

Analytical pyrolysis of microwave-pretreated *Eucalyptus nitens*: Influence of pretreatment on the pyrolysis compounds obtained

Diego Venegas-Vásquez⁽¹⁾ (dvenegas@ubiobio.cl), Luis Tipanluisa-Sarchi⁽²⁾ (luis.tipanluisa@esepoch.edu.ec), Lourdes Orejuela-Escobar⁽³⁾ (lorejuela@usfq.edu.ec), Víctor H. Guerrero⁽⁴⁾ (victor.guerrero@epn.edu.ec), Serguei Alejandro-Martín⁽¹⁾ (salejandro@ubiobio.cl)

⁽¹⁾ Universidad del Bío-Bío; Departamento de Ingeniería de Maderas

⁽²⁾ Escuela Superior Politécnica de Chimborazo; Facultad de Mecánica

⁽³⁾ Universidad San Francisco de Quito; Departamento de Ingeniería Química

⁽⁴⁾ Escuela Politécnica de Nacional; Departamento de Materiales

ABSTRACT: *In this work, analytical pyrolysis is applied to Eucalyptus nitens (EN) previously treated by microwave irradiation (MW). Analysis of variance (ANOVA) was used to determine the most influential factor during pretreatment. The chemical composition of untreated and MW-treated EN was determined. Surface variations produced by the pretreatment were observed with scanning electron microscopy (SEM). Pyrolysis coupled with gas chromatography/mass spectrometry (Py-GC/MS) allowed for the identification of chemical compounds. It was determined that the irradiation power significantly affects the average and maximum temperatures reached by the MW-pretreated samples, while the irradiation time more significantly impacts moisture loss. The pretreatment affects the formation of extractables, which increase from 1.8% (untreated) to 5.9% (700 W – 5 min of MW). A decrease in oxygenated compounds, such as acids from 27.71% to 25.94% w.w⁻¹ and phenols from 23.40% to 20.59% w.w⁻¹, was observed from the untreated condition to the 700 W-5 min condition. These results demonstrate the positive influence of MW pretreatment on the reduction of oxygenated compounds obtained from biomass pyrolysis.*

KEYWORDS: *Biomass pyrolysis, Microwave pretreatment, Oxygenated compounds.*

Pirólise analítica de *Eucalyptus nitens* pré-tratado com micro-ondas: Influência do pré-tratamento nos compostos obtidos na pirólise

RESUMO: *Neste trabalho, a pirólise analítica é aplicada ao Eucalyptus nitens (EN) previamente tratado por irradiação de micro-ondas (MW). A análise de variância (ANOVA) foi utilizada para determinar o fator mais influente durante o pré-tratamento. A composição química do EN não tratado e tratado com MW foi determinada. Variações de superfície produzidas pelo pré-tratamento foram observadas com microscopia eletrônica de varredura (MEV). A pirólise acoplada à cromatografia gasosa/espectrometria de massa (Py-GC/MS) permitiu a identificação de compostos químicos. Determinou-se que a potência de irradiação afeta significativamente as temperaturas média e máxima atingidas pelas amostras pré-tratadas com MW, enquanto o tempo de irradiação impacta mais significativamente a perda de umidade. O pré-tratamento afeta a formação de extraíveis, que aumentam de 1,8% (não tratado) para 5,9% (700 W - 5 min de MW). Observou-se uma diminuição nos compostos oxigenados, como ácidos de 27,71% para 25,94% w.w⁻¹ e fenóis de 23,40% para 20,59% w.w⁻¹, da condição não tratada para a condição de 700 W - 5 min. Esses resultados demonstram a influência positiva do pré-tratamento com MW na redução de compostos oxigenados obtidos da pirólise de biomassa.*

PALAVRAS-CHAVE: *Pirólise de biomassa, Pré-tratamento com micro-ondas, Compostos oxigenados.*

1. INTRODUCTION

The short- and medium-term availability of petroleum-based energy and industrial resources and products is becoming increasingly limited (Xiu & Shahbazi, 2012). This situation has prompted researchers to seek new alternatives to fossil fuels, identifying renewables (geothermal, solar, wind, hydroelectric, and biomass) as potential sources of energy and valuable resources (Ahmed et al., 2021). Lignocellulosic biomass, a category of energy products derived from organic matter produced through biological means, is the most abundant raw material available on earth for the production of sustainable biofuels and high-value industrial products (Azevedo et al., 2019). It is primarily composed of cellulose, hemicellulose, lignin, and to a lesser extent, ash, proteins, extractives, and inorganic compounds; additionally, in its raw state, it contains significant amounts of water (Amit et al., 2018).

