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The use of microwave pre-treatment of orange wastes for enhancing biodegradability: Optimisation studies

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ABSTRACT

Orange wastes are an example of a raw material, which pose a problem when used in biological processes. This is mainly due to the content of poorly biodegradable lignin, phenols, and essential oils, mainly d-limonene. For this reason, a new techniques are constantly being sought to enable their further utilisation, e.g. in the anaerobic digestion process. This study evaluated the effectiveness of microwave irradiation as a pre-treatment method allowing for improving biodegradability of orange wastes. The experiment was conducted at a temperature of T = 50 °C and input power of P = 900 W. The following time intervals were evaluated: 0, 3, 5, 10, 20 and 30 min. Among various analyses cases, the most beneficial results were found at the time of 5 min. Therein, the major growth of biodegradability index of 16.36% and higher solubilisation yield of 23.9% was achieved with moderate release of inhibitors. These operating conditions allowed for an increase in biogas and methane production by 8.9 and 10.4%, respectively, compared to untreated orange wastes.

Keywords: citrus wastes, solubilisation yield, biodegradability index, d-limonene, phenols, energy, biogas production.

INTRODUCTION

Growing energy demand and the need to ensure energy security focuses more attention on obtaining energy from renewable resources. The lignocellulosic biomass represented by agro-industrial and forestry residues might be applied as a promising renewable energy resource (Sawhney et al., 2023). Due to the worldwide prevalence of lignocellulosic biomass and the problem of their effective management, their utilisation in the anaerobic digestion process (AD) has gained more attention (Manyi-Loh et al., 2023). In this technology in the absence of free oxygen various organic wastes are degraded into a biogas an energy carrier (Uddin and Wright 2023; Alengebaw et al., 2024). Nevertheless, this technology includes multiple steps with complex interactions between different types of microorganisms. Therefore, process stability and biogas production might be easily inhibited. Anaerobic decomposition of lignocellulosic biomass still is a challenge, mainly due to complex structure of this biomass that is resistant for anaerobic decomposition. Such wastes are characterised by slow hydrolysis rate and formation of inhibitors, as well as nutrient imbalance (Agregán et al., 2022).

Among various lignocellulosic wastes, the residues generated in orange juice production constitute a major environmental issue. Globally, orange juice is one of the most popular beverages, in 2024–2025 its global production was established at 1.4 million tons, which represents an increase of 4% compared to previous years (USDA, 2025). Its industrial production contributes to the generation of substantial volume of residues. It is estimated that out of every kilogram of oranges, more than half becomes a waste (mainly peels, seeds and fibres) that is difficult to manage (Singh et al., 2025). Additionally, due to the significant moisture content, orange wastes (OW) are susceptible to bacterial growth (Lin et al., 2025).

Another problematic issue is related with the presence of essential oils, mainly d-limonene, indicating antibacterial properties, which might destabilise the degradation pathways of the AD process (Trujillo-Reyes et al., 2024).

Therefore, one of the possible solutions to overcome those difficulties is application of adequate pre-treatment strategy prior their anaerobic decomposition. However, to choose the appropriate method, a number of factors should be considered, such as: effective lignin elimination, prevention of sugar degradation, no generation of AD inhibitors, low energy consumption (Sawhney et al., 2023).

Recently, the application of microwave irradiation (MR) has been attracting increasing attention from researchers (Hoang et al., 2021). This technique has been applied for various substrates including municipal solid waste and lignocellulosic biomass resulting in improved accessibility and bioavailability of treated wastes due to effective destruction of complex organic matter, thus ensuring improved biogas production of difficult biodegradable substrates (Singht et al., 2024; Anoopkumar et al., 2023; Kumar and Verma 2025). In this technique, microwaves heat both the surface and the core of the treated biomass. Intermolecular collisions resulted by microwave oscillations realign polar molecules providing both thermal and non-thermal effects. However, the crucial factor in effective application is selection of operational parameters, such as power, time, adopted solvent and temperature. In comparison to other methods, this technology presents several advantages, such as rapid heat transfer, compactness of equipment, easy combination with other methods, as well as generation of a small amount of toxic by-products. However, due to significant investment costs, there are only few installations operating on a technical scale (Ethaib et al., 2024).

