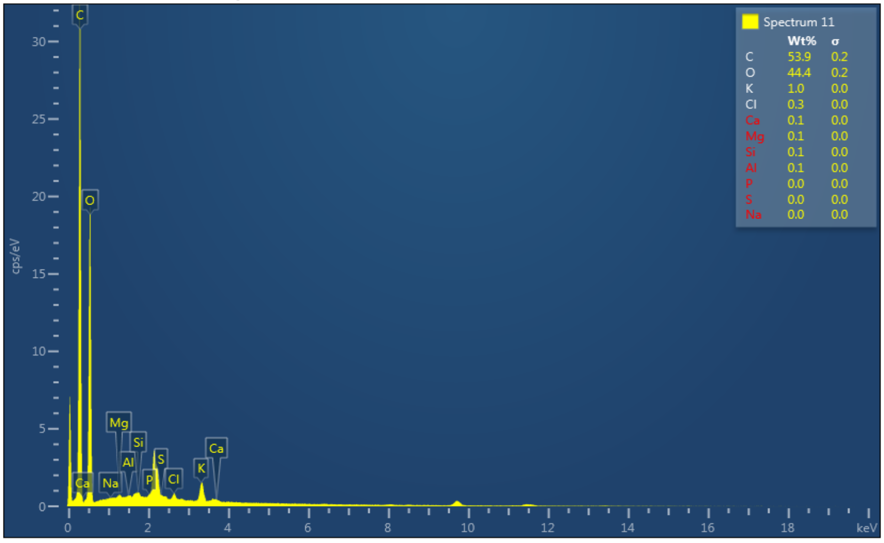

Fig. 2: EDX Spectra of Maize Cob (MC).

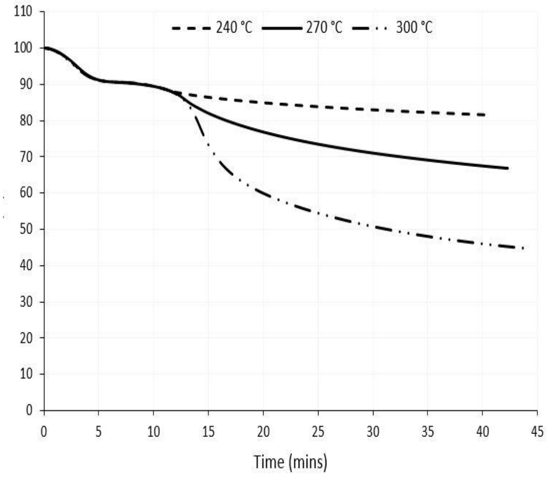

Fig. 3: TG plots for CO2 Torrefaction of MC.

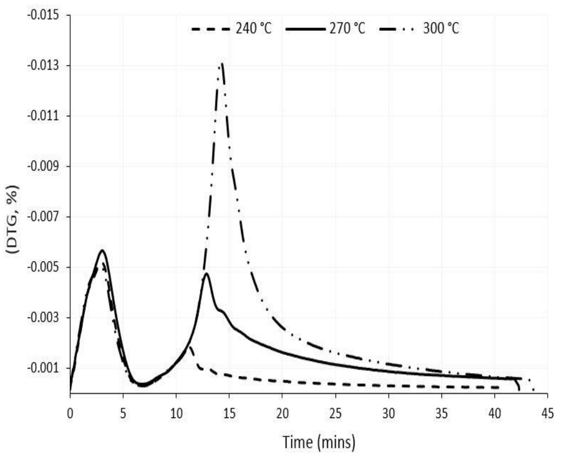

Fig. 4: TG plots for CO2 Torrefaction of MC.