The thermal decomposition of biomass leads to the breaking of bonds that can transform into high-value-added products (Parihar et al., 2007). One thermochemical conversion process is pyrolysis, which, in the absence of oxygen, can produce more than 300 organic compounds, mainly condensable gases, biochar, and non-condensable gases (Roy & Dias, 2017). One of the challenges in biomass pyrolysis is the low calorific value of the resulting products (16.7-19 MJ/kg compared to 40-45 MJ/kg for fossil fuels), which is mainly due to the high-water content of the raw material (Chen et al., 2013). A pretreatment that minimizes the moisture content of the raw material could become a critical step for converting lignocellulosic biomass into sustainable energy products and could also deconstruct the biomass cell wall organization (cellulose, hemicellulose, and lignin), facilitating its conversion processes (Ahmed et al., 2022).

Biomass microwave drying pretreatment is gaining popularity due to advantages such as rapid, selective, volumetric, uniform heating, easy control, operation at low temperatures, energy savings in the process, and portable equipment (Cao et al., 2017). Lignocellulosic biomass is a material that absorbs MW converting it into heat energy internally (Huang et al., 2016). MW heating induces disruption in lignocellulosic biomass through molecular collision caused by dielectric polarization; this MW radiation accelerates chemical, biological, and physical processes, generating heat due to the vibration of polar molecules and ionic movement (Bhutto et al., 2017). This movement of polar molecules results in rapid heating, leading to shorter processing times compared to other heating methods (Aguilar-Reynosa et al., 2017). The effectiveness of MW irradiation on lignocellulosic biomass depends on several factors, with the most important being irradiation power and time, and the chemical composition of the biomass (Saini et al., 2015). Increasing the MW irradiation time and power produces vibration and forms hot spots within the biomass, which removes water, reorients and fractures the crystalline chains of cellulose, and breaks various bonds in hemicellulose and lignin, potentially altering the chemical composition of the treated biomass (Bichot et al., 2020).

Pyrolysis products can be non-condensable gases, condensable gases, and solids. Condensable gases generally contain a high-water content and hundreds of organic components such as acids, alcohols, ketones, furans, phenols, ethers, esters, sugars, aldehydes, alkenes, nitrogen, and also oxygenated compounds (Hu & Gholizadeh, 2019). In previous studies, we have investigated the influence of MW pretreatment on the pyrolysis of *Pinus radiata* and *Eucalyptus globulus*. The pretreatment affected the chemical composition of the biomass, reduced moisture content, and decreased oxygenated compounds during pyrolysis.

This study evaluates the yields of analytical pyrolysis products from *Eucalyptus nitens* (EN) originating from the Bío-Bío Region in Chile. The EN was pretreated with MW, and the influence of the pretreatment on its chemical composition and the compounds and compound families during its analytical pyrolysis was investigated.

2. MATERIALS AND METHODS

a. Materials

The Investigaciones Forestales Bioforest S.A. company from Bío Bío, Chile, provided *Eucalyptus nitens* samples as chips. The chips were exclusively made of wood without bark, and the position of the sample within the trunk was not considered (sapwood/heartwood). Samples were ground and sieved to achieve an average particle size of 0.33 mm and stored in a desiccator until further use, according to the procedure reported elsewhere (Venegas-Vásquez et al., 2024).

b. Methods

i. Microwave pretreatment

The biomass was irradiated in a Thomas TH-18B03 microwave oven (230 V, 50 Hz, operating frequency 2450 MHz). The irradiation power levels were 259, 462, 595, and 700 W, with irradiation times of 1, 2, 3, and 5 minutes. The average temperature was obtained using an Optris PI160 infrared camera (320x240 pixels) and the PI Connect software. The temperature values correspond to the average temperature recorded at six points in the biomass at the end of the microwave application. Mass loss was recorded on a Boeco digital balance (BAS 31 Plus) with a resolution of 1×10^{-4} g, and the moisture content of the samples was measured on an XY200MW thermobalance with a resolution of 1×10^{-3} g, according to the procedure reported elsewhere (Venegas & Alejandro-Martín, 2021)

ii. Statistical analysis

Statistical analysis was performed using Design Expert software, with a categorical multilevel experimental design used to evaluate the factors with the greatest influence on the response

variables, as shown in Table 1. The tests were conducted in triplicate, and statistical analysis was carried out in Design Expert with a significance level of $\alpha=0.05$.

