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Hydrodynamic cavitation as a pretreatment to enhance organic matter solubilization in orange waste prior to anaerobic digestion

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Abstract: This study evaluated the effectiveness of using hydrodynamic cavitation (HC) as a pre-treatment strategy for orange wastes. Two pressures, 3 and 5 bar, were tested, and time intervals of 0, 5, 10, 20, 30, and 45 min were investigated. An orifice plate with one concentric conical hole (3/10 mm) was used as the cavitation inducer. Among all the examined cases, the results of the multi-stage author's Multi-Criteria Determination of Recommended Variants (MCDRV) analysis showed that the most advantageous variant, in terms of enhancing the solubilization of organic matter and energy consumption, was a pressure of 3 bar and a cavitation time 20 min. under these conditions, the DOC/TOC (dissolved to total organic carbon) ratio was enhanced 2.45-fold. Additionally, a two-fold increase in the sCOD/COD (soluble to total chemical oxygen demand) ratio was achieved with an energy consumption of 0.164 kWh. In turn, in the second step of the MCDRV method, the determined optimal sub-set contained variants with a pressure of 3 bar and cavitation times of 10 and 30 min, respectively. Importantly, in all analyzed cases, the releases of both d-limonene and phenols was observed, which might potentially inhibit subsequent anaerobic digestion

Introduction

In recent years, due to the growing energy demand and the depletion of conventional fuels, energy recovery from organic wastes through anaerobic digestion (AD) has gained great importance. Currently, AD is recognized as the leading treatment process for converting organic residues into renewable energy, i.e., biogas (Hovorukha, 2024; Ponzelli et al. 2024). Importantly, this technology is widely applied at many wastewater treatment and biogas plants (Dauknys et al., 2024). Despite significant progress in the development of AD, the energy potential of some wastes is still not being fully utilized. This is related to the presence of substrate components that are not readily biodegradable or that are toxic to AD microorganisms. AD is recognized as a highly complex process whose stability, and consequently biogas production, can be easily disturbed by those factors (Chen et al. 2024).

Oranges are among the most widely cultivated fruits in the world. During their processing into various products, a huge amount of residues is generated; it is estimated that approximately half of the fruit becomes waste, including peels, seeds, and pulp. Those by-products have considerable potential

for various applications, such as in the pharmaceutical, cosmetic and food industries (Wang et al. 2026, Vasconcellos and Saavedra 2026). However, their utilization requires additional expenditures; therefore, most orange wastes (OW) remain unused. Moreover, their effective application in AD still constitutes a technological challenge due to the presence of lignin and essential oils.

Previous studies have indicated that the application of an appropriate pre-treatment method of lignocellulosic biomass has become a necessity for its efficient conversion into biogas through AD (Prasad et al. 2024). However, the selection of a suitable pre-treatment method can be challenging and requires advanced technological research. An appropriate strategy should effectively disturb the recalcitrant structure of lignocellulosic biomass while minimizing the potential for generating toxic by-products. Moreover, the selected technology should take into account energy efficiency and technical scalability (Mankar et al. 2021).

Among the various pre-treatment strategies, hydrodynamic cavitation (HC) has demonstrated proven effectiveness in many fields of environmental engineering. It has been applied

for the degradation of dyes and other emerging contaminants, disinfection, sludge treatment, biodiesel production, and improving the biodegradability of side-streams such as landfill leachate (Roy et al. 2025). Additionally, numerous studies have investigated the application of this strategy for biomass pre-treatment (Muhandiram and Rudra 2025). Compared with other well-described pre-treatment methods, HC offers several advantages, including high effectiveness, scalability, cost- and energy efficiency, and low environmental impact (Galloni et al. 2025).

Although HC has previously been applied to the pre-treatment of various wastes, the complexity of the process and the transformations involved require that each residue be considered individually. It should be emphasized that there is an optimal range of cavitation intensity in which solubilization is maximized without excessive energy consumption or excessive release of inhibitors. In this context, time-consuming optimization studies are necessary. Such HC experiments frequently generate multiple results that are difficult to compare, making the selection of the most favorable variant challenging. Therefore, the application of multi-criteria analysis may provide a valuable tool for technologists and researchers (Montusiewicz et al. 2024).