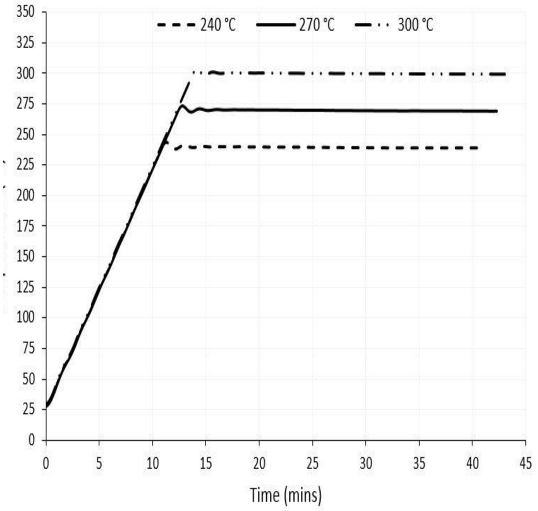

Fig. 5: Temperature Profiles for the CO_2 Torrefaction of MC.

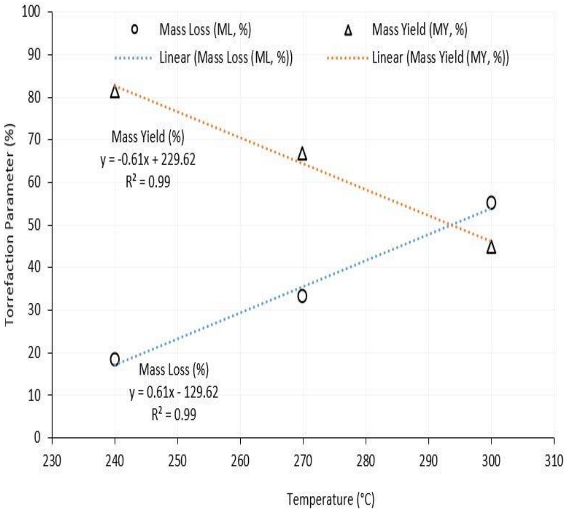

Fig. 6: Mass loss and Residual Mass Plots for the CO2 Torrefaction of MC.

Table 1

Torrefaction Performance of Maize Cobs (MC).

Torrefaction Temperature (°C)	HHV (MJ/kg)	EY (%)	DE (n.u.)
240	21.58	98.89	1.21
270	22.95	98.01	1.47
300	25.01	96.69	2.16

°C to 300 °C for 30 mins resulted in various degrees of mass loss (ML) of the sample. As observed, the most significant mass loss occurred at 300°C, whereas the least was at 240°C, which indicates that higher temperatures result in higher degradation of the sample. The ML observed during the torrefaction process can be ascribed to the degradation of hemicellulose, cellulose, and lignin in decreasing order during the CO_2 torrefaction process [16, 33]. Hemicellulose is considered the most reactive component of biomass and reportedly undergoes thermal degradation in the temperature range 150 - 350 °C, whereas cellulose and lignin occur from 275 – 350°C and 250 – 500°C, respectively [36]. The degradation of lignocellulosic biomass components typically results in ML.

The DTG plots for the CO_2 torrefaction of MC in Fig. 4 reveal two sets of endothermic peaks, which indicates a three-stage degradation process. The first set of nearly symmetric peaks observed in the range of 0 – 7 mins resulted in an average mass loss of 9.47%. Hence, the degradation of MC during this stage could be reasonably ascribed to evaporation of surface moisture (i.e. drying or dehydration) or Stage I [33, 38]. The second set of endothermic peaks are observed in the range of

7 – 20 mins resulting in mass losses of 5.6%, 13.67% and 30.67% for the torrefaction temperatures 240°C, 270°C, and 300°C, respectively. The degradation of MC during this stage could be ascribed to the loss of the lignocellulosic components of MC such as hemicellulose, cellulose and lignin [33, 39]. The last stage III could be attributed to the formation of char, which is computed as the mass yield (MY) of the torrefaction process. The resulting ML during this stage at the torrefaction temperatures 240°C, 270°C, and 300°C are 3.39%, 10.00% and 15.08%, respectively.