TABLE 1. Factors and Variables in Experimental Design

Raw material	Power (W)	Time (min)	Response Variables
<i>Eucalyptus nitens</i>	259	1	T_{avg} (°C)
	462	2	T_{max} (°C)
	595	3	Humidity loss (%)
	700	5	

Source: Own Authorship (2024).

iii. Biomass chemical composition

To separate the extractives from the raw material, 300-400 mg of ground wood sieved between 45-60 mesh is required. The procedure is performed in a Soxhlet apparatus with 90% acetone for 16 hours according to TAPPI T 280 pm-99 (2015) and is connected to a constant water flow, following the procedure reported by Cruz et al. [45]. To quantify the extracted compounds, approximately 200 μ L of extractable material was mixed with 100 μ L of pyridine (1.5 mg DMAP/mL of pyridine) and 200 μ L of BSTFA (10% TMCS) (Torri et al., 2013), and heated for 30 minutes at 60 °C under a flow of N₂. Quantification was performed using GC-MS on an Agilent 7890A column with helium as the carrier gas. Holocellulose is obtained by oxidation of the extractive-free wood according to TAPPI T 9 m-54 (2015), as previously reported by Aguayo et al. (Aguayo et al., 2010). Once the percentage of holocellulose is obtained, insoluble alpha-cellulose is quantified using a concentrated solution of 17.5% w/v NaOH, and the percentage of hemicellulose is determined by difference. Finally, the percentage of lignin is determined using the Klason method according to TAPPI T22 om-06 (2006), as described previously by Mendonça et al. (Mendonça et al., 2008). The mass of insoluble lignin was determined by filtration, and soluble lignin was determined by UV spectroscopy. The total lignin content was obtained by summing the insoluble and soluble lignin.

iv. Statistical analysis

The changes in the biomass surface resulting from microwave pretreatment (MWI) were analyzed using a JEOL JSM-6610LV scanning electron microscope (SEM) (JEOL, Tokyo, Japan) operating at 5 kV and with a magnification of 500X.

v. Pyrolysis analysis

The pyrolysis was conducted at a temperature of 550°C using a CDS Analytical Pyroprobe 5200 HPR coupled with gas chromatography (Clarus 690) and mass spectrometry (SQ8), following a previously reported procedure (Alejandro-Martín et al., 2019). A sample size of 0.5 mg and hydrogen (Indura 99.999%) as the carrier gas were used. The identification of compounds was performed using the NIST 2017 library and TurboMass 6.1.0 software. Analytical pyrolysis was applied to both extractive-containing and extractive-free samples.

3. RESULTS

i. Microwave pretreatment

The effectiveness of the microwave irradiation pretreatment depends on the following factors: irradiation power and time, and biomass composition (Saini et al., 2015). Table 2 shows the recorded average temperature (T_{avg}), maximum temperature (T_{max}), and mass loss (M_{loss}) for EN samples, according to the irradiation power and time. Drying is a thermal process to remove water from the biomass, occurring between ambient temperature and 150 °C (S. Wang et al., 2017). As the power and irradiation time increase, the average temperature, maximum temperature, and mass loss increase proportionally. The highest temperature differentials are obtained at higher powers and irradiation times, as demonstrated by Oliveira & Franca (Oliveira & Franca, 2002) in their study of the effects of microwave power, irradiation time, and moisture content in barley, canola, and wheat. Manickavasagan et al. (Manickavasagan et al., 2006) also concluded that temperature differentials were greater at higher processing times due to the increased energy delivered. Since the water molecule present in the biomass is polar, it absorbs microwave energy very easily, generating collisions, vibration, and increased internal kinetic energy, resulting in a more pronounced increase in the biomass temperature (X. Wang et al., 2008).

TABLE 2. Microwave pretreatment

Time (min)	259 W			462 W			595 W			700 W		
	T_{avg}	T_{max}	M_{loss}	T_{avg}	T_{max}	M_{loss}	T_{avg}	T_{max}	M_{loss}	T_{avg}	T_{max}	M_{loss}
1	59.3	65.9	0.65	75.2	83.0	1.08	77.3	87.6	1.21	83.7	90.3	1.73
2	64.5	69.8	1.26	81.7	89.1	1.86	84.2	89.2	2.15	88.1	93.7	3.17
3	67.4	72.1	2.54	89.8	95.2	3.15	95.6	104.0	3.98	98.6	123.9	4.46
5	71.6	77.5	3.11	104.9	112.0	3.74	103.5	117.0	4.57	106.8	143.0	4.98

Source: Own Authorship (2024)

ii. Statistical analysis

Table 3 presents the ANOVA results (F-value) for the selected factors: power and irradiation time, and their interaction for EN. The presented values correspond to a p-value < 0.05 , indicating that all the models are significant. The standard deviation and R^2 values are also shown. The ANOVA analysis for all models across all biomass species indicates that they are significant. The response variables T_{avg} and T_{max} are the most significant for irradiation power, while time is the most significant variable for M_{loss} . There is greater standard deviation in T_{avg} compared to T_{max} for EN. The interaction between the two factors is significant for all models except for T_{avg} . The R^2 values for all models are above 80%, indicating that the models are robust. Therefore, it can be confirmed that the variation in power is the most significant factor affecting T_{avg} and T_{max} achieved during EN microwave pretreatment. This is why subsequent characterizations and analyses were performed considering the minimum and maximum power at the maximum irradiation time ((259 and 700 W) and 5 min).

