Nigerian Journal of Physics (NJP)
ISSN online: 3027-0936
ISSN print: 1595-0611
DOI: https://doi.org/10.62292/njp.v35i4.2026.600
Volume 35(4), December 2026

Effect of Temperature and Retention Time on the Quality of Biochar of Some Agricultural Waste

Lawan Haruna, Aliyu Aminu Safana, Ibrahim Murtala Musa, Hafeez Y. Hafeez, Umar M. Dankawu and Muhammad M. Machina

Department of Physics, Federal University Dutse, Dutse, Jigawa state, Nigeria.

*Corresponding Author's Email: lawanharuna95@gmail.com

Keywords:
Pyrolysis,
Biochar,
Proximate analysis,
Ultimate analysis,
Calorific values

Submitted: 25 May 2026

Accepted: 15 June 2026

Published: 02 July 2026

ABSTRACT

Cadmium sulfide (CdS) thin films have attracted significant attention for environmental remediation applications due to their suitable band gap, high photosensitivity, and excellent photocatalytic properties. In this study, non-aqueous CdS thin films were hydrothermally synthesized using cadmium nitrate tetrahydrate and thiourea as precursor materials in methanol medium. The films were prepared at varying precursor concentrations of 0.10 M, 0.20 M, and 0.30 M. To enhance the photocatalytic performance of CdS, 0/005g of bismuth ferrite ($BiFeO_3$) and 0.005g tin oxide (SnO_2) were incorporated as dopants in separate preparation. The synthesis was carried out in a Teflon-lined autoclave at 180 °C for 8 h, followed by washing, drying, and pulverization of the obtained products. The structural, morphological, optical, and photocatalytic properties of the synthesized samples were investigated using appropriate characterization techniques. The influence of precursor concentration and dopant incorporation on crystallite size, surface morphology, and optical band gap was evaluated. Photocatalytic activity was assessed through the degradation of methylene blue dye under natural sunlight irradiation. The results revealed that both $BiFeO_3$ and SnO_2 doping significantly improved the photocatalytic efficiency of non aqueoud CdS to 70.0 & 72.8% respectively by enhancing charge separation and reducing electron–hole recombination. The findings demonstrate that hydrothermally synthesized non-aqueous CdS thin films doped with $BiFeO_3$ and SnO_2 are promising photocatalysts for the degradation of organic pollutants in wastewater under sunlight irradiation, offering a cost-effective and environmentally friendly approach for water purification applications.

INTRODUCTION

The growing world population and accelerating industrialization keep increasing the energy demand. The concurrent global warming and concerns about the depletion of fossil fuel reserves necessitate the development of sustainable ways to produce energy (Hasan *et al.*, 2019). Biomass is a renewable energy source that can partly relieve the energy crisis and environmental problems. Then the technology of converting biomass effectively and efficiently into fuel and chemical stock receives more and more attention. The growing world population and accelerating in industrialization keep increasing the energy demand. The concurrent global warming and concerns about the depletion of fossil fuel reserves necessitate the development of sustainable ways to produce energy (Muhammad *et al.*, 2012). Because biomass is considered a carbon neutral source of energy, partial replacement of coal with biofuels in commercial combustion units lowers the carbon dioxide emissions. However, biomass properties, such as heterogeneous and tenacious structure, hydrophilic nature, and high moisture content are posing challenges to using biomass for energy production (Keipi *et al.*, 2014).

Pyrolysis is one of the most attractive processes to convert biomass into economically renewable intermediates (Mettler *et al.*, 2012). As pyrolysis is an endothermic thermochemical process, external heat is required. In standard pyrolysis, the heat is obtained by combustion of fossil fuel or of part of the initial biomass, which reduces the energy efficiency and increases the pollution discharge (Nzihou *et al.*, 2012).