In this work, the optimisation studies pertaining to the microwave pre-treatment of OW for improving biodegradability has been performed. The criteria taken into account to select the most favourable variant were: the amount of energy needed for pre-treatment and the improvement of biodegradability index and generation of toxic intermediates. Additionally, both biogas and methane productions were determined for the most favourable variant achieved form optimisation studies to confirm the beneficial effect of microwave irradiation.

MATERIAL AND METHODS

Substrates

The oranges applied in the experiment were taken from local market, they were washed before use. The orange waste was obtained from a laboratory scale juicer, it mainly consisted of peels and fibres. Prior to the experiment, it was blended to the particle size up to 5 mm. The samples prepared in this manner were suspended in wastewater after primary treatment from the primary settling tank outflow. Wastewater, which was a medium for suspending the OW was taken from municipal wastewater treatment plant located in Lublin (Poland). The adopted dose of material was 21.3 g of fresh OW per 1 L of wastewater. In turn, the sewage sludge (SS) obtained from municipal wastewater treatment plant located in Lublin (Poland) was used in the studies on biogas and methane productions. It was a mixture of primary and secondary sludges mixed in a volumetric ratio of 60:40. The composition of all substrates is presented in Table 1.

Laboratory installation and operational set-up

To perform microwave pre-treatment an Advanced Microwave Digestion System provided by Ethos Easy (Milestone) has been engaged. This laboratory device consisted two 950 W magnetrons for a total power of 1900 W. This system is equipped with rotating diffuser that distributes microwave irradiation evenly throughout the cavity. Additionally, to maintain the adopted temperature range, infrared sensors combined with an in-situ temperature sensor were applied.

The adopted operational parameters of microwave pre-treatment were: temperature of T = 50 °C and input power of P = 900 W. The following time intervals were evaluated: 0, 3, 5, 10, 20 and 30 min, respectively.

For the most beneficial result obtained from the optimisation studies, biogas and methane productions were established using BioReactor Simulator provided by BPC Instruments. This device consists of six batch anaerobic reactors with a volume of 2 L, each. The experiment conducted under mesophilic conditions at a temperature of 37 °C and it lasted 21 days. The produced biogas was monitored using the integrated module. The detailed description and procedure of conducting

Table 1. The physico-chemical composition of substrates (averages values and standard deviation are given)

Parameter	Unit	MW	OP	SS
TS	g/kg	1.15± 0.03	240.5±4.6	41.28±3.74
VS	g/kg	0.30± 0.02	227.3±5.1	29.97±2.71
VS/TS	-	0.29	0.95	0.73

batch anaerobic digestion experiments is presented in authors' previous studies (Szaja et al., 2025). The feedstock to the reactors was a mixture of SS and pre-treated OW (reactor A). Moreover, to compare the obtained results the control reactor supplied by SS and raw OW (reactor B). The experiments were repeated three times under unchanged operational conditions, the tables and figures show average values with standard deviations.

Analytical methods

To evaluate the effectiveness of microwave pre-treatment on biodegradability of OW in raw and pre-treated samples the following parameters were controlled: COD (chemical oxygen demand), sCOD (soluble chemical oxygen demand), DOC (dissolved organic carbon) and TOC (total organic carbon), TS (total solids) and VS (volatile solids). Moreover, the presence of AD inhibitors, e.g. d-limonene and phenols as well as pH and temperature (T) were monitored in the samples.

The COD, sCOD and phenol content were measured by the means of a Hach Lange DR 3900 spectrophotometer involving standard cuvette test. In turn, TS and VS were controlled according to the standard methods (APHA, 2012). LECO RC 612 multiphase analyser (LECO Corporation) was applied for TOC analysis. The following measurement parameters were used: gas pressure of 40 psi, flow rate of 0.75 L/min. The samples were burned at 1000 °C. TOC-L analyser (Shimadzu) was used to establish the content of DOC. Prior to the analyses, the samples were filtered through a 0.45 µm membrane filter. This equipment applied the combustion catalytic oxidation method at temperature of 680 °C. The pH value and T were controlled using a HQD Hach Lange multiparameter meter.

The d-limonene content was measured by the means of an Agilent GC-MS 8890/5977b. The samples was diluted with MiliQ water in a 3:1 ratio. The sample in the amount of 1 g was extracted with GC-grade POCH dichloromethane. The prepared extract was passed using on a 0.45 mm Alfatec nylon filter. To conduct the analyses the following column was applied: Supelco Equity 5MS 30 m × 0.25 mm ID x 0.25 mm df.