TABLE 3. ANOVA (F-Value) for EN

Raw material	F-Value		
	T_{avg}	T_{max}	M_{loss}
Model	44.79	46.96	110.05
Power	55.86	130.11	22.65
Time	33.73	91.26	417.50
Power-Time	NA	9.47	3.36
Std. Dev.	5.94	5.15	0.22
R^2	0.87	0.95	0.98

Source: Own Authorship (2024).

iii. Chemical composition

Saini et al. (Saini et al., 2015) indicate that the effectiveness of microwave irradiation on lignocellulosic biomass depends, among other factors, on the chemical composition of the biomass. Table 4 shows the chemical composition of EN before and after MW treatment. The interactions between electromagnetic waves and matter are expressed as conductive losses, dielectric losses, and magnetic losses (Sun et al., 2016). The alignment of polar molecules caused by microwave irradiation forces the dipoles of the molecules to align in the radiation field (polarised). The rearrangement of the polar molecules produces a displacement within the material that generates heat (Motasemi & Afzal, 2013). In the maximum condition (700 W-5 min), the cellulose content decreased after pretreatment. Such variation can be associated with microwave irradiation that leads to intense molecular collisions due to dielectric polarisation, potentially decomposing the cellulose content

(Mankar et al., 2021). Then, the cellulose structure’s hydroxyl groups (-OH) result in a polar molecule. Then, as the microwaves pass through these polar molecules, they realign and vibrate them, increasing their internal temperature and displacement (Aguilar-Reynosa et al., 2017). In this condition, the lignin content decreased after pretreatment. The glass transition temperature of lignin (130–200 °C) is usually lower than that of cellulose (230–250 °C) and close to hemicellulose (160–200 °C) under a dry state (Tsubaki & Azuma, 2011); therefore, the decrease in this component may be associated with the initiation of decomposition due to reaching its glass transition temperature, which results in the disorder of the lignin structure. Additionally, under these circumstances, new extractives can be generated when the wood is thermally treated due to the degradation of other components, such as lignin (Li et al., 2016). This generation of new extractives is reflected in its increase.

TABLE 4. Chemical composition for EN

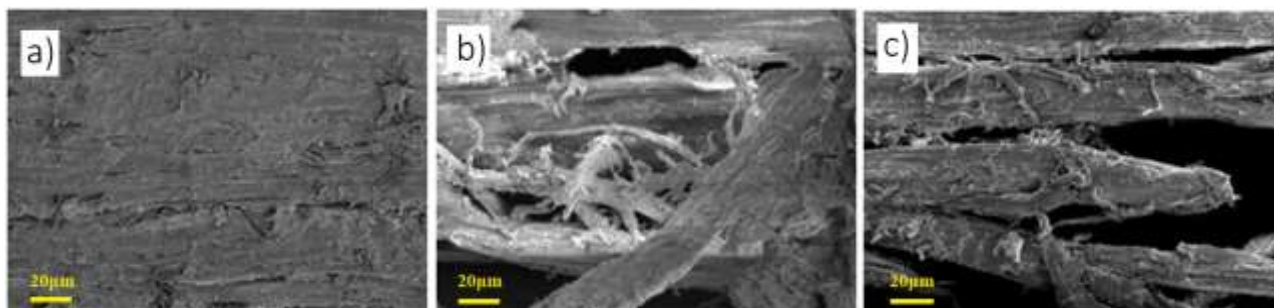
MW treatment	Hemicellulose	Cellulose	Lignin	Extractives
-	23.4	51.5	23.3	1.8
259 W – 5 min	23.5	51.0	22.4	3.1
700 W – 5 min	22.0	49.9	22.2	5.9

Source: Own Authorship (2024).

iv. Surface Morphology

The SEM images with different treatments are shown in Fig. 1 and are used to analyze the effect of microwave pretreatment on the surface structure of EN. It can be observed that the surfaces remain in the form of compact blocks and the structure appears intact. As microwave irradiation at 259 W and 5 min is applied, the structures begin to crack slightly. Under conditions of 700 W and 5 min, the fragmentation is very pronounced due to microwave irradiation, with some fragments visibly detached from the biomass surface. These results are similar to those obtained by Liang et al. (Liang et al., 2019) in pine sawdust treated with microwaves prior to pyrolysis, and by Amini (Amini et al., 2018) in microwave pretreatment prior to enzymatic saccharification of eucalyptus sawdust. Microwaves act by heating biomass rapidly and locally, making it more porous and with increased surface area due to the cleavage of the raw material, resulting in greater availability for subsequent transformation into commercially valuable products (Cantero et al., 2019).