The effects of pyrolysis conditions on the structure and reactivity of biomass chars have been investigated in several studies in relation to the following parameters: temperature (Fu *et al.*, 2012a, 2011; Kim *et al.*, 2012; Min *et al.*, 2011; Bonelli *et al.*, 2001), heating rate (Mermoud *et al.*, 2006; Guerrero *et al.*, 2005; Fushimi *et al.*, 2003), residence time and pressure (Zeng *et al.*, 2015) 'Pyrolysis', a thermochemical conversion route is emerging as a promising technology for the production of bio-oil. Pyrolysis is the thermal decomposition of a feed substance into smaller fragments by heating in the absence of oxygen. The main benefit of pyrolysis process is that the liquid fuel produced can be easily stored and transported. Apart from the energetic use of bio-oil, a unique advantage of bio-oil over other renewable energies is that an array of value-added chemicals can be isolated from it. Several industrially important organic compounds which are conventionally synthesized from crude oil can be obtained in the pyrolytic oils, thus providing a green route for their production and efficient utilization of carbon (Dhyani *et al.*, 2017).

MATERIALS AND METHODS

Rice husk, millet husk and groundnut shell

Sample Collection and Sample Preparation

Samples of rice husk (Oryza sativa.), millet husk (Pennisetum glaucum), and groundnut shell (Arachis hypogaea) were collected from grain and legume processing mills located in kiyawa and surrounding communities in Jigawa State, Northern Nigeria. Representative composite samples were obtained by collecting multiple sub-samples from different points within the processing facility to minimize sampling heterogeneity. Each sample was air-dried at ambient temperature for 14 days to reduce initial moisture content. The dried samples were then comminuted using a laboratory hammer mill and sieved through a 250 µm mesh to obtain a uniform fine powder suitable for elemental analysis and analytical determinations. All prepared samples were stored in sealed airtight polyethylene containers in a desiccator prior to analysis to prevent moisture re-adsorption. All laboratory analyses were conducted at Bayero University Kano (BUK), Kano State, Nigeria.

pyrolysis Process

Pyrolysis experiment of the biomass was conducted three times separately using a stainless steel reactor. The reactor was fed with raw biomass and place inside the electric furnace and heated to $400^0C, 450^0C, 500^0 and\ 550^0C$ at a constant rate of 10^0Cmin-1 for 30 minute, first phase of the experiment. Also the optimum temperature obtained and constant heating rate of $10C$min-1 will be used for the second pyrolysis to determine the effect of retention time at 30, 45 and 60 minute.

Characterization

Proximate and Ultimate Analysis

Proximate analysis is a laboratory test that determines the composition of biomass in percentage of moisture content, ash content, volatile matter content and fixed carbon content.

Ultimate Analysis

Ten gram of each sample was prepared placed on a sample boat, in an Ultimate analyzer at Bayero University Kano, Kano State then the chamber is closed for a while to choose the CHNSO mode, selected using the temperature to allow for heating up to 1000℃ inside before the display of the result on the screen. Presence of Carbon, Hydrogen, Nitrogen, Sulphur and Oxygen being displayed and recorded in percentage.

Calorific Value Determination (CV)

The samples were crushed using mortar and pestle after being completely dried, then measured to observe the required weight of each.

The test sample of the biomass material to be determined was prepared then inserted in to a seal bomb calorimeter to burn at a respective temperature on ideal value of each of the placed crucibles inside the bomb calorimeter and then result is display on the screen in the bomb calorimeter at center for dry land agriculture Bayero University Kano.

RESULTS AND DISCUSSIONS

Calorific Value of Biochar

Table 1: Calorific Value of the Biochar Obtained after Pyrolysis at Difference Temperature

Temperature (ºC)	Calorific Value (MJ/kg)	Rice Husk Char (g)	Millet Husk Char (g)	Groundnut shell Char (g)
400 ºC	Calorific Value (MJ/kg)	17.18	23.95	17.91
450 ºC	Calorific Value (MJ/kg)	15.91	22.09	26.93
500 ºC	Calorific Value (MJ/kg)	15.76	24.58	25.38
550 ºC	Calorific Value (MJ/kg)	15.40	24.97	25.64