The indicators used to assess the efficacy of the process were: biodegradability index (BI index), solubilisation yield and removal efficiency of TOC, TS and COD. The biodegradability index was expressed as DOC/TOC ratio. In turn, the solubilisation yield was calculated according to the following formula:

$$S_Y = \frac{sCOD_t - sCOD_r}{COD_r} \times 100\ [\%] \qquad (1)$$

where: $sCOD_t$ is soluble chemical oxygen dement in a pre-treated sample at specific time interval (mg/L), $sCOD_r$ is soluble chemical oxygen dement in a raw sample (mg/L), COD_r is total chemical oxygen demand in a raw sample (mg/L).

Removal efficiency was established based on following equation:

$$RE = \frac{P_r - P_t}{P_r} \times 100\ [\%] \qquad (2)$$

where: P_r is COD, TS or TOC contents in a raw sample, P_t is COD, TS or TOC contents in a pre-treated sample at specific time interval.

Additionally, the energy aspect has been investigated, the energy needed for pre-treatment was calculated using the relationship:

$$E = \frac{IP \times t}{m}\ [\text{MJ/kg}] \qquad (3)$$

where: IP is the power input of microwave device (W), t is the pre-treatment time (s), m is the amount of pre-treated OW (kg).

The biogas composition was determined using a ThermoTrace GC-Ultra gas chromatograph (Thermo Fisher Scientific, Milan, Italy).

RESULTS AND DISCUSSION

The results in terms organic compounds are presented in Figure 1. The use of microwave resulted in destruction of complex organic compounds expressed as COD, TS and TOC. As compared to the un-treated sample, COD was reduced by 10.3–15.5% (Figure 1a). The highest removal efficiency was found for time of 10 and 20 min, respectively. In terms of the TS content, the removal efficiency was varied between 10–13% (Figure 1b). The minor effect was observed in the case of the TOC content, for this parameter the removal efficiency was established at the level from 2 to 5%, with the highest value for the 10 min (Figure 1c).

Importantly, the release of soluble organic matter was achieved as result of MR. The concentration of sCOD was enhanced by 15–33% (Figure 1a). However, this growth was achieved only for the shorter times of 3, 5, and 10 min, respectively. The prolongation of time and hence providing more energy input did not resulted in improved solubilisation. It should be noticed that for the time of 10 min, a downward trend was noted. In turn, for longer times of 20 and 30 min, comparable results were recorded with the raw sample. The major improvement of 33% was achieved for the time of 5 min. The similar tendency was observed with regard to DOC (Figure 1c). For the time of 5 min, the major increase of DOC by approx. 14% content was found. In other cases, this parameter was improved by 8–10%. As in the case of sCOD, the prolongation of duration of the experiment the did not lead to increase release of soluble organic matter. The minor effect of MR was found with regard to VS; in all samples, comparable results were found in all cases (Figure 1b).

Those results corresponded to the achieved values of solubilisation yield, this parameter determines the amount of the total organic matter that is converted into a soluble form (Table 2). The highest value was found for the time of 5 min. This effect was also accompanied by significant improvement of BI index and sCOD/COD ratio. As compared to the raw sample, the BI index was improved by 16.36%, in turn sCOD/COD was enhanced by 36%. However, in this case the release of phenols occurred. For the time of 5 min, their contents were enhanced by 46%; however, the obtained value did not exceed the limit concentration considered as inhibitory for AD. Importantly, with regard to d-limonene, another toxic compound to AD microbes a significant drop of 70.5% was achieved.

It should be noticed that for the shortest time of 3 min, a minor release of phenol occurred, reaching approx. 8%; in turn, d-limonene was reduced by approx. 54%. Additionally, for this time – as compared to an un-treated sample – the BI index and sCOD/COD ratio were improved by 13% and 23%, respectively. Moreover, the observed effects were obtained for the lowest energy consumption. In turn, the prolongation of time to 20 and 30 min led to deterioration of results with regard to sCOD and S_Y. Additionally, those conditions promoted the formation of phenols, the release of these compounds reached a value of about 90%. In turn, with regard to d-limonene, comparable results were obtained as for the 5-minute time. It should also be noted that those results were obtained with significant energy consumption.

