



Statistical models for predicting the higher heating value of torrefied kesambi leaves

Jemmy J.S. Dethan  

Artha Wacana Christian University, Kupang, East Nusa Tenggara, Indonesia, 85111

RECEIVED 05.05.2024

ACCEPTED 25.09.2024

AVAILABLE ONLINE 02.12.2024

Abstract: The study investigates the effects of temperature and residence time on the energy density of kesambi leaves through experimental torrefaction, proximate analysis, and response surface methodology with a central composite design (*RSM-CCD*). The torrefaction process enhances the energy density of kesambi leaves by increasing fixed carbon content while reducing volatile matter. The *RSM-CCD* models developed in this research are both statistically significant and exhibit robust predictive accuracy for estimating higher heating value (*HHV*), providing valuable insights into optimal torrefaction conditions. Surface plots effectively illustrate the relationships between *HHV*, temperature, and residence time, enabling the identification of ideal process parameters. Additionally, a desirability analysis reveals opportunities to enhance correlations between *HHV* and key measured properties, such as moisture content, ash, and volatile matter. This research makes a significant contribution to understanding and optimising the torrefaction process for kesambi leaves, with practical implications for improving energy density and advancing the development of sustainable biofuel sources. By offering a novel approach to predicting *HHV* in kesambi leaf-based biofuels, the findings highlight the potential for optimising torrefaction processes to enhance the viability of renewable energy resources. Further research is suggested to refine these predictive models and explore additional factors influencing *HHV*, aiming to bolster the production of sustainable biofuels.

Keywords: biomass conversion, energy density, kesambi leaves, proximate analysis, torrefaction

INTRODUCTION

Deforestation mitigation practices have the potential to increase carbon stocks in existing forests, thus encouraging sustainable management of forest resources (Ahiduzzaman and Sadrul Islam, 2016). Improving biomass quality through torrefaction not only provides an alternative source of raw materials but also increases the overall value of biomass itself. This shift towards sustainable charcoal production supports more efficient use of renewable resources and reduces dependence on traditional woody biomass (Hwangdee *et al.*, 2021). Torrefaction of kesambi leaves is a crucial process in biomass conversion, aimed at enhancing energy density and suitability for energy applications. This study focuses on modelling and optimising the torrefaction process of kesambi leaves to improve their energy density. Temperature and residence time are two key variables that affect the torrefaction process, significantly affecting the quality and characteristics of the torrefied biomass. Understanding the influence of these

characteristics and adjusting them are essential steps in obtaining the required level of product quality (Orisaleye *et al.*, 2022).

The study involves comprehensive analysis, starting with experimental torrefaction of kesambi leaves under varying temperature and residence times. Kesambi leaves are composed of 15.27% hemicellulose, 29.51% lignin, and 27.62% cellulose (Mardyaningsih, Leki and Engel, 2016). Proximate analysis is conducted to track changes in biomass composition, including moisture content (*MC*), volatile matter (*VM*), fixed carbon (*FC*), and ash content (*ASH*), which are crucial indicators of torrefaction quality (García *et al.*, 2013; Oyeboode and Ogunsuyi, 2021; Torres Ramos *et al.*, 2023). The fixed carbon, representing the solid carbon that remains after volatile components are removed, reflects the combustion characteristics and energy potential (Dimiyati and Kurniasih, 2020).

This statistical approach facilitates the creation of mathematical models that explain the link between the independent variables, such as temperature, and residence time, and the

response variable, energy density (Etemadi and Khashei, 2021). By analysing the response surface, optimal process conditions are identified to maximise the energy density of torrefied kesambi leaves. Additionally, calculating the higher heating value (*HHV*) under various operating conditions provides insights into the effects of torrefaction parameters on biomass energy content (Majamo and Amibo, 2023).