Table 1 shows the calorific values (CV) of the biochars this shows a distinct trend with pyrolysis temperature and varies notably among feedstocks. For RH char, calorific value decreases from 17.18 MJ/kg at 400 °C to 15.40 MJ/kg at 550 °C, indicating that higher temperatures reduce its energy content. This is somewhat unexpected because biochar produced at higher temperatures typically becomes more carbonized and should have a higher heating value on a dry ash-free basis. However, the observed decline may be due to increased ash content or structural changes that lower the combustible fraction. For MH char, calorific value increases from 23.95 MJ/kg at 400 °C to 24.97 MJ/kg at 550 °C, with a slight dip at 450 °C. This positive trend aligns with typical behavior: higher temperatures remove more volatiles, concentrating fixed carbon and raising the energy content. GS char shows a different pattern calorific value raises from 17.91 MJ/kg at 400 °C to 26.93 MJ/kg at 450 °C, then plateaus around 25.38 to 25.6 MJ/kg at 500 to 550 °C. This large jump between 400 and 450 °C implies that GS undergoes rapid devolatilization and carbon enrichment in that narrow temperature range, after which further heating yields little additional gain.

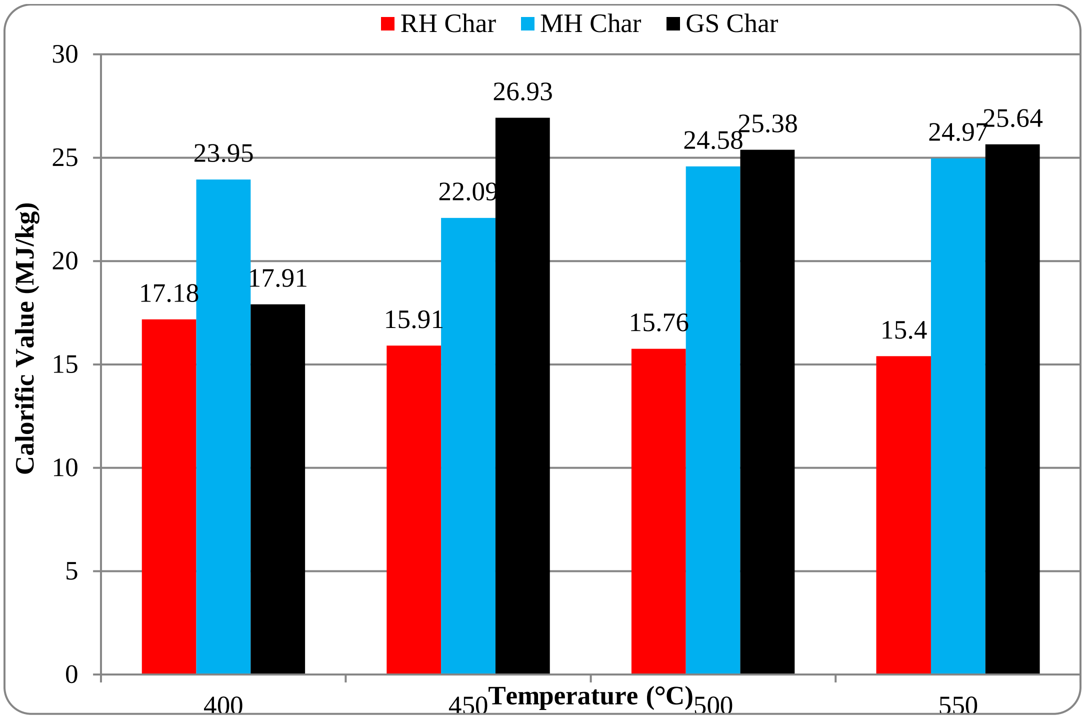

Figure 1: Chart showing Variations in Calorific Value (Energy Content) of Biochar

Figure 1 shows the calorific value of RH char, MH char and GS char based on selected optimum temperature as shown above.