This research concerns the use of hydrodynamic cavitation (HC) as a method for enhancing the solubilization of organic matter in orange waste. To ensure the technical and financial feasibility of the process, optimization studies involving multi-criteria analysis were conducted. To evaluate the changes occurring through HC, surface tension, particle size, and physiochemical properties of the tested samples were determined. Moreover, to provide a comprehensive assessment, the potential generation of toxic by-products, i.e., d-limonene, phenols, ammonia nitrogen, and volatile fatty acids, was also evaluated. In addition, the FT-IR/ATR method was employed to identify the functional groups present in the samples.

Materials and methods

Materials

The orange waste (OW) used in this experiment was obtained from a local market. First, it was thoroughly washed with water and crushed using a laboratory blender to obtain particles with a size of 2 mm. For each HC experiment, a fresh portion of OW was used. To conduct the HC experiments, 640 g of OW was suspended in 30 L of primarily-treated wastewater collected from the municipal wastewater treatment plant

“Hajdów” in Lublin, Poland. The applied substrate dose was established based on the acceptable total solids (TS) content in the mixture subjected to HC, taking into account the technical capabilities of the device. The composition of the materials used in the experiment is presented in Table 1.

HC experiments

The HC experiments were performed using a laboratory-scale device consisting of cavitation reactor, a circulation tank with the active volume of 30 L, a submersible pump with a flow rate of 0.65 L/s and a head of up to 80 m. The control and measurement system included manometers, an electromagnetic flow meter, piezoelectric pressure gauges, and a computer unit. All components were connected by pipes equipped with the necessary valves, allowing the treated medium to circulate in a closed loop and repeatedly pass through the cavitation zone. The laboratory setup did not include a cooling system. An orifice plate with a diameter of 64 mm and a thickness of 10 mm, made of stainless steel, was used as a cavitation device. This plate contained a single concentric conical hole with an inlet/outlet diameter of 3/10 mm located on its axis. The hole had an area of 7.0686 mm² and a total perimeter of 9.42 mm, while the geometric parameters α and β were 1.333 mm⁻¹ and 0.0023, respectively. The laboratory setup and the applied orifice plate are shown in Figure 1.

To conduct the HC experiments, two inlet pressures, 3 and 5 bar, were selected. Under these conditions, the flow rates and velocities in the orifice were as follows: 0.268 L/s and 40.4 m/s at a pressure of 3 bar, and 0.361 L/s and 51.03 m/s at a pressure of 5 bar, respectively. The corresponding cavitation numbers were 0.114 and 0.068 for pressures of 3 and 5 bar, respectively. The influence of HC on the properties of the pre-treated waste was examined at treatment times (t) of 5, 10, 20, 30, and 45 min. The corresponding numbers of passes through the cavitation zone were 2.9, 5.7, 11.4, 17.1, and 25.7 at a pressure of 3 bar, and 3.6, 7.2, 14.4, 21.6, and 32.5 at a pressure of 5 bar, respectively.

The experiment was repeated five times for each pressure. For each replicate, a new, fresh portion of wastewater and OW was prepared. The tables and figures present the mean values together with the standard deviations.

Analytical methods

In the mixture before and after HC, the following parameters were analyzed: total and soluble chemical oxygen demand (COD and sCOD), total and volatile solids (TS and VS), dissolved and total organic carbon (DOC and TOC), phenols, ammonia nitrogen (TAN), and d-limonene. Each analysis was performed in at least triplicate.

Moreover, during the experiment, the pH value and temperature (T) were monitored. A DR 3900 spectrophotometer (Hach Lange, Loveland, CO, USA) was used to determine the sCOD, COD, TAN and phenol contents. In turn, the VS and TS contents were analyzed according to the procedure described in APHA (2012). The TOC content was determined using an RC 612 LECO analyzer (LECO, USA). Moreover, a TOC-L analyzer (Shimadzu, Japan) was used to determine the DOC content. The sCOD and DOC contents were measured in the supernatant obtained by centrifuging the samples at a 4500 rpm for 25 min. The

Table 1: Composition of orange wastes (OW) and wastewater after primary treatment (PW).