Optimising response surface methodology (RSM) with a central composite design (CCD) offers a methodical way to comprehend how residence time and torrefaction temperature affect *HHV*. The creation of proximate analysis results-based *HHV* prediction models for diverse torrefaction conditions is another unique characteristic that makes *HHV* prediction possible even in the absence of direct data. By integrating proximate analysis, RSM, and torrefaction, this research presents a novel approach to predict the *HHV* of kesambi leaves undergoing torrefaction, offering useful insights for improving torrefaction conditions to obtain the desired *HHV* in kesambi leaf-based biofuels.

The combination of variables and methods employed in the study into the *HHV* of charred kesambi leaves provides a unique contribution to the field. A mathematical model to estimate the *HHV* value of kesambi leaves torrefaction is developed based on the prior research findings (Dethan *et al.*, 2023; Dethan *et al.*, 2024), as kesambi leaves have not been thoroughly explored in the context of RSM-CCD optimisation and are relatively rare.

STUDY MATERIALS AND METHOD

STUDY MATERIALS

The objective of this research was to examine the models proposed by Wahid, Nhuchhen, and Kieseler by using data from torrefaction experiments conducted on kesambi leaves. The torrefaction process was conducted at varying temperature and

residence time to assess their effects. The analysis utilised RSM with a CCD methodology, supported by the use of Design Expert 13 software.

The study was conducted at the Biosciences Laboratory at Nusa Cendana University and the Exact Sciences Laboratory at Artha Wacana Christian University. In previous research, Design Expert 13 (Dethan *et al.*, 2024) was used to establish coefficients of polynomial regression. This study focused at two variables: residence time (minutes) and temperature, denoted by variables x_1 and x_2 . The temperature range for torrefaction was 200–300°C, and residence time 10–20 min to settle.

Moisture content (*MC*) was measured using the ASTM D3173 method. The ASTM D1102-84 was used to calculate the amount of ash, while volatile matter (*VM*) and fixed carbon (*FC*) were quantified using ASTM D3175, which follows ASTM D3172 guidelines, (Dethan and Lalel, 2024). The model's validity was assessed through the coefficient of determination, and a lack-of-fit test to confirm stability. Based on proximate analysis data, a model was created to predict the properties of torrefied biomass. Table 1 shows the results from Design Expert 13, which generated 13 randomised treatment combinations.

STUDY METHODS

Statistical analysis was conducted to assess the suitability of the models proposed by Abdul Wahid, Saleh and Abdul Samad (2017) in Equation (1), Nhuchhen and Afzal (2017) in Equation (2), and Kieseler, Neubauer and Zobel (2013) in Equation (3) utilising the *F*-value model for torrefied kesambi leaves, lack of fit, and evaluations of R^2 , modified R^2 , predicted R^2 , and sufficient R^2 . A sufficient R^2 of more than 4 and a discrepancy between adjusted and anticipated R^2 of less than 0.2 show that the model fits the observed data fairly well (Dong, Li and Feng, 2019; Frost, 2019; Bhandari, 2020). The three aforementioned Equations are as follows:

Table 1. Response parameter proximate analysis

Std	Run	x_1 : temperature (°C)	x_2 : residence time (min)	<i>MC</i>	<i>ASH</i>	<i>VM</i>	<i>FC</i>
				(%)			
2	1	300	10	6.46	4.87	39.14	49.52
5	2	250	8	8.07	5.48	50.77	35.68
3	3	250	15	5.34	4.16	39.58	50.93
4	4	250	15	5.49	4.56	33.91	56.03
10	5	250	22	3.65	4.31	29.56	62.48
9	6	300	20	3.63	3.75	27.9	64.72
7	7	180	15	6.67	5.02	42.85	45.46
6	8	200	10	7.64	5.28	50.47	36.62
1	9	200	20	4.14	4.54	35.51	55.81
12	10	250	15	5.43	4.14	33.16	57.27
11	11	320	15	4.01	3.65	25.52	66.83
8	12	250	15	5.39	4.84	31.13	58.64
13	13	250	15	4.01	4.12	31.49	60.38

Explanations: Std = standard order, *MC* = moisture contents, *ASH* = ash content, *VM* = volatile matter, *FC* = fixed carbon.