Proximate Analysis, Ultimate Analysis and Calorific Value of Biochar

Table 2: Proximate Analysis and Calorific Value of the Biochar Produced at Optimum Temperature selected from Table 2 Results (RH Char 400, MH Char 450 and GS Char 450°C) and Constant Time of 30 Minutes

S/N	Samples	Moisture content (%)	Ash content (%)	Volatile matter (%)	Fixed carbon (%)	Calorific Value (MJ/kg)
1.	Rice Husk Char (g)	2.84	21.10	36.18	21.18	17.18
2.	Millet Husk Char (g)	4.50	3.90	55.58	20.99	24.97
3.	Groundnut shell Char (g)	4.55	2.00	15.15	64.49	26.93

Table 2 presents the proximate analysis and calorific values of three agricultural residue chars: rice husk char, millet husk char, and groundnut shell char. These parameters, moisture content, ash content, volatile matter, fixed carbon, and calorific value are essential for evaluating the quality and combustion behavior of each material. Regarding moisture content, all three samples show low values, ranging from 2.84% for RH char to 4.55% for GS char. Low moisture is advantageous for thermochemical conversion processes such as combustion or gasification because it reduces ignition difficulties and minimizes heat losses caused by water evaporation. RH char has the lowest moisture (2.84%), which gives it a slight advantage, though the differences among the samples are relatively small.

Ash content, however, varies dramatically. Rice husk char has the highest ash content at 39.80%, indicating a large proportion of inorganic mineral matter. A high ash content lead to a lower heating value, increase the risk of slagging and fouling, and creates greater disposal challenges. In contrast, MH char shows a moderate ash content of 18.90%, while GS char has the lowest ash content at 15.80%, which is beneficial for cleaner combustion and higher energy density. The high ash content in RH char is typical for rice residues due to silica accumulation. Consequently, RH char would be less suitable for domestic or small-scale combustion unless blended with low-ash fuels or used in applications where ash can be recovered, such as in cement production (Ezealigo et al., 2021; Nguyen *et al.*, 2023).

Volatile matter reflects the ease of ignition and flame stability. MH char exhibits very high volatile matter (55.58%), suggesting that it will ignite readily and burn with a long flame. However, high volatile matter can also lead to smoke and incomplete combustion if the air supply is insufficient. GS char, on the other hand, has very low volatile matter (15.15%), indicating a more carbonized, charcoal like material that burns slowly and steadily with little smoke. RH char falls in between, with moderate volatile matter of 36.18%. The low volatile matter of GS char, combined with its high fixed carbon, points to a well carbonized product suitable for applications requiring sustained heat, such as metallurgical reduction or slow combustion. Fixed carbon is directly correlated with the char's energy density and burn duration. GS char has the highest fixed carbon content at 64.49%, which explains its superior calorific value. High fixed carbon means more carbon is available for oxidation, producing more heat per unit mass. By comparison, RH char and MH char have similar fixed carbon contents of approximately 21%, reflecting their lower energy contents. In general, fixed carbon is inversely related to ash content and, to a lesser extent, volatile matter. GS char benefits from both low ash and low volatile matter, resulting in a high fixed carbon value

Finally, the calorific values follow the same trend as fixed carbon and ash. GS char achieves an excellent heating value of 26.93 MJ/kg, which is comparable to some low rank coals and makes it suitable for high efficiency boilers, briquetting, or co-firing. MH char gives a still good value of 24.97 MJ/kg, though it is slightly lower due to its higher ash and volatile matter R (Kumar *et al.*, 2020).H char has a significantly lower calorific value of 17.18 MJ/kg, primarily because of its very high ash content (approximately 40%). This value is marginal for energy recovery; however, RH char may still be useful for non-energy applications such as soil amendment (biochar) or as a filter medium.