FIGURE 1. SEM images before and after EG MW treatment.

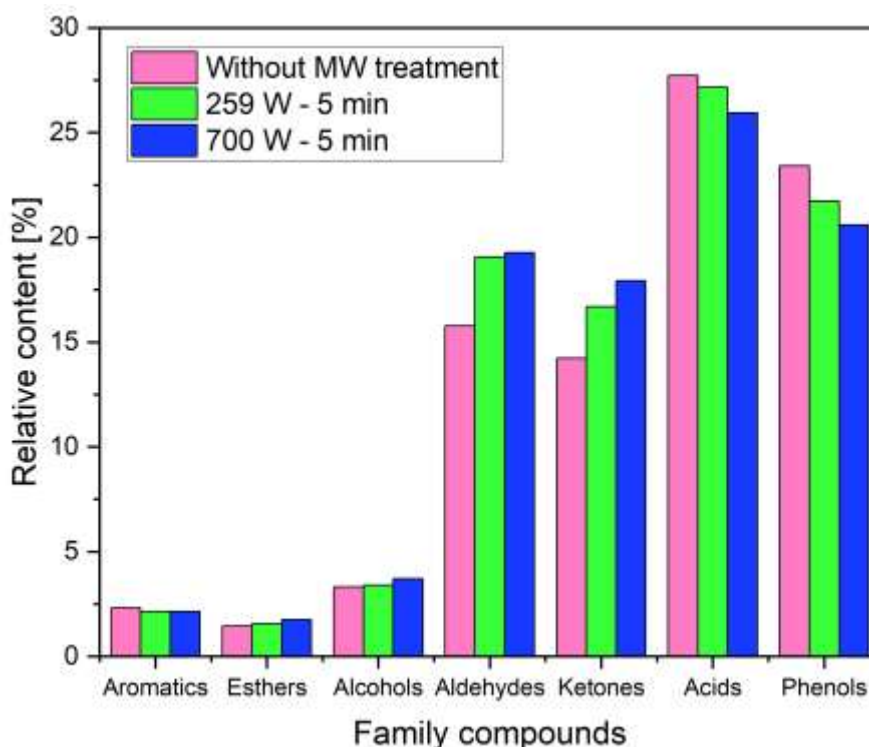


Source: Own Authorship (2024).

v. Analytical pyrolysis

The identified compound families (Figure 2) constitute the typical families of compounds formed during pyrolysis: oxygenates (acids, esters, alcohols, ketones, aldehydes), phenols, and aromatic compounds, as previously reported (Venegas-Vásquez et al., 2023). During microwave treatment, the cellulose contents were slightly reduced, due to the thermal decomposition of this component caused by the passage of microwaves (Liang et al., 2019). This reduction in acidic compounds leads to secondary ketonisation reactions, resulting in condensable gases enriched in ketones and aldehydes. These results are consistent with the study of the thermal behavior of *Robinia pseudoacacia* conducted by Mészáros et al. (Mészáros et al., 2007). Depolymerization reactions at the end of the polymer chain break the β bond to form levoglucosan and acetic acid (Piskorz et al., 1989). Then, dehydration, fragmentation, and condensation reactions form acids, furans, and aldehydes (Trubetskaya et al., 2021). Hemicellulose pyrolysis comprises decomposition and polymerization reactions, leading to the cleavage of glycosidic bonds, the formation of oligosaccharides, and their subsequent breakdown into xylose (Yogalakshmi et al., 2022). The second stage is the depolymerization of these intermediates, converting them into aromatic alcohols known as monolignols. Additionally, the decrease in lignin content led to a reduction in phenols, which is associated with depolymerization reactions and the cleavage of C-O and C-C bonds present in lignin due to the breakage of β -O-4 bonds in lignin as microwave power increases (Motasemi & Afzal, 2013). These organic compounds are then transformed into methoxyphenols by breaking the C=O and C=C bonds (Ren et al., 2012). As the irradiation power intensifies, the microwaves rearrange the lignin (Ma et al., 2009). This effect explains why the yield of the phenolic compounds formed decreased as the microwave power increased.

FIGURE 2. Family compounds formed during analytical pyrolysis of EN.



Source: Own Authorship (2024).

4. CONCLUSIONS

This work evaluated the effects of microwave pretreatment on the analytical pyrolysis of *Eucalyptus nitens*. The most significant variations in maximum temperature and mass loss were obtained for the 700 W-5 min pretreatment. Microwave pretreatment influenced the decrease in cellulose content due to the vibration and realignment of the (-OH) groups and the reduction in lignin content upon reaching its glass transition temperature. This lignocellulosic component's rearrangement increased extractives. The surface fragmentation of biomass structures was confirmed after microwave heating. The pyrolysis of pretreated biomass showed a reduction in oxygenated compounds: acids and phenols, demonstrating a positive influence of microwave pretreatment, finally favoring properties of potential liquid pyrolysis products. Therefore, the microwave pretreatment of *Eucalyptus nitens* is a promising technology for obtaining valuable compounds in pyrolytic processes.