As it is shown in Table 2, a gradual increase of temperature occurred during the experiment. With regard to the pH value, a minor effect was noticed, as a result of MR a slight decline of its value was achieved. The decrease of pH value was also observed in this study. This effect was related with the generation of acid compounds caused by thermolysis effect of MR (Bougrier et al., 2006; Jackowiak et al., 2011).

The breakdown of cell membranes and walls, and consequently release of organic matter to the soluble phase results from the mechanisms occurring within MR. It includes thermal and non-thermal effects, additionally accompanied by catalytic oxidation (Bozkurt et al., 2019). The thermal effect is directly related to superheating or "hot spots", as well as the selective absorption of MR. It is generated within the interaction of the oscillating electrical field between dipolar molecules and various organic complexes. The occurring molecular rotation of both the permanently and induced dipoles resulted in friction, which in turn leads to heating of the pre-treated material (Hoz et al., 2005; Fernandes et al., 2023). In turn, non-thermal effect is associated with the rapidly changing dipole orientation in the polarised side chains of the cell membrane macromolecules, which results in the breakage of hydrogen bonds, subsequently leading to the disintegration of the material (Hoang et al., 2021). Their impact is related to the non-thermal interaction occurring between the pre-treated substrate and MR (Remya and Lin, 2011;

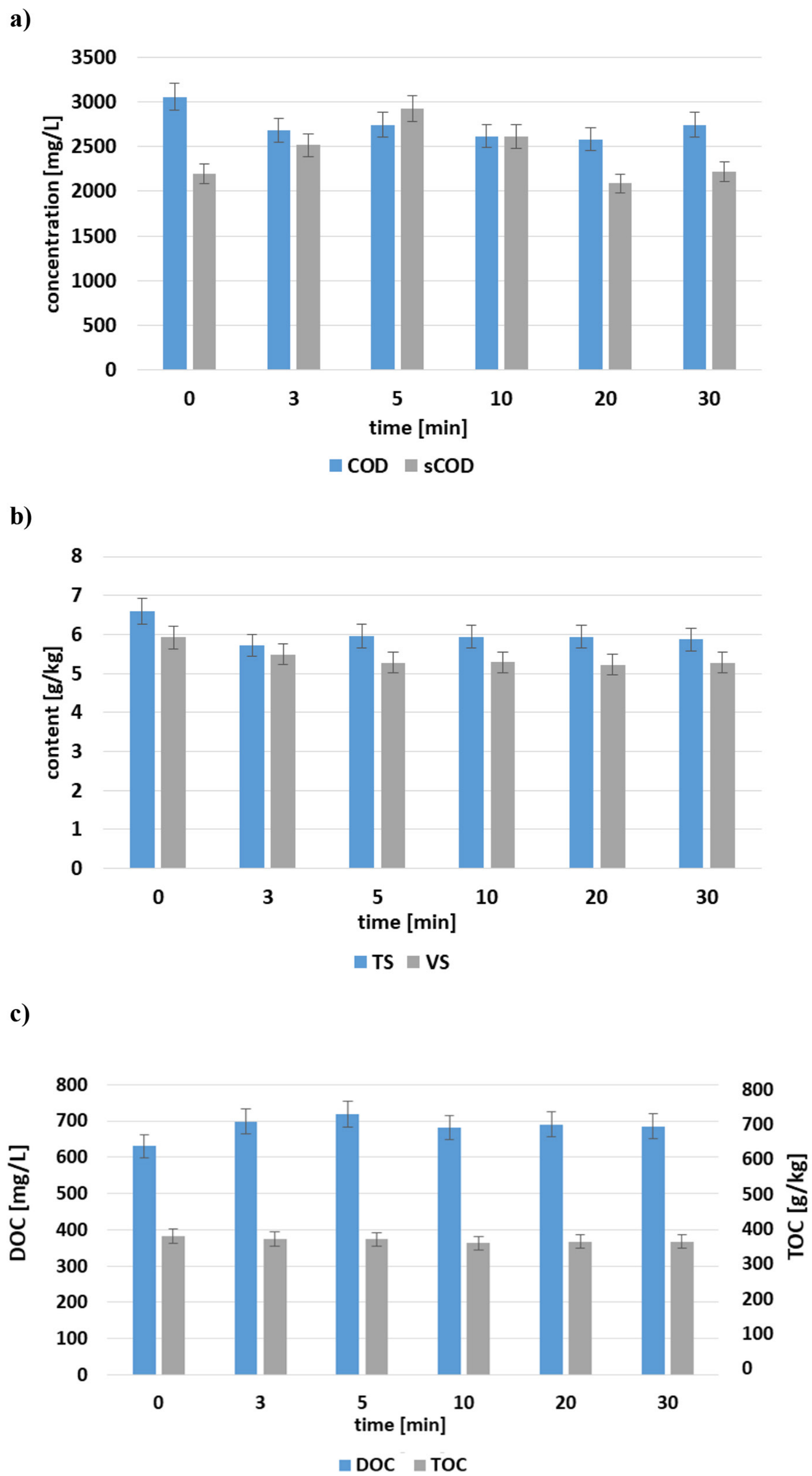

Figure 1. The variation of a) COD and sCOD b) TS and VS c) DOC and TOC contents during the experiment (averages values and standard deviation are given)

Anoopkumar et al., 2023). The enhanced solubilisation as result of MR application was confirmed by other researchers. This pre-treatment strategy amended the solubilisation of kitchen waste by 40% (Marin et al., 2011), organic fraction of municipal solid waste (Shahriari et al., 2013), switchgrass by 8.8–12.8% (Jackowiak et al., 2011), waste activated sludge by 8.5% (Chang et al., 2011) and rice straw by approx. 70% (Kainthola et al., 2019).

Table 2. The results of BI index, sCOD/COD ratio, solubilisation yield and input energy in a specific time intervals (averages values and standard deviation are given)

Time	BI index	sCOD/COD	S_Y	phenols	d-limonene	T	pH	E
min	-	-	%	mg/L	ppm	°C	-	MJ/kg