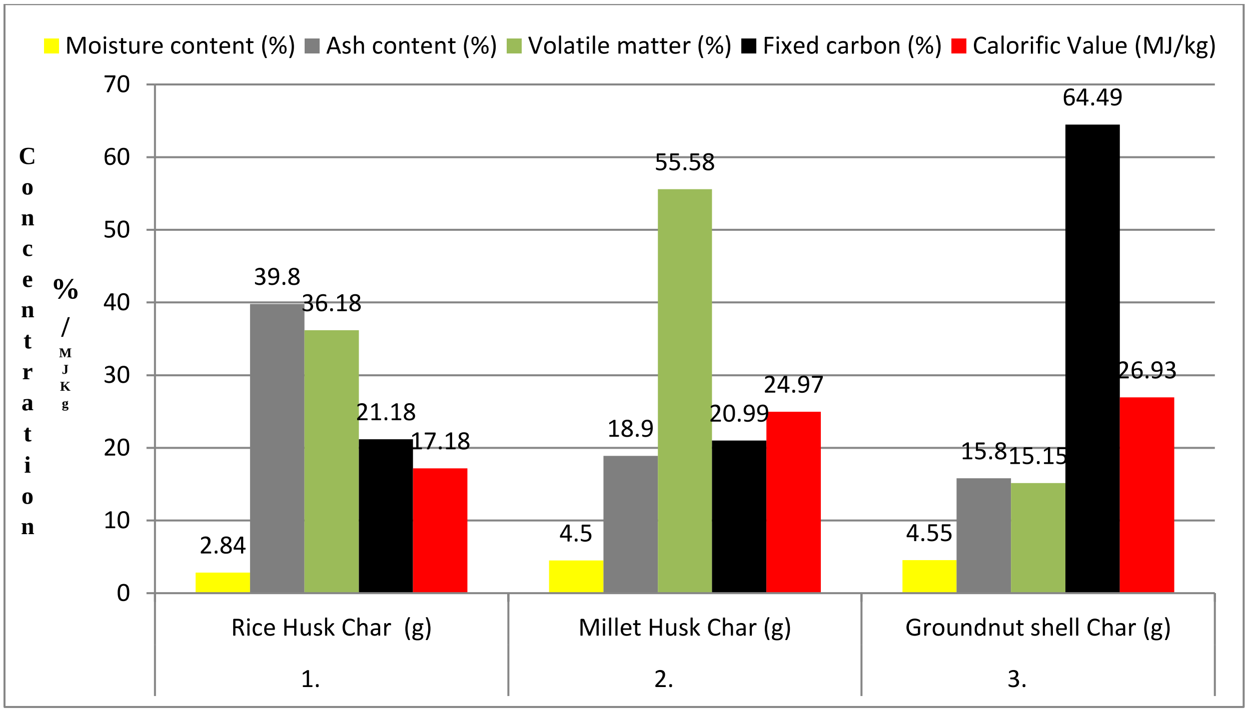

Figure 2: Chart showing Variations in Physical Parameters of Biochar

Figure 2 translates the variations in moisture content, Ash content, Volatile matter content, fixed carbon content and calorific value of the samples (RH, MH and GS).

Ultimate Analysis of the Biochar Produced at Optimum Pyrolysis Conditions

Table 3: Ultimate Analysis of the Biochar Produced at Optimum Pyrolysis Conditions

S/N	Sample ID	Carbon (%)	Hydrogen (%)	Nitrogen (%)	Sulphur (%)	Oxygen (%)
1	Rice Husk char (g)	56.94	4.15	0.78	0.012	38.12
2	Millet Husk char (g)	79.27	1.80	1.15	0.01	17.77
3	Groundnut shell char(g)	67.03	4.12	1.62	0.02	27.21

Table 3 presents the ultimate (elemental) composition of three agricultural residues: rice husk, millet husk, and groundnut shell. The values are reported on a dry basis and sum to approximately 100% for each sample, confirming good analytical accuracy. Ultimate analysis is crucial for predicting combustion behaviour, heating value, emissions potential (especially nitrogen and sulphur oxides), and the stoichiometric air requirement for complete combustion. Carbon content is the primary contributor to calorific value. Millet husk shows the highest carbon content at 79.27%, which is notably high for a raw biomass material and suggests a high energy density. Groundnut shell follows with 67.03% carbon, while rice husk has the lowest carbon at 56.94%. The high carbon in millet husk indicates that it would produce a substantial amount of heat per unit mass during combustion, consistent with its expected calorific value.