ACKNOWLEDGMENTS

This research is funded by the Chilean National Research and Development Agency (Project Grant Number EQM 170077) and the University of Bío-Bío (Project Grant Number 2160341 IF/R). To the Escuela Politécnica Nacional EPN-Ecuador, with the research line: Sustainable Manufacturing in

Manufacturing Processes for the support provided in the preparation of this work. The authors would like to thank Francisco Medina from the Laboratorio de Cromatografía Gaseosa y Pirólisis Analítica of the University of Bío Bío, Chile, for his collaboration in the pyrolysis analyses. The authors acknowledge the cooperation of Karina Lagos from the Laboratorio de Nuevos Materiales at the Escuela Politécnica Nacional in Quito, Ecuador. The authors also want to thank María Graciela Aguayo, William Gacitúa and Judith Vergara from the Centro de Biotecnología y Nanotecnología (CBN) at the University of Bío Bío for their collaboration.

STATEMENT OF RESPONSIBILITY

The authors are solely responsible for this work.

REFERENCES

- Aguayo, M. G., Quintupill, L., Castillo, R., Baeza, J., Freer, J., & Mendonça, R. T. (2010). Determination of differences in anatomical and chemical characteristics of tension and opposite wood of 8-year old *Eucalyptus globulus*. *Maderas: Ciencia y Tecnologia*, 12(3), 241–251. <https://doi.org/10.4067/S0718-221X2010000300008>
- Aguilar-Reynosa, A., Romani, A., Ma. Rodríguez-Jasso, R., Aguilar, C. N., Garrote, G., & Ruiz, H. A. (2017). Microwave heating processing as alternative of pretreatment in second-generation biorefinery: An overview. *Energy Conversion and Management*, 136, 50–65. <https://doi.org/10.1016/j.enconman.2017.01.004>
- Ahmed, S. F., Liu, G., Mofijur, M., Azad, A. K., Hazrat, M. A., & Chu, Y. M. (2021). Physical and hybrid modelling techniques for earth-air heat exchangers in reducing building energy consumption: Performance, applications, progress, and challenges. *Solar Energy*, 216(February), 274–294. <https://doi.org/10.1016/j.solener.2021.01.022>
- Ahmed, S. F., Mofijur, M., Chowdhury, S. N., Nahrin, M., Rafa, N., Chowdhury, A. T., Nuzhat, S., & Ong, H. C. (2022). Pathways of lignocellulosic biomass deconstruction for biofuel and value-added products production. *Fuel*, 318(November 2021), 123618. <https://doi.org/10.1016/j.fuel.2022.123618>
- Alejandro-Martín, S., Acaricia, A. M., Cerda-Barrera, C., & Pérez, H. D. (2019). Influence of chemical surface characteristics of ammonium-modified chilean zeolite on oak catalytic pyrolysis. *Catalysts*, 9(5), 1–13. <https://doi.org/10.3390/catal9050465>
- Amini, N., Haritos, V. S., & Tanksale, A. (2018). Microwave assisted pretreatment of eucalyptus sawdust enhances enzymatic saccharification and maximizes fermentable sugar yield. *Renewable Energy*, 127, 653–660. <https://doi.org/10.1016/j.renene.2018.05.001>
- Amit, K., Nakachew, M., Yilkal, B., & Mukesh, Y. (2018). A review of factors affecting enzymatic hydrolysis of pretreated lignocellulosic Biomass. *Research Journal of Chemistry and Environment*, 22(7), 62–67.