Parameter	Unit	PW	OW
TS	g/kg	1.11± 0.05	244.6±7.1
VS	g/kg	0.33± 0.04	229.2±5.9
VS/TS	-	0.30	0.94
COD	mg/L	544.7±9.3	-
sCOD	mg/L	462.3±5.4	-
pH	-	7.39±0.05	-
limonene	µg/g	nd	298
phenols	mg/L	3.2±0.05	-

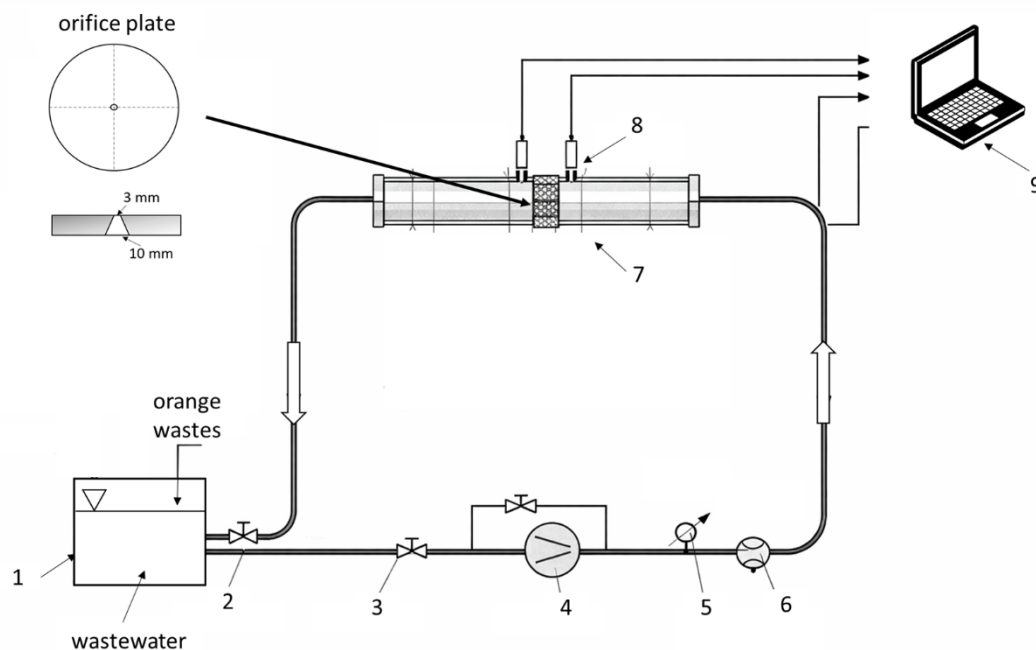


Fig 1. Laboratory installation for HC experiments 1– circulation tank, 2– pipe, 3 – valve, 4 – pump, 5 – manometer, 6 – electromagnetic flow meter, 7 – hydrodynamic cavitation reactor, 8 – piezoelectric pressure gauges, 9 – computer unit to collect data.

d-limonene content was determined using a Thermo GC Ultra PolarisQ Ion Trap gas chromatograph (Waldbronn, Germany) equipped with an Equity-5MS capillary column (30 m x 0.25 mm ID x 0.25 µm df). Helium (99.99% purity) was used as carrier gas at a flow rate of 1.0 mL/min. The following temperature program was applied: an initial temperature of 40°C maintained for 2 min, followed by an isotherm increase of 10°C/min to 250°C, which was maintained for 3 min. The pH of the samples was measured using a pH meter (Elmetron CX-401, Poland) by directly immersing the electrode (EPS-1) into the liquid sample. Electrical conductivity was measured using a conductivity meter (Elmetron CX-401, Poland) equipped with an EC-60 conductometric electrode.