0	0.00165	0.78	-	9.67±0.30	8.88±1.72	22.2	6.96	-
3	0.00187	0.96	10.5	10.4±0.41	4.10±0.79	27.9	6.87	37.8
5	0.00192	1.06	23.9	14.1±0.29	2.62±0.55	29.4	6.92	62.8
10	0.00188	0.93	13.7	14.6±0.71	2.22±0.18	30.3	6.91	125.6
20	0.00188	0.60	0.05	18.4±0.42	2.14±0.39	32.5	6.89	251.2
30	0.00187	0.71	0.64	19.1±0.55	2.49±0.25	33.7	6.89	376.72

However, crucial factors in successful implementation of this method are microwave irradiation time, frequency or reaction time are the main factors that affect the pretreatment process (Gan et al., 2020). Generally, increasing the reaction time and power might lead to the generation of increased pressure and temperature that effectively destroy organic matter (Hoang et al., 2021). However, beyond a certain point, a loss of volatile organic matter might occur, resulting in decline of Ys (Bozkur and Apul, 2019). This effect was observed in the presented study. Moreover, a similar observation was found in the research conducted by Bohdziewicz et al. (2011). Additionally, an enhanced temperature and time might cause the formation of compounds that present a toxic effect on AD microorganisms. Hendriks and Zeeman (2008) showed that at that temperature exceeding 160 °C, the use of MR for pre-treatment of LB might cause the formation of phenolic compounds. A similar finding was found in the case of switchgrass; therein, also the enhanced temperature led to formation of phenolic acids (Jackowiak et al., 2011).

Importantly, the shorter times prevent the generation of inhibitory compounds that might positively affect the AD performance (Kainthola et al., 2019). This effect is particularly important in the pre-treatment of lignocellulose biomass, the processing of which favours the generation of e.g. phenols or other bioactive compounds potentially affecting AD. Despite enhanced solubilisation, another important effect of MR is improvement of the efficiency of chemical, physical and biological reactions. As compared to the conventional thermal heat, MR has up to 60% lesser energy demand and allows effective distribution of heat; additionally it might improve the biomass structure. Another benefit is preventing the deterioration of the quality of pre-treated biomass, which might be achieved within thermal heating (Kostas et al., 2017). It should be noted that this is an very energy-intensive method; therefore, the cost-effectiveness analysis of such a solution should be carried out.