Hydrogen content influences the heat released during combustion and the formation of water vapour in flue gases. Rice husk and groundnut shell have similar hydrogen values (4.15% and 4.12%, respectively), while millet husk has a much lower hydrogen content of only 1.80%. This low hydrogen percentage in millet husk is unusual for raw biomass (which typically contains 5–6% hydrogen) and may indicate a different sample condition, such as partial charring or a specific variety. Lower hydrogen reduces the heating value slightly because hydrogen contributes to the heat of combustion. Nitrogen content is important because nitrogen oxides (NO_x) formed during combustion are environmental pollutants. All three samples have relatively low nitrogen levels, ranging from 0.78% (rice husk) to 1.62% (groundnut shell). Groundnut shell has the highest nitrogen at 1.62%, followed by millet husk at 1.15%, and rice husk at 0.78%. While these values are moderate compared to some coals, they still imply that NO_x emissions would be manageable, especially with proper combustion control. Rice husk is the most favourable in terms of low NO_x formation.

Sulphur content is very low across all three samples, with values of 0.012% (rice husk), 0.01% (millet husk), and 0.02% (groundnut shell). These are negligible compared to coal (which often contains 0.5–3% sulphur). Consequently, all three biomasses produce minimal sulphur dioxide (SO_2) emissions, making them environmentally attractive fuels. No special flue gas desulphurisation equipment would be required. Oxygen content is obtained by difference and reflects the proportion of bound oxygen in the biomass. High oxygen content reduces the net heating value because oxygen atoms are already partially oxidised. Rice husk has the highest oxygen at 38.12%, which explains its lower carbon content and expected lower calorific value. Millet husk has the lowest oxygen at 17.77%, consistent with its high carbon and low hydrogen. Groundnut shell has an intermediate oxygen value of 27.21%.

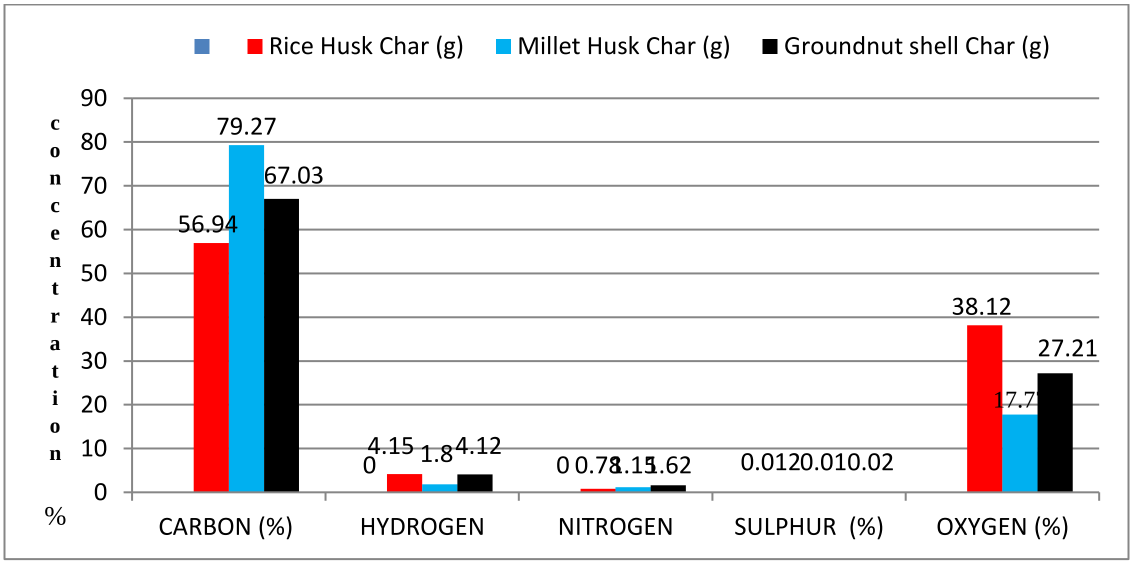

Figure 3: Ultimate Analysis Chart for Raw Sample

The Figure 3 shows the variations in the compositional values of carbon, Hydrogen, Nitrogen, Oxygen and Sulphur in biochar samples.