A Zetasizer Nano ZS (Malvern Instruments, UK) was used to determine the zeta potential of the samples. The electrophoretic mobility was converted to zeta potential using the Smoluchowski equation. All measurements were carried out at 25°C and repeated at least three times. The average particle size was determined using dynamic light scattering (DLS) with a Zetasizer Nano ZS (Malvern Instruments, UK). The mean particle size was expressed as the Z-average (intensity to noise ratio). The samples were sonicated for 5 min using a Sonics Vibra Cell ultrasonicator (Sonics & Materials, Inc.) and analyzed in disposable polystyrene cuvettes. The surface tension of the samples was determined at 25°C using the ring method with a K9 tensiometer (Krüss, Hamburg, Germany). Before each measurement, the platinum ring was cleaned and flame-dried. It was then immersed in the sample (50 cm³) and subsequently withdrawn to determine the surface tension. The surface tension values were calculated from the maximum force required to pull the ring through the liquid-air interface.

The structural transformations of the organic matter during HC were evaluated using Fourier-transform infrared

spectroscopy (FT-IR). Before the measurements, all samples were subjected to freeze-drying (lyophilization) to remove water, which would otherwise interfere with the accurate interpretation of the spectroscopic results. The spectra were recorded at room temperature using a Nicolet 6700 spectrometer equipped with a Meridian Diamond ATR accessory (Harrick Scientific, USA). Data acquisition was performed over the spectral range of 3800–600 cm⁻¹ with a resolution of 4 cm⁻¹. To ensure a high signal-to-noise ratio, each individual spectrum consisted of 128 scans. To confirm the reproducibility and reliability of the analytical data, each sample was measured at least three times. Furthermore, no smoothing functions were applied during spectral processing in order to preserve the integrity of the raw spectroscopic features for detailed structural evaluation.

HC parameters

The cavitation number was calculated according to the following equation:

$$C_v = \frac{p_2 - p_v}{0.5 \cdot \rho \cdot v_o^2}$$

Where: p_2 – the fully recovered downstream pressure measured by pressure gauges, Pa; p_v – the vapor pressure of the medium, Pa; ρ – the density of the medium, kg/m³; v_o – the velocity of the medium, m/s.

Both the fully recovered downstream pressure and the vapor pressure were recorded using Keller PR-33X pressure gauges with a resolution of 0.0001 MPa. The flow rate was measured using an electromagnetic flowmeter.

The energy usage (E) was calculated based on the pump inverter readings. The inverter displayed the current [I] and voltage [V] values; by multiplying these values, the power (P)

was determined. The energy usage was then calculated using the following equation:

$$E = P \cdot t \text{ [kWh]}$$

Multi-criteria decision support

A multi-criteria analysis was conducted according to the authors' Multi-Criteria Determination of Recommended Variants (MCDRV) method, following the procedure described by Montusiewicz et. al. (2024). The criteria selected for the analysis were as follows: DOC/TOC ratio (1), the concentration of d-limonene (2) (increased as a result of waste degradation), the sCOD/COD ratio (3), and energy consumption (4). The first three criteria were maximized, whereas the last one was minimized. Despite the negative impact of d-limonene on AD, this criterion was maximized due to its correlation with the DOC/TOC ratio ($R^2 = 0.8767$). Its increase is associated with the decomposition of complex cellular structures and, consequently, the release of readily biodegradable compounds; therefore, the more effective the decomposition, the higher the d-limonene content. The variables considered in the four-dimensional criteria space included the HC inlet pressure and the number of times the stream passed through the cavitation zone, corresponding to the experimental time intervals. In the first step, the Pareto-optimal (non-dominated) variants were identified. Subsequently, of the weighted min-max analysis procedure was implemented based on the following assumptions: (1) all criteria were equally important and assigned identical weights, and (2) the criteria were assigned different weights according to their technological or economic importance.