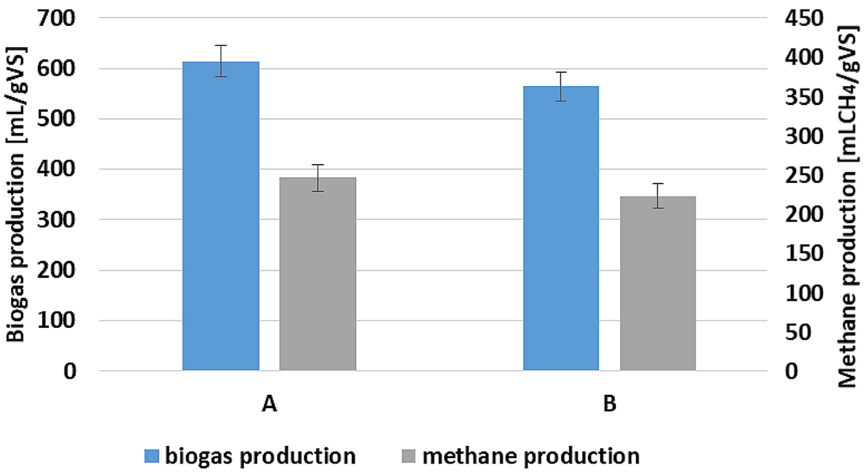

Figure 2. The biogas and methane productions in reactors A and B supplied by pre-treated OW and SS, raw OW and SS, respectively (averages values and standard deviation are given)

For the next part of the study, the OW pre-treated MR for 5 min was chosen. Figure 2 shows the results of biogas and methane productions.

The obtained results indicated that biogas production was enhanced by 8.9%, as compared to raw OW. Similarly, in reactor A supplied by pre-treated OW, the methane production was improved by 10.4%. This effect is related with improving the accessibility and biodegradability of the biomass exposed to microwave irradiation. The previous studies also confirmed this result. In the case of kitchen waste, the use of MW at 1000 W for 2 min resulted in the biogas production enhanced by 54.2%, as compared to raw sample (Achouri et al., 2026). Similarly, microwave irradiation pretreatment applied to microalgae resulted in 60% higher methane production (Carrere et al., 2016). Microwave irradiation effectively destroys the lignin structure, increasing the surface area of the substrate, as well as diminishing cellulose polymerisation and crystallinity, therefore improving accessibility for AD microbes that corresponded into higher methane production (Shah et al., 2025).

CONCLUSIONS

Microwave pre-treatment is a well-recognised method that allows improving the degree of biodegradability of various groups of waste, including orange waste. This study analysed the influence of time and, consequently, the amount of energy introduced on the biodegradability of OW. The obtained results indicated that using a shorter exposure time e.g. 3 and 5 min resulted in enhanced solubilisation yield and biodegradability index with moderate release of inhibitors. However, the most beneficial results were achieved for the time of 5 min and energy input of 62.8 MJ/kg; therein, the BI index was improved by 16.36% and sCOD/COD by 36% as compared to the raw sample. In this case, the major value of solubilisation yield was achieved among all analyses cases. In turn, extending the time results in deterioration of biodegradability with major release of inhibitors and significant energy consumption. Moreover, the use of pre-treated OW using MR for 5 min resulted in an increase in biogas and methane production by 8.9 and 10.4%, respectively, compared to the mixture not subjected to pre-treatment, thus confirming the beneficial effect of microwave irradiation.

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REFERENCES

1. Achouri, O., Panico, A., Derbal, K., Bouteraa, M., Zerdazi, R., Annalinda, C., Bencheikh-le-Hocine, M. (2026). *Conversion of kitchen waste to bioenergy: Influence of microwave and thermal pretreatment*. *Biomass and Bioenergy*, *209*, 108918. https://doi.org/10.1016/j.biombioe.2025.108918
2. Agregán, R., Lorenzo, J. M., Kumar, M., Shariati, M. A., Khan, M. U., Sarwar, A., Sultan, M., Rebezov, M., Usman, M. (2022). Anaerobic digestion of lignocellulose components: Challenges and novel approaches. *Energies, 15*, 8413. https://doi.org/10.3390/en15228413
3. Alengebawy, A., Ran, Y., Osman, A. I., Jin, K., Samer, M., Ai, P. (2024). Anaerobic digestion of agricultural waste for biogas production and sustainable bioenergy recovery: A review. *Environmental Chemistry Letters, 22*, 2641–2668. https://doi.org/10.1007/s10311-024-01789-1
4. American Public Health Association. (2012). *Standard methods for the examination of water and wastewater* (22nd ed.). APHA.
5. Anoopkumar, A. N., Reshmy, R., Aneesh, E. M., Madhavan, A., Kuriakose, L. L., Awasthi, M. K., Pandey, A., Binod, P., Sindhu, R. (2023). Progress and challenges of microwave-assisted pretreatment of lignocellulosic biomass from circular bioeconomy perspectives. *Bioresource Technology, 369*, 128459. https://doi.org/10.1016/j.biortech.2022.128459
6. Bohdziewicz, J., Kuglarz, M., Grubel, K. (2011). The influence of microwave irradiation on the increase of waste activated sludge biodegradability. *Archives of Civil and Environmental Engineering*, 123–130.
7. Bougrier, C., Albasi, C., Delgenès, J.-P., Carrère, H. (2006). Effect of ultrasonic, thermal and ozone pre-treatments on waste activated sludge solubilisation and anaerobic biodegradability. *Chemical Engineering and Processing, 45*, 711–718. https://doi.org/10.1016/j.cep.2006.02.005
8. Bozkurt, Y. C., Apul, O. G. (2020). Critical review for microwave pretreatment of waste-activated sludge prior to anaerobic digestion. *Current Opinion in Environmental Science & Health, 14*, 1–9. https://doi.org/10.1016/j.coesh.2019.10.003