- Azevedo, S. G., Sequeira, T., Santos, M., & Mendes, L. (2019). Biomass-related sustainability: A review of the literature and interpretive structural modeling. *Energy*, 171(2019), 1107–1125. <https://doi.org/10.1016/j.energy.2019.01.068>
- Bhutto, A. W., Qureshi, K., Harijan, K., Abro, R., Abbas, T., Bazmi, A. A., Karim, S., & Yu, G. (2017). Insight into progress in pre-treatment of lignocellulosic biomass. *Energy*, 122, 724–745. <https://doi.org/10.1016/j.energy.2017.01.005>
- Bichot, A., Lerosty, M., Radoiu, M., Méchin, V., Bernet, N., Delgenès, J. P., & García-Bernet, D. (2020). Decoupling thermal and non-thermal effects of the microwaves for lignocellulosic biomass pretreatment. *Energy Conversion and Management*, 203(August 2019), 112220. <https://doi.org/10.1016/j.enconman.2019.112220>
- Cantero, D., Jara, R., Navarrete, A., Pelaz, L., Queiroz, J., Rodríguez-Rojo, S., & Cocero, M. J. (2019). Pretreatment processes of biomass for biorefineries: Current status and prospects. *Annual Review of Chemical and Biomolecular Engineering*, 10, 289–310. <https://doi.org/10.1146/annurev-chembioeng-060718-030354>
- Cao, X., Zhang, M., Fang, Z., Mujumdar, A. S., Jiang, H., Qian, H., & Ai, H. (2017). Drying kinetics and product quality of green soybean under different microwave drying methods. *Drying Technology*, 35(2), 240–248. <https://doi.org/10.1080/07373937.2016.1170698>
- Chen, D., Zheng, Y., & Zhu, X. (2013). In-depth investigation on the pyrolysis kinetics of raw biomass. Part I: Kinetic analysis for the drying and devolatilization stages. *Bioresource Technology*, 131, 40–46. <https://doi.org/10.1016/j.biortech.2012.12.136>
- Hu, X., & Gholizadeh, M. (2019). Biomass pyrolysis: A review of the process development and challenges from initial researches up to the commercialisation stage. *Journal of Energy Chemistry*, 39(x), 109–143. <https://doi.org/10.1016/j.jechem.2019.01.024>
- Huang, Y. F., Chiueh, P. Te, & Lo, S. L. (2016). A review on microwave pyrolysis of lignocellulosic biomass. *Sustainable Environment Research*, 26(3), 103–109. <https://doi.org/10.1016/j.serj.2016.04.012>
- Li, L., Wang, X., & Wu, F. (2016). Chemical Analysis of Densification, Drying, and Heat Treatment of Scots pine (*Pinus sylvestris* L.) through a Hot-Pressing Process. *BioResources*, 11(2), 3856–3874.
- Liang, J., Xu, X., Yu, Z., Chen, L., Liao, Y., & Ma, X. (2019). Effects of microwave pretreatment on catalytic fast pyrolysis of pine sawdust. *Bioresource Technology*, 293(August), 122080. <https://doi.org/10.1016/j.biortech.2019.122080>
- Ma, H., Liu, W. W., Chen, X., Wu, Y. J., & Yu, Z. L. (2009). Enhanced enzymatic saccharification of rice straw by microwave pretreatment. *Bioresource Technology*, 100(3), 1279–1284. <https://doi.org/10.1016/j.biortech.2008.08.045>
- Manickavasagan, A., Jayas, D. S., & White, N. D. G. (2006). Non-uniformity of surface temperatures of grain after microwave treatment in an industrial microwave dryer. *Drying Technology*, 24(12), 1559–1567. <https://doi.org/10.1080/07373930601030796>

- Mankar, A. R., Pandey, A., Modak, A., & Pant, K. K. (2021). Pretreatment of lignocellulosic biomass: A review on recent advances. *Bioresource Technology*, 334(March), 125235. <https://doi.org/10.1016/j.biortech.2021.125235>
- Mendonça, R. T., Jara, J. F., González, V., Elissetche, J. P., & Freer, J. (2008). Evaluation of the white-rot fungi *Ganoderma australe* and *Ceriporiopsis subvermisporea* in biotechnological applications. *Journal of Industrial Microbiology and Biotechnology*, 35(11), 1323–1330. <https://doi.org/10.1007/s10295-008-0414-x>
- Mészáros, E., Jakab, E., & Várhegyi, G. (2007). TG/MS, Py-GC/MS and THM-GC/MS study of the composition and thermal behavior of extractive components of *Robinia pseudoacacia*. *Journal of Analytical and Applied Pyrolysis*, 79(1-2 SPEC. ISS.), 61–70. <https://doi.org/10.1016/j.jaap.2006.12.007>
- Motasemi, F., & Afzal, M. T. (2013). A review on the microwave-assisted pyrolysis technique. *Renewable and Sustainable Energy Reviews*, 28, 317–330. <https://doi.org/10.1016/j.rser.2013.08.008>
- Oliveira, M. E. C., & Franca, A. S. (2002). Microwave heating of foodstuffs. *Journal of Food Engineering*, 53, 347–359.
- Parihar, M. F., Kamil, M., Goyal, H. B., Gupta, A. K., & Bhatnagar, A. K. (2007). An experimental study on pyrolysis of biomass. *Process Safety and Environmental Protection*, 85(5 B), 458–465. <https://doi.org/10.1205/psep07035>
- Piskorz, J., Radlein, D. S. A. G., Scott, D. S., & Czernik, S. (1989). Pretreatment of wood and cellulose for production of sugars by fast pyrolysis. *Journal of Analytical and Applied Pyrolysis*, 16(2), 127–142. [https://doi.org/10.1016/0165-2370\(89\)85012-0](https://doi.org/10.1016/0165-2370(89)85012-0)
- Ren, S., Lei, H., Wang, L., Bu, Q., Chen, S., Wu, J., Julson, J., & Ruan, R. (2012). Biofuel production and kinetics analysis for microwave pyrolysis of Douglas fir sawdust pellet. *Journal of Analytical and Applied Pyrolysis*, 94, 163–169. <https://doi.org/10.1016/j.jaap.2011.12.004>
- Roy, P., & Dias, G. (2017). Prospects for pyrolysis technologies in the bioenergy sector: A review. *Renewable and Sustainable Energy Reviews*, 77, 59–69. <https://doi.org/10.1016/j.rser.2017.03.136>
- Saini, A., Aggarwal, N. K., Sharma, A., & Yadav, A. (2015). Prospects for Irradiation in Cellulosic Ethanol Production. *Biotechnology Research International*, 2015, 1–13. <https://doi.org/10.1155/2015/157139>
- Sun, J., Wang, W., Yue, Q., Ma, C., Zhang, J., Zhao, X., & Song, Z. (2016). Review on microwave-metal discharges and their applications in energy and industrial processes. In *Applied Energy* (Vol. 175, pp. 141–157). Elsevier Ltd. <https://doi.org/10.1016/j.apenergy.2016.04.091>
- Torri, C., Soragni, E., Prati, S., & Fabbri, D. (2013). Py-SPME-GC-MS with on-fiber derivatization as a new solvent-less technique for the study of polar macromolecules: Application to natural gums. *Microchemical Journal*, 110, 719–725. <https://doi.org/10.1016/j.microc.2013.08.006>
- Trubetskaya, A., Hunt, A. J., Budarin, V. L., Attard, T. M., Kling, J., Surup, G. R., Arshadi, M., & Umeki, K. (2021). Supercritical extraction and microwave activation of wood wastes for enhanced syngas production and