Results and discussion

The effect of HC on physicochemical parameters

The results of the HC physicochemical analyses and energy usage for the specified experimental variants are shown in Table 2. Due to the variable composition of OW and wastewater during the experiment, the characteristics of the untreated samples varied between the pressure conditions. However, in all analyzed cases, the application of HC resulted in effective destruction of complex organic matter, as indicated by reductions in TOC, COD, and TS. More favorable results were obtained at a pressure of 3 bar. At this pressure, the TOC content decreased from 30.3 to 21.4%, with the highest removal observed after the longest treatment time of 45 min; however, significant reductions were also achieved after 20 and 30 min. A similar trend was observed for TS and VS. The removal efficiencies of TS and VS ranged from 15.9 to 24.9% and from 18.4 to 28.8%, respectively. As in the case of TOC, the greatest reductions were recorded after the longest cavitation time. At a pressure of 5 bar, a less pronounced effect on TOC was observed. In this case, the TOC reduction ranged from 15.3 to 26.8%. The most notable effect was observed for VS, for which the highest removal efficiency of 32% was achieved after the longest treatment time. With regard to TS, only a slight effect of cavitation was observed. Analysis of the effect of cavitation on the VS/TS ratio revealed only a minor decrease, which was comparable for both pressure conditions.

During HC, the decomposition of complex organic matter was accompanied by simultaneous solubilization. This effect was observed at both pressures of 3 and 5 bar; however, it was

more pronounced at the lower pressure of 3 bar. With regard to DOC, an increase of more than 1.5-fold was observed after only 5 min of treatment. The greatest increase was achieved after 10 and 20 min. Thereafter, a slight decrease in DOC concentration was noted. Importantly, the application of the higher pressure of 5 bar did not result in a greater release of DOC. In this case, the DOC concentration increased by 22.2-36.2%, with the highest increase observed after 20 min of treatment. Beyond this time, a slight decrease in DOC concentration was observed, which might indicate that the rate of DOC oxidation exceeded its releasing. These findings suggest that, during shorter treatment times, the physical (thermo-mechanical) effects of cavitation predominate, whereas prolonged treatment favors chemical transformations due to the increasing formation of hydroxyl radicals over time. This effect depended on both the inlet pressure and treatment time and was more pronounced at higher pressures. A similar effect was observed for sCOD. At a pressure of 3 bar, increasing the treatment time enhanced the release of soluble organic compounds. The increase in sCOD ranged from 17 to 30.7%. Importantly, comparable values were recorded within the treatment time interval of 10-45 min. Extending the treatment time did not result in a significant increase in sCOD, probably due to the oxidation of the soluble organic fraction. At a pressure of 5 bar, the effect was less pronounced, and only a slight increase in the sCOD value was observed.

Changes were also observed in the biodegradability index expressed as the DOC/TOC ratio. As previously mentioned, more favorable results were obtained at the lower pressure of 3 bar. Under these conditions, the DOC/TOC ratio doubled after only 5 min of treatment, with the highest value recorded after 20 min. Beyond this treatment time, a slight decrease in the DOC/TOC ratio was observed. A less pronounced effect on the biodegradability index was found at the higher pressure of 5 bar. Under these conditions, the DOC/TOC ratio increased by 55.2-68.8%, with the highest increase observed after 20 min of treatment. A similar trend was observed for the sCOD/COD ratio. At a pressure of 3 bar, this ratio doubled after only 10 min of treatment. At the higher pressure, the same effect was observed after 20 min of treatment. The observed destruction of complex and recalcitrant compounds, together with the accompanying solubilization, is particularly important because microorganisms involved in the AD process metabolize readily biodegradable substances more rapidly. Consequently, HC pretreatment may shorten the retention time and increase methane production, thereby reducing treatment costs and enabling an energy surplus (Yeneneh et al. 2024). The degradation of lignocellulosic biomass during HC occurs as a result of physical and chemical transformations taking place within the hotspot formed after the collapse of microbubbles, leading to a sudden local increase in temperature and pressure (Bimestre et al. 2022).

HC involves the generation, growth, and implosion of cavities resulting from spatial pressure variations within the liquid. Physical and chemical transformations occur in the hotspot region following the collapse of microbubbles, leading to intense localized temperatures and pressures. The physical effects include the action of shock waves, microjet streaming, and elevated temperatures, whereas the chemical effects involve the generation of free radicals (Das et al.