CONCLUSION

Pyrolysis, as a pretreatment, has the potential to improve the quality of the biochar yield. Increases in the pyrolysis temperature on some biomass also increases the quality of biochar also decreases in temperature in some biomass also increases the quality of char dependent on type of the biomass and longer time give lower yield of biochar. Other properties that are decreasing as the pyrolysis temperature and time increased are volatile matter content, moisture content, ash content and fixed carbon content. In contrast fixed carbon content and carbon content increased as the pyrolysis temperature and time increased. Increasing pyrolysis temperature and time also improved grindability of biochar.

ACKNOWLEDGEMENTS

The authors acknowledge the technical staff of chemistry Laboratory, Federal University Dutse, for their assistance during experimental analyses. The authors also acknowledge the Department of Physics, Federal University Dutse, Center for Dryland Agriculture Bayero University Kano and Advanced Nanotechnology and Computational Physics Laboratory, Jigawa State for technical support. The collection of biomass samples from processing mills in Jigawa State is gratefully acknowledged

REFERENCES

Dhyani, V., Awasthi, A., Kumar, J., & Bhaskar, T. (2017). Pyrolysis of Sorghum straw: Effect of temperature and reaction environment on the product behavior. *Journal of Energy and Environmental Sustainability*, *4*, 64–69. https://doi.org/10.47469/JEES.2017.v04.100049

Ezealigo U.S., B.N. Ezealigo, F. Kemausuor, L.E.K. Achenie, A.P. Onwualu Biomass valorization to bioenergy: assessment of biomass residues' availability and bioenergy potential in Nigeria Sustain., 13 (2021), p. 13806, 10.3390/su132413806

Filipovici, A., Tucu, D., Bialowiec, A., Bukowski, P., Crisan, G. C., Lica, S., Pulka, J., Dyjakon, A., & Debowski, M. (2017). Effect of Temperature and Heating Rate on the Char Yield in Sorghum and Straw Slow Pyrolysis. *Revista de Chimie*, *68*(3), 576–580. https://doi.org/10.37358/RC.17.3.5504

Filipovici, A., Tucu, D., Bialowiec, A., Bukowski, P., Crisan, G. C., Lica, S., Pulka, J., Dyjakon, A., & Debowski, M. (2017). Effect of Temperature and Heating Rate on the Char Yield in Sorghum and Straw Slow Pyrolysis. *Revista de Chimie*, *68*(3), 576–580. https://doi.org/10.37358/RC.17.3.5504

Ibrahim, H. A.-H. (2020). Introductory Chapter: Pyrolysis. In H. A.-H. Ibrahim (Ed.), *Recent Advances in Pyrolysis*. IntechOpen. https://doi.org/10.5772/intechopen.90366

Jones, M., *et al*. (2021). "Standardization of biomass briquette quality: Importance of calorific value." *Energy Reports*, 7, 1067-1076.

Keipi, T., Tolvanen, H., Kokko, L., & Raiko, R. (2014). The effect of torrefaction on the chlorine content and heating value of eight woody biomass samples. *Biomass and Bioenergy*, *66*, 232–239. https://doi.org/10.1016/j.biombioe.2014.02.015

Kumar, A., *et al*. (2020). "Effect of calorific value on the performance of biomass briquettes." *Journal of Cleaner Production*, 261, 121-132.

Kumar, A., *et al*. (2020). "Effect of calorific value on the performance of biomass briquettes." *Journal of Cleaner Production*, 261, 121-132.