Source: own elaboration based on data sourced from processed research findings.

1. Wahid model equation:

$$HHV = 15.85 + 1.93 \frac{FC}{VM} + 0.04 \frac{VM}{ASH} + 0.14 \frac{ASH}{FC} + 0.02 t + 0.01 T \quad (1)$$

2. Nhuchhen model equation:

$$HHV = 0.18 VM + 0.35 FC \quad (2)$$

3. Kieseler model equation:

$$HHV = 0.41 FC + 0.1934 VM \quad (3)$$

where: HHV = higher heating value, FC = fixed carbon, VM = volatile matter, ASH = amount of ash, MC = moisture content.

RESULTS AND DISCUSSION

WAHID MODEL RESPONSE

The regression model overall significance is evaluated using the F -value, with a high F -value indicating a statistically significant model. In this study, an F -value of 44.44 reflects strong statistical significance of the regression model. A low the lack of fit value suggests a strong alignment between the model and the observed data. In this case, a lack of fit value of 1.24 indicates an excellent fit.

With a coefficient of determination (R^2) value of 0.90, the regression model is able to account for 90% of the variation in the observed data. The adjusted R^2 , which accounts for sample size and the number of independent variables, is 0.88. This suggests that the model can account for around 88% of the variability in the observed data. The predicted R^2 , which serves as an indicator of the regression model's predictive ability for new data, stands at 0.82, demonstrating a robust capacity for accurate predictions. An adequate precision value of 19.64 further supports the regression model's predictive strength. These numbers show that the regression model has a strong predictive capacity for new data, it is statistically significant, and effectively captures the variability in the observed data.

$$HHV \text{ Wahid} = 21.69 + 1.23x_1 + 1.03x_2 \quad (4)$$

The Wahid HHV model's surface plot displays the colour-coded contour lines that correspond to the expected HHV of a substance (Fig. 1). Higher HHV values are shown in red and are located in the plot's upper left corner, following the colour legend, while lower HHV values are displayed in blue and are located in the plot's lower right corner. This plot displays the precise range of HHV values, which is 19.54–24.18 $\text{MJ}\cdot\text{kg}^{-1}$. Plotted along the boundaries of the plot are two data points: one with a resident time of 20 min and a temperature of 250°C, and another with a residence period of 10 min. The ideal HHV of 34.37 $\text{MJ}\cdot\text{kg}^{-1}$ can be achieved by torrefying coconut shells at 275°C for 30 min, according to the study findings of Alhinai *et al.* (2018). Upon examining the plot's particular range of HHV values 19.54–24.18 $\text{MJ}\cdot\text{kg}^{-1}$, it can be inferred that the red regions correspond to HHV values exceeding 22 $\text{MJ}\cdot\text{kg}^{-1}$. Less than 21 $\text{MJ}\cdot\text{kg}^{-1}$ is the