9. Carrere, H., Antonopoulou, G., Affes, R., Passos, F., Battimelli, A., Lyberatos, G., Ferrer, I. (2016). *Review of feedstock pretreatment strategies for improved anaerobic digestion: From lab-scale research to full-scale application*. *Bioresource Technology, 199*, 386–397. https://doi.org/10.1016/j.biortech.2015.09.007
10. Chang, C. J., Tyagi, V. K., Lo, S. L. (2011). Effects of microwave- and alkali-induced pretreatment on sludge solubilization and subsequent aerobic digestion. *Bioresource Technology, 102*(17), 7633–7640. https://doi.org/10.1016/j.biortech.2011.05.031
11. de la Hoz, A., Díaz-Ortiz, A., Moreno, A. (2005). Microwaves in organic synthesis: Thermal and non-thermal microwave effects. *Chemical Society Reviews, 34*(2), 164–178. https://doi.org/10.1039/b411438h
12. Ethaib, S. (2024). Microwave-assisted pretreatment for lignocellulosic biomass energy conversion path. *Bioresource Technology Reports, 28*, 102006. https://doi.org/10.1016/j.biteb.2024.102006
13. Fernandes, A., Cruz-Lopes, L., Esteves, B., Evtuguin, D. V. (2023). Microwaves and ultrasound as emerging techniques for lignocellulosic materials. *Materials, 16*, 7351. https://doi.org/10.3390/ma16237351
14. Gan, Y. Y., Ong, H. C., Chen, W.-H., Sheen, H. K., Chang, J.-S., Chong, C. T., Ling, T. C. (2020). Microwave-assisted wet torrefaction of microalgae under various acids for coproduction of biochar and sugar. *Journal of Cleaner Production, 253*, 119944. https://doi.org/10.1016/j.jclepro.2019.119944
15. Hendriks, A. T. W. M., Zeeman, G. (2009). Pretreatments to enhance the digestibility of lignocellulosic biomass. *Bioresource Technology, 100*, 10–18. https://doi.org/10.1016/j.biortech.2008.05.027
16. Hoang, A. T., Nižetić, S., Ong, H. C., Mofijur, M., Ahmed, S. F., Ashok, B., Bui, V. T. V., Chau, M. Q. (2021). Insight into the recent advances of microwave pretreatment technologies for the conversion of lignocellulosic biomass into sustainable biofuel. *Chemosphere, 281*, 130878. https://doi.org/10.1016/j.chemosphere.2021.130878
17. Jackowiak, D., Frigon, J.-C., Ribeiro, T., Pauss, A., Guiot, S. (2011). Enhancing solubilisation and methane production kinetic of switchgrass by microwave pretreatment. *Bioresource Technology, 102*(3), 3535–3540. https://doi.org/10.1016/j.biortech.2010.11.069
18. Kainthola, J., Shariq, M., Kalamdhad, A. S., Goud, V. V. (2019). Enhanced methane potential of rice straw with microwave assisted pretreatment and its kinetic analysis. *Journal of Environmental Management, 232*, 188–196. https://doi.org/10.1016/j.jenvman.2018.11.052
19. Kostas, E. T., Beneroso, D., Robinson, J. P. (2017). The application of microwave heating in bioenergy: A review on the microwave pre-treatment and upgrading technologies for biomass. *Renewable and Sustainable Energy Reviews, 77*, 12–27. https://doi.org/10.1016/j.rser.2017.03.135
20. Kumar, B., Verma, P. (2025). Integration of microwave assisted pretreatment for ameliorating anaerobic digestion for clean energy. *The Microbe, 6*, 100235. https://doi.org/10.1016/j.microb.2025.100235
21. Lin, Y., Yu, J., Huang, D., Chen, Y., Fu, Y., Zhang, L., Jia, G. (2025). The effect of ultrasonic pretreatment on the drying kinetics of orange peels under different drying methods. *Industrial Crops and Products, 233*, 121479. https://doi.org/10.1016/j.indcrop.2025.121479
22. Manyi-Loh, C. E., Lues, R. (2023). Anaerobic digestion of lignocellulosic biomass: Substrate characteristics (challenge) and innovation. *Fermentation, 9*, 755. https://doi.org/10.3390/fermentation9080755
23. Marin, J., Kennedy, K. J., Eskicioglu, C. (2011). Enhanced solubilization and anaerobic biodegradability of source-separated kitchen waste by microwave pre-treatment. *Waste Management & Research, 29*(2), 208–218. https://doi.org/10.1177/0734242X10362
24. Remya, N., Lin, J.-G. (2011). Current status of microwave application in wastewater treatment—A review. *Chemical Engineering Journal, 166*, 797–813. https://doi.org/10.1016/j.cej.2010.11.100
25. Sawhney, D., Vaid, S., Bangotra, R., Sharma, S., Dutt, H. C., Kapoor, N., Mahajan, R., Bajaj, B. K. (2023). Proficient bioconversion of rice straw biomass to bioethanol using a novel combinatorial pretreatment approach based on deep eutectic solvent, microwave irradiation and laccase. *Bioresource Technology, 375*, 128791. https://doi.org/10.1016/j.biortech.2023.128791
26. Shah, V., Patel, N., Prajapati, P., Jouhara, H. (2025). *Unlocking biogas potential: A comprehensive study on pretreatment techniques of organic substrate for enhanced anaerobic digestion*. *Energy, 335*, 138244. https://doi.org/10.1016/j.energy.2025.138244
27. Shahriari, H., Warith, M., Hamoda, M., Kennedy, K. (2013). Evaluation of single vs. staged mesophilic anaerobic digestion of kitchen waste with and without microwave pretreatment. *Journal of Environmental Management, 125*, 74–84. https://doi.org/10.1016/j.jenvman.2013.03.042
28. Singh, H., Goraya, R. K., Singla, M., Talwar, G., Kumar, Y. (2025). Modeling and optimization of orange peel drying using thin-layer equations and artificial neural networks for standardized powder production. *Food Chemistry Advances, 9*, 101124. https://doi.org/10.1016/j.focha.2025.101124
29. Singh, R., Shukla, A., Tiwari, S., Srivastava, M. (2014). A review on delignification of lignocellulosic biomass