Fig. 6 shows the overall mass loss (ML) and residual masses (RM) for the CO_2 torrefaction of MC computed from the TGA data. It was observed that the ML increased from 18.45% to 55.17%, whereas the RM (computed as mass yield) decreased from 81.55% to 44.83% with increasing temperatures from 240°C to 300°C during the CO_2 torrefaction process. The plots also revealed high correlation R^2 values of 0.99 for ML and RM. The average ML and RM for CO_2 torrefaction are 35.60% and 64.40% based on the conditions examined in this TGA study for MC. Consequently, energy contents (HHV), yield and densities of the MC based char produced were examined.

2.5. Torrefaction Product and Energy Yield Analysis

Table 2 presents the computed values of the higher heating values, energy yields and energy density of the biochar produced from the TGA CO_2 torrefaction of MC. The parameters are typically employed to examine the performance of torrefaction as well as the bioenergy potential of the fuel produced from the process. As observed in Table 2, the higher heating value (HHV) of the resulting MC biochar is in the range of 21.58 MJ/kg - 25.01 MJ/kg (or an average of 23.18 MJ/kg). Hence, the biochar HHVs are significantly higher than MC (HHV = 17.34 MJ/kg), which suggests that torrefaction enhanced the bioenergy content by 24.455% - 44.25% (or an average of 33.70%). Similar results have been reported for other biomass in the literature [15, 40]. However, the energy yield (EY) decreased from 98.89% to 96.69%, which is due to the decrease in the MY (81.55% to 44.83%), as earlier reported. Nonetheless, the energy density increased from 1.21 to 2.16 (or 1.61 on average) with increasing temperatures from 240°C to 300°C during the CO_2 torrefaction process. The increase in energy density can be ascribed to the increase in the HHV of the biochar, which indicates the biochars have a high potential for energy recovery and future clean energy applications.

Conclusion

The paper examined the carbon dioxide torrefaction of maize cobs (MC) through thermogravimetric analysis to estimate the yield and energy of the biochar products for future energy recovery. Besides, the physicochemical, microstructure and bulk chemical properties of the MC was highlighted in detail. The findings demonstrated that MC is a potentially suitable feedstock for torrefaction due to the high mass yields, energy content, and energy density. Furthermore, the CO_2 torrefaction process significantly enhanced the energy recovery potential by 24.45% - 44.25% and energy density from 1.21 to 2.16. Overall, the thermogravimetric CO_2 torrefaction process is a useful approach to examine the energy recovery potential of MC. Hence, the study can be extended to bench-scale tests using fixed bed reactors to further demonstrate the applicability of the process to other agricultural wastes in the future.

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Торефікація у вуглекислому газі качанів кукурудзи за допомогою термогравіметрії: продуктивність та потенціал відновлення енергетики

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Метою дослідження є вивчення потенційної врожайності продукту та відновлення енергії качанів кукурудзи (MC) шляхом торрефікації термогравіметричного аналізу (TGA) за допомогою двоокису вуглецю. Торефікацію MC за допомогою CO_2 проводили від 240°C до 300°C (Δ30°C) протягом 30 хвилин на основі вибраної програми неізотермічного/ізотермічного нагрівання TGA. Крім того, досліджено фізико-хімічні, мікроструктурні та мінеральні характеристики MC. Результати показали, що CO_2-торрефікація MC призвела до втрати маси у діапазоні від 18,45% до 55,17%, що спричинило спад по масі у діапазоні від 81,55% до 44,83%. HHV твердого продукту знаходився в діапазоні від 22,55 МДж/кг до 26 МДж/кг, що вказує на підвищення енергетичного вмісту MC у процесі CO_2-торрефікації на 40-60%. Результати показали, що торрефікація CO_2 є практичним, стійким та економічно ефективним підходом для перетворення MC в чисте тверде біопаливо для покращеного відновлення енергії.

Ключові слова: дірксид вуглецю, торрефікація, качани кукурудзи, термогравіметрія, відновлювальна енергетика.