Lakshmi Durga, M., Pal, L., & Wahid, A. (2024). Production of Biochar from Oilseed Residue (Deoiled Cakes): State-of-the-Art. IntechOpen. doi: https://doi.org10.5772/intechopen.114228

Mermoud, F., Salvador, S., Van de steene, L., Golfier, F., 2006. Influence of the pyrolysis heating rate on the steam gasification of large wood char particle. Fuel 85, 1473–1482. Mettler, M.S., Vlachos, D.G., Dauenhauer, P.J., 2012. Top ten fundamental challenges of biomass pyrolysis for biofuels. Energy Environ. Sci. 5, 7797–7890.

Muhammad, N., Omar, W. N., Man, Z., Bustam, M. A., Rafiq, S., & Uemura, Y. (2012). Effect of Ionic Liquid Treatment on Pyrolysis Products from Bamboo. *Industrial & Engineering Chemistry Research*, *51*(5), 2280–2289. https://doi.org/10.1021/ie2014313

Mohd Hasan, M., Bachmann, R., Loh, S., Manroshan, S., & Ong, S. (2019). Effect of Pyrolysis Temperature and Time on Properties of Palm Kernel Shell-Based Biochar. *IOP Conference Series: Materials Science and Engineering*, *548*(1), 012020. https://doi.org/10.1088/1757-899X/548/1/012020

Muhammad, N., Omar, W. N., Man, Z., Bustam, M. A., Rafiq, S., & Uemura, Y. (2012). Effect of Ionic Liquid Treatment on Pyrolysis Products from Bamboo. *Industrial & Engineering Chemistry Research*, *51*(5), 2280–2289. https://doi.org/10.1021/ie2014313

Nzihou, A., Flamant, G., Stanmore, B., 2012. Synthetic fuels from biomass using concentrated solar energy – a review. Energy 42, 121–131.

N.Abdullah, Safana A.A. and Fauziah S. (2017) Investigation Into The Effects Of Torrefaction On The Quality Of Pyrolysis Products

Nguyen, T. *et al*. (2023). "Impact of biomass properties on bio-briquette combustion characteristics." *Energy Conversion and Management*, 200, 112456

Patel, S., *et al*. (2023). "Integrative analysis of biomass briquettes for improved energy performance." *Bioenergy Research*, 16(1), 45-56.

Soria-Verdugo, A., Rubio-Rubio, M., Goos, E., & Riedel, U. (2020). On the characteristic heating and pyrolysis time of thermally small biomass particles in a bubbling fluidized bed reactor. *Renewable Energy*, *160*, 312–322. https://doi.org/10.1016/j.renene.2020.07.008

Sanchez, A., *et al*. (2018). "Comparative study of calorific values in biomass briquettes." *Renewable and Sustainable Energy Reviews*, 82, 1857-1865.

Safana A.A. Nurhayati Abdullah, Fauziah S. & Sadik Umar (2018) Combustion Characteristics of Palm Pressed fibres bio-char and sub-bituminous Malaysian coal Malaysian Journal of Fundamental and Applied Sciences Vol.14(3) 334-337

Smith, R., *et al*. (2019). "Quality control in biomass briquetting: The role of calorific value." *Energy Conversion and Management*, 183, 285-292.

S. Namadi, A. O. Musa and A. Bala (2017) determination of Physical properties and proximate Analysis of biomass briquette produced from cornstalk and waste paper bayero journal of physics and mathematical Sciences Vol. 8(1)53-60.

Wang, Y. *et al*. (2024). "Predicting the calorific value of lignocellulosic biomass using machine learning techniques." *Biomass and Bioenergy*, 155, 106312

Yadav, S., Kumar, A., & Kumar, N. (2020). Thermal compaction of biomass briquettes. *Journal of Renewable Energy*, 2020, 1-9.

Zeng, K., Minh, D. P., Gauthier, D., Weiss-Hortala, E., Nzihou, A., & Flamant, G. (2015). The effect of temperature and heating rate on char properties obtained from solar pyrolysis of beech wood. *Bioresource Technology*, *182*, 114–119. https://doi.org/10.1016/j.biortech.2015.01.112