Table 2: The HC results of physicochemical analyses of and energy usage for the specified experimental variants

t	TOC	DOC	DOC/TOC	limonene	TS	VS	VS/TS	phenols	COD	sCOD	sCOD/ COD	N-NH ⁺ ₄	VFA	Energy	T	Variant number
min	g/kg	mg/L	-	µg/mL	g/kg	g/kg	-	mg/L	mg/L	mg/L	-	mg/L	mg/L	kWh	°C	
3 bar																
0	381.4±19.1	573.9±28.7	0.00147	17.1±0.9	5.11±0.26	4.24±0.21	0.83	15.87±0.78	6623.2±87.7	2490.0±54.1	0.376	84.8±4.1	230±5.7		11.8	v1
5	299.7±15.0	906.5±39.4	0.00302	148.5±3.7	4.30±0.21	3.47±0.17	0.81	27.20±1.35	4444.0±74.1	2915.0±37.4	0.656	80.9±3.7	329±6.6	0.041	12.4	v2
10	291.0±14.6	965.3±41.1	0.00332	159.1±8.0	4.22±0.21	3.39±0.16	0.80	30.20±1.51	4282.0±97.1	3137.0±78.3	0.733	80.3±5.1	307±5.8	0.082	13.2	v3
20	273.4±13.7	937.0±44.1	0.00361	140.5±7.1	4.02±0.20	3.20±0.16	0.80	32.40±1.62	4183.0±41.1	3214.0±41.1	0.768	80.1±6.4	305±10.1	0.164	14.1	v4
30	273.5±13.1	916.6±45.8	0.00348	137.2±6.9	3.99±0.16	3.18±0.17	0.80	36.20±1.81	3895.0±104.1	3201.0±68.4	0.822	80.2±7.4	287±11.4	0.246	15.0	v5
45	265.7±13.3	891.5±43.1	0.00329	155.1±7.8	3.84±0.15	3.02±0.21	0.79	35.20±2.1	3815.0±107.1	3255.0±47.1	0.853	80.3±9.7	336±9.7	0.369	16.9	v6
5 bar																
0	344.4±17.1	579.8±29.0	0.00168	36.9±1.8	4.57±0.23	3.73±0.78	0.82	13.78±1.2	8700.0±124.0	2275.0±57.1	0.261	73.7±7.7	220±10.4	0.0	16.8	v7
5	291.6±14.6	712.8±35.6	0.00262	112.9±5.6	4.56±0.22	3.77±0.89	0.83	20.48±2.1	6080.0±147.1	2303.0±78.4	0.379	83.4±5.6	295±14.1	0.059	17.3	v8
10	283.5±	740.6±37.0	0.00261	93.9±4.7	4.52±0.36	3.68±0.90	0.82	22.08±2.2	5190.0±87.2	2300.0±85.4	0.443	84.1±4.7	273±13.1	0.118	18.0	v9
20	277.9±14.2	789.7±39.5	0.00284	80.0±4.0	4.52±0.98	3.66±0.88	0.81	22.80±3.1	4310.0±97.7	2298.0±125.4	0.533	82.4±5.9	306±14.1	0.236	19.3	v10
30	256.5±12.8	708.8±35.4	0.00281	67.5±3.4	4.43±0.47	3.56±0.95	0.80	23.50±1.7	4240.0±157.1	2346.0±94.6	0.553	75.1±8.7	280±9.7	0.354	20.6	v11
45	252.0±12.6	747.8±37.4	0.00281	78.4±3.9	3.92±0.47	3.10±0.57	0.79	23.03±2.7	3880.0±75.4	2309.0±73.1	0.595	78.6±8.4	286±9.8	0.531	22.1	v12

2021). However, several factors may affect the effectiveness of HC, including cavitation intensity, inlet pressure, substrate composition, treatment duration, and the design of the HC reactor. Generally, higher cavitation intensity and longer treatment duration are associated with greater treatment efficiency because more violent reactions and an increased number of passes through the cavitation zone (Sun et al. 2022).

In the present study, the most favorable results were obtained at the lower pressure of 3 bar and treatment times of up to 20 min. It was hypothesized that an excessive increase in the inlet pressure may lead to the onset of supercavitation or the formation of dense cavitation clouds. Under such conditions, interactions between bubbles may result in a cushioning effect, in which the collapse intensity of individual cavities is reduced, thereby diminishing the overall mechanical and chemical efficiency of the process (Brennen, 2014). This mechanism could explain the lower degree of solubilization observed at 5 bar compared to 3 bar, despite the higher energy input.