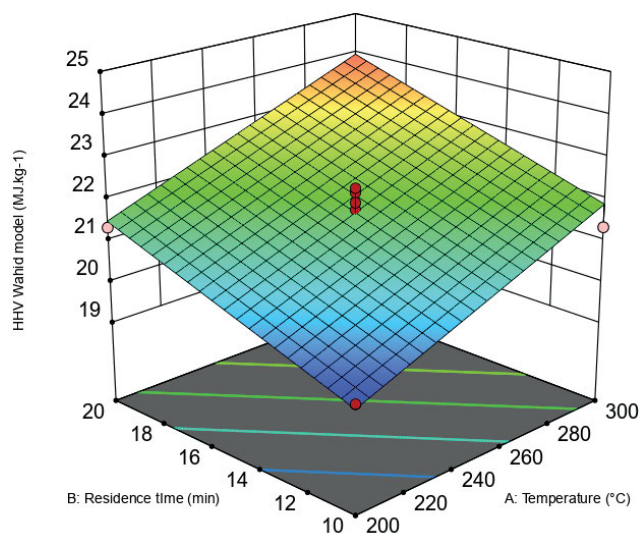


Fig. 1. Surface plot of Wahid model; source: own study

HHV value shown by the blue area. The plot's centre contains a green band that represents HHV levels between 21 and 22 $\text{MJ}\cdot\text{kg}^{-1}$. Given that the contour lines seem to be slightly bent, it is possible that the relationship is not entirely linear (Martín-Pascual *et al.*, 2020; Yun *et al.*, 2021). This suggests that at higher temperatures and longer residence times, the chemical reactions that occur in torrefied kesambi leaves are amplified (Nhuchhen, Basu and Acharya, 2014).

NHUCHHEN MODEL RESPONSE

The F -value of the Nhuchhen model, which is 27.53, indicates the overall statistical significance for the regression model. The regression model provides a good match to the observed data, as indicated by the low lack-of-fit value of 0.43. The independent variables in the model explain around 95% of the variability seen in the dependent variable, with a coefficient of determination (R^2) of 0.95. With the sample size and number of predictors taken into consideration, the estimated adjusted R^2 is 0.92. It still demonstrates, though, that the model penalises for overfitting while offering a decent match to the data. The model's ability to predict future observations properly is indicated by the predicted R^2 score of 0.86. With a score of 16.22, the model is considered to have an adequate precision and a decent signal-to-noise ratio. This implies that within the experimental zone, it can produce predictions with reasonable accuracy.

$$HHV \text{ Nhuchhen} = 29.73 + 1.64x_1 + 2.37x_2 - 0.23x_1x_2 - 0.11x_1^2 - 1.01x_2^2 \quad (5)$$

The projected HHV of torrefied kesambi leaves is represented by the surface colour, according to the surface plot of the Nhuchhen model, which is used to predict this feature (Fig. 2). Higher HHV values are shown in red and are located in the plot's upper left corner, following the colour-coding. Lower HHV values are displayed in blue and are located in the plot's lower right corner. This plot displays the precise range of HHV values (21.95–28.27 $\text{MJ}\cdot\text{kg}^{-1}$), plotting of which suggests that HHV values higher than 26 $\text{MJ}\cdot\text{kg}^{-1}$ are represented by the red area of the plot. An HHV value of <26 $\text{MJ}\cdot\text{kg}^{-1}$ is shown by the blue region.