for enhancement of ethanol production potential. *Renewable and Sustainable Energy Reviews, 32*, 713–728. https://doi.org/10.1016/j.rser.2014.01.051

30. Szaja, A., Montusiewicz, A., Cydzik-Kwiatkowska, A., Pasieczna-Patkowska, S., Lebiocka, M. (2025). Enhancing methane production through co-digestion of sewage sludge, citrus waste and brewery spend grain with natural zeolite: Mechanisms and microbiome analysis. GCB Bioenergy, 17. https://doi.org/10.1111%2Fgcbb.70029
31. Trujillo-Reyes, Á., Pérez, A. G., Cuéllar, S. G., Serrano, A., Cubero-Cardoso, J., Jeison, D., Fermoso, F. G. (2024). Evaluation of toxic effect of monoterpene compounds on anaerobic digestion. *Journal of Environmental Chemical Engineering, 12*(2), 112035. https://doi.org/10.1016/j.jece.2024.112035
32. Uddin, M., Wright, M. (2023). Anaerobic digestion fundamentals, challenges, and technological advances. *Physical Sciences Reviews, 8*(9), 2819–2837. https://doi.org/10.1515/psr-2021-0068
33. United States Department of Agriculture, Foreign Agricultural Service. (n.d.). *Citrus: World markets and trade*. https://www.fas.usda.gov