generation of fullerene-like soot particles. *Fuel Processing Technology*, 212(May 2020), 106633. <https://doi.org/10.1016/j.fuproc.2020.106633>

Tsubaki, S., & Azuma, J.-I. (2011). Application of Microwave Technology for Utilization of Recalcitrant Biomass. *Advances in Induction and Microwave Heating of Mineral and Organic Materials*, 697–722. <https://doi.org/10.5772/14040>

Venegas, D., & Alejandro-Martín, S. (2021). Influencia del pretratamiento con microondas en la pirólisis analítica de biomasa lignocelulósica. *Encuentro Iberoamericano de Redes de Biomasa y Bioenergía*, 103–108. <https://doi.org/ISBN: 978-84-15413-45-5>

Venegas-Vásquez, D., Arteaga-Pérez, L. E., Aguayo, M. G., Romero-Carrillo, R., Guerrero, V. H., Tipanluisa-Sarchi, L., & Alejandro-Martín, S. (2023). Analytical Pyrolysis of Pinus radiata and Eucalyptus globulus: Effects of Microwave Pretreatment on Pyrolytic Vapours Composition. *Polymers*, 15(3790). <https://doi.org/10.3390/polym15183790>

Venegas-Vásquez, D., Orejuela-Escobar, L., Valarezo-Garcés, A., Guerrero, V. H., Tipanluisa-Sarchi, L., & Alejandro-Martín, S. (2024). Biomass Valorization through Catalytic Pyrolysis Using Metal-Impregnated Natural Zeolites: From Waste to Resources. *Polymers*, 16(13), 1912. <https://doi.org/10.3390/polym16131912>

Wang, S., Dai, G., Yang, H., & Luo, Z. (2017). Lignocellulosic biomass pyrolysis mechanism: A state-of-the-art review. *Progress in Energy and Combustion Science*, 62, 33–86. <https://doi.org/10.1016/j.pecs.2017.05.004>

Wang, X., Chen, H., Luo, K., Shao, J., & Yang, H. (2008). The influence of microwave drying on biomass pyrolysis. *Energy and Fuels*, 22(1), 67–74. <https://doi.org/10.1021/ef700300m>

Xiu, S., & Shahbazi, A. (2012). Bio-oil production and upgrading research: A review. *Renewable and Sustainable Energy Reviews*, 16(7), 4406–4414. <https://doi.org/10.1016/j.rser.2012.04.028>

Yogalakshmi, K. N., Poornima Devi, T., Sivashanmugam, P., Kavitha, S., Yukesh Kannah, R., Varjani, S., AdishKumar, S., Kumar, G., & Rajesh Banu, J. (2022). Lignocellulosic biomass-based pyrolysis: A comprehensive review. *Chemosphere*, 286. <https://doi.org/10.1016/j.chemosphere.2021.131824>