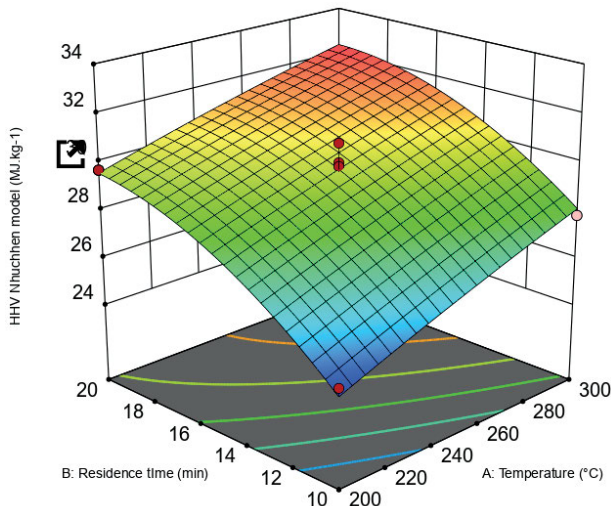


Fig. 2. Surface plot of Nhuchhen model; source: own study

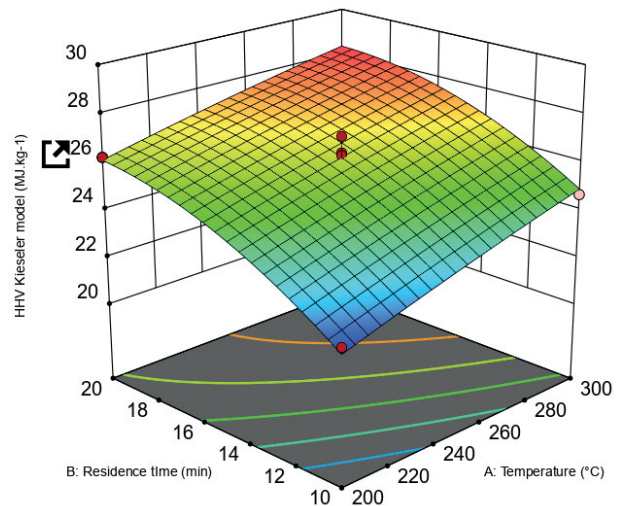


Fig. 3. Surface plot of Kieseler model; source: own study

KIESELER MODEL RESPONSE

The regression model appears to be statistically significant when considered in its entirety, as indicated by the Kieseler model’s *F*-value of 27.47. The regression model appears to match the observed data well, as indicated by its low lack-of-fit value of 0.41. With an R^2 of 0.95, the independent variables in the model can account for around 95% of the variability in the dependent variable.

Although the adjusted R^2 , which accounts for sample size and predictor count, is slightly lower at 0.92, it still indicates that the model provides a strong fit to data, even after considering model complexity.

With a predicted R^2 of 0.86, the model is highly accurate in projecting future observations. The model has a sufficient signal-to-noise ratio, indicating that it can be used to produce accurate predictions within the experimental range, according to the value of 16.19 for adequate precision. Based on these measures, it appears that the regression model is reliable, accounting for a significant amount of data variation, and capable of making accurate predictions about the results of the experiment under the given parameters.

$$HHV \text{ Kieseler} = 26.22 + 1.29x_1 + 1.89x_2 - 0.18x_1x_2 - 0.09x_1^2 - 1.79x_2^2 \quad (6)$$

The scatter plot (Fig. 3) illustrates the predicted versus actual values for the *HHV* based on Kieseler model. The plotted model values range from 24.36–32.31 MJ.kg⁻¹. The hue of the data points show the *HHV* Wahid model’s anticipated value. Higher expected *HHV* levels are shown by red points, while lower predicted values are shown in blue. The figure has a variety of hues, which indicate a range of predicted *HHV* values.

Desirability values of the Wahid, Nhuchhen, and Kieseler models used for predicting the water content are all equal to 1 (Fig. 4). This indicates that all three models accurately predict the water content in this scenario. A composite desirability score are based on the three models and four attributes. In this instance, the combined desirability value is 0.98, the lowest value in the table. This shows that while all models predict individual properties adequately, it might not fully capture the correlations between *HHV* and all four properties.

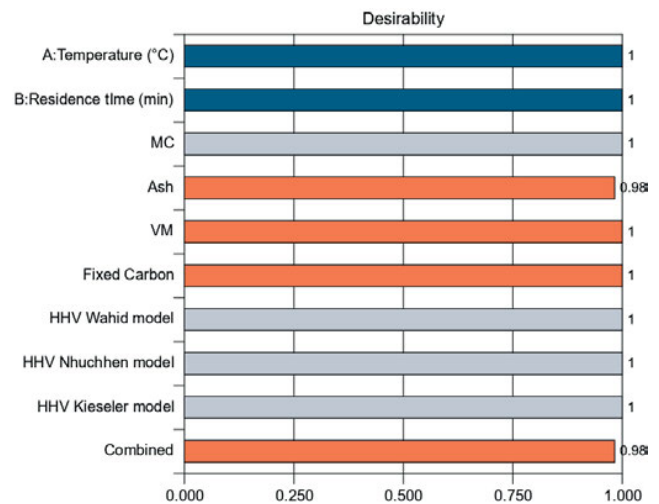


Fig. 4. Desirability of response models; MC = moisture content, VM = volatile matter, ASH = amount of ash, HHV = higher heating value; source: own study

CONCLUSIONS

In conclusion, this study provides a significant contribution to the understanding and optimisation of the torrefaction process for kesambi leaves to enhance their energy density as biofuel. Through experimental torrefaction and proximate analysis, predictive models for *HHV* were developed using statistical methods such as response surface methodology with central composite design. Three models, Wahid, Nhuchhen, and Kieseler, demonstrated overall significance and good predictive ability for *HHV*, offering valuable insights into optimal torrefaction conditions. The link between *HHV*, temperature, and residence time was shown using surface plots, which helped determine the ideal process parameters. While the models generally performed well in predicting *HHV*, there were some discrepancies between predicted and actual values, indicating areas for improvement. Desirability analysis highlighted opportunities to enhance correlations between *HHV* and measured properties.

This research provides a novel approach to predicting *HHV* in kesambi leaf-based biofuels, offering practical implications for

optimising torrefaction processes and improving energy density. Further research could focus on refining predictive models and investigating additional factors influencing HHV to advance the development of sustainable biofuel sources.

CONFLICT OF INTERESTS

The author declares no conflict of interests.

REFERENCES

- Abdul Wahid, F.R.A., Saleh, S. and Abdul Samad, N.A.F. (2017) "Estimation of higher heating value of torrefied palm oil wastes from proximate analysis," *Energy Procedia*, 138, pp. 307–312. Available at: <https://doi.org/10.1016/j.egypro.2017.10.102>.
- Ahiduzzaman, M. and Sadrul Islam, A.K.M. (2016) "Assessment of rice husk briquette fuel use as an alternative source of woodfuel," *International Journal of Renewable Energy Research*, 6(4). Available at: <https://doi.org/10.20508/ijrer.v6i4.4854.g6948>.
- Alhinai, M. et al. (2018) "Characterisation and thermochemical conversion of rice husk for biochar production," *International Journal of Renewable Energy Research*, 3, pp. 1648–1656. Available at: <https://www.ijrer.org/ijrer/index.php/ijrer/article/view/8054> (Accessed: Jun 24, 2024).
- Bhandari, A. (2020) *Difference between R-squared and adjusted R-squared*. Analytics Vidhya. Available at: <https://www.analyticsvidhya.com/blog/2020/07/difference-between-r-squared-and-adjusted-r-squared/> (Accessed: Jun 24, 2024).
- Dethan, J. and Lalel, H. (2024) "Optimization of particle size of torrefied kesambi leaf and binder ratio on the quality of bio-briquettes," *Journal of Sustainable Development of Energy, Water and Environment Systems*, 12(1), 1120490. Available at: <https://doi.org/10.13044/j.sdewes.d12.0490>.
- Dethan, J.J.S. (2023) "Optimalisasi suhu dan waktu tinggal pada proses torefaksi daun kesambi (*Schleichera oleosa*)," *Seminar Nasional Politani Kupang Ke-6 Kupang*. Available at: <https://ejurnal.politanikoe.ac.id/index.php/psnp/article/view/293/229> (Accessed: Jun 24, 2024).
- Dethan, J.J.S. et al. (2024) "Characteristics of kesambi leaf torrefaction biomass," *AIP Conference Proceedings*, 3073, 050016. Available at: <https://doi.org/10.1063/5.0193717>.
- Dimiyati, T.T. and Kurniasih, D. (2020) "Financial analysis of establishing micro industry of corn cobs briquettes in Majalengka Regency," *International Journal of Renewable Energy Research*, 10(1). Available at: <https://doi.org/10.20508/ijrer.v10i1.10382.g7856>.
- Dong, C., Li, G. and Feng, X. (2019) "Lack-of-fit tests for quantile regression models," *Journal of the Royal Statistical Society. Series B: Statistical Methodology*, 81(3), pp. 629–648. Available at: <https://doi.org/10.1111/rssb.12321>.
- Etemadi, S. and Khashei, M. (2021) "Etemadi multiple linear regression," *Measurement Journal of the International Measurement Confederation*, 186, 110080. Available at: <https://doi.org/10.1016/j.measurement.2021.110080>.
- Frost, J. (2019) *How to interpret adjusted R-squared and predicted R-squared in regression analysis*. Statistics By Jim. Available at: <https://statisticsbyjim.com/regression/interpret-adjusted-r-squared-predicted-r-squared-regression/> (Accessed: Jun 25, 2024).
- García, R. et al. (2013) "Biomass proximate analysis using thermogravimetry," *Bioresource Technology*, 139, pp. 1–4. Available at: <https://doi.org/10.1016/j.biortech.2013.03.197>.
- Hwangdee, P. et al. (2021) "Physical characteristics and energy content of biomass charcoal powder," *International Journal of Renewable Energy Research*, 11(1). Available at: <https://doi.org/10.20508/ijrer.v11i1.11658.g8122>.
- Kieseler, S., Neubauer, Y. and Zobel, N. (2013) "Ultimate and proximate correlations for estimating the higher heating value of hydrothermal solids," *Energy and Fuels*, 27(2), pp. 908–918. Available at: <https://doi.org/10.1021/ef301752d>.
- Majamo, S.L. and Amibo, T.A. (2023) "Modeling and optimization of chemical-treated torrefaction of wheat straw to improve energy density by response surface methodology," *Biomass Conversion and Biorefinery*, 14, pp. 21213–21227. Available at: <https://doi.org/10.1007/s13399-023-04192-y>.
- Mardyaningsih, M., Leki A. and Engel, S.S. (2016) "Teknologi Pembuatan Liquid Smoke Daun Kesambi sebagai Bahan Pengasapan Se'i Ikan Olahan Khas Nusa Tenggara Timur [Liquid Smoke Making Technology from Kesambi Leaves as a Smoking Ingredient for Se'i Fish, a Typical Processing of East Nusa Tenggara]," in *Prosiding Seminar Nasional Teknik Kimia "Kejuangan"*, Yogyakarta, 17 Mar 2016. Yogyakarta: UPN Veteran Yogyakarta. pp. 1–6. Available at: <http://jurnal.upnyk.ac.id/index.php/kejuangan/article/view/1589/1463> (Accessed: Apr 10, 2024).
- Martín-Pascual, J. et al. (2020) "Determination of the optimal operative conditions for the torrefaction of olive waste biomass," *Sustainability*, 12(16), 6411. Available at: <https://doi.org/10.3390/SU12166411>.
- Nhuchhen, D., Basu, P. and Acharya, B. (2014) "A comprehensive review on biomass torrefaction," *International Journal of Renewable Energy*, 2014, 506376. Available at: <https://doi.org/10.5171/2014.506376>.
- Nhuchhen, D.R. and Afzal, M.T. (2017) "HHV predicting correlations for torrefied biomass using proximate and ultimate analyses," *Bioengineering*, 4(1), 7. Available at: <https://doi.org/10.3390/bioengineering4010007>.
- Orisaley, J.I. et al. (2022) "Investigation of the effects of torrefaction temperature and residence time on the fuel quality of corncobs in a fixed-bed reactor," *Energies*, 15(14), 5284. Available at: <https://doi.org/10.3390/en15145284>.
- Oyebode, W.A. and Ogunsuyi, H.O. (2021) "Impact of torrefaction process temperature on the energy content and chemical composition of stool tree (*Alstonia congenisis* Engl) woody biomass," *Current Research in Green and Sustainable Chemistry*, 4, 100115. Available at: <https://doi.org/10.1016/j.crgsc.2021.100115>.
- Torres Ramos, R. et al. (2023) "Torrefaction under different reaction atmospheres to improve the fuel properties of wheat straw," *Processes*, 11(7), 1971. Available at: <https://doi.org/10.3390/pr11071971>.
- Yun, H. et al. (2021) "Identification of suitable biomass torrefaction operation envelopes for auto-thermal operation," *Frontiers in Energy Research*, 9. Available at: <https://doi.org/10.3389/fenrg.2021.636938>.