Another important aspect that should be investigated when evaluating a particular pre-treatment strategy is the fate of inhibitory compounds. In this study, the concentrations of d-limonene, phenols, ammonia nitrogen, and volatile fatty acids (VFA) were analyzed, as all of these compounds may potentially inhibit AD. With regard to d-limonene, the application of HC promoted its release, with the greatest increase observed at a pressure of 3 bar. Under these conditions, an almost 9-fold increase in the d-limonene concentration was achieved after only 5 min of treatment intervals, although at a lower rate. Beyond 20 min, a slight decrease in d-limonene concentration was observed. At a pressure of 5 bar, a less pronounced release occurred, with the highest concentration recorded after 5 min of treatment. During the subsequent treatment intervals, a slight decrease in d-limonene concentration occurred; however, the values remained higher than those of the raw sample.

A similar effect was observed for phenols. At both pressures, a release of this component was noticed, with the greatest increase observed for the lower pressure of 3 bar. Under these conditions, from 20 min onwards, the phenol concentration in the pre-treated mixture was more than double that of the untreated sample. At the higher pressure of 5 bar, the phenol concentration increased by 49-70.6%, with the highest values observed after 30 and 45 min of treatment. Another adverse effect of HC was the release of VFA. As previously, greater VFA release was observed at a lower pressure of 3 bar. The smallest increase (24.8%) was recorded after 30 min of treatment, whereas moderate increases of approximately 33% were observed after 10 and 20 min, respectively. At a pressure of 5 bar, the lowest VFA release was observed after 10 and 30 min, and the increase did not exceed 30%. HC is a well-established method for the extraction of various natural compounds, such as pectin, polyphenols, and terpenes (Meneguzzo et al. 2019). This effect is associated with enhanced diffusion and mass transfer, which facilitate efficient solvent penetration into cells. The performance of HC depends on several operational variables, including solution pH and temperature, inlet pressure, HC reactor type and its geometry, as well as the concentration of pollutants (Panda et al. 2020). In contrast, at a pressure of 3 bar, a slight decrease of TAN was initially observed, whereas extending the treatment time

did not result in any further decrease in its concentration. A different trend was observed at the pressure of 5 bar, where TAN concentration was observed during the treatment period from 5 to 20 min, after which the concentration decreased. Nevertheless, TAN concentrations remained higher than those measured in the untreated sample.

Differences in the release of individual inhibitors resulted from the different operating parameters of HC. This finding also demonstrates that HC requires time-consuming optimization studies conducted individually for each substrate. Due to the complexity of the transformations occurring during cavitation, a thorough analysis of the potential formation of toxic by-products should be carried out, particularly when treating complex mixtures. In many cases, establishing the optimal operating conditions of HC presents a technological challenge, highlighting the need to modify the proposed technology. When volatile toxic compounds are generated, it may be necessary to remove them by stripping with an inert gas before introducing the pre-treated biomass as a substrate for AD. Such a procedure was also applied in the present study for the removal of d-limonene.

Physicochemical properties of the medium also changed as a result of HC (Fig. 2). The results show that, regardless of the applied pressure, HC causes an increase in both pH (Fig. 2a) and electrical conductivity (Fig. 2b). These changes indicate that ionic alkaline substances were released during HC. The increase in pH may be attributed to the disruption of cell walls, which releases compounds contained within the plant tissues. Mineral salts (e.g., potassium, calcium, and magnesium) are released into the solution, often in the form of citrates, which may exhibit slightly alkaline properties (Chegukrishnamurthi et al. 2025). Nitrogen-containing compounds, such as amino acids, amines, and small amounts of ammonia, may also be released. These compounds can generate OH^- ions in aqueous solution, thereby increasing the pH (Balasundaram and Harrison 2006). Additionally, some organic acids present in the orange peel, such as citric acid, may undergo decomposition or oxidation during HC, thereby reducing the acidity of the solution (Chegukrishnamurthi et al. 2025).

Interestingly, at a pressure of 5 bar, the treated samples exhibited lower pH values compared to those cavitated at 3 bar, while their electrical conductivity was higher. This may indicate the release of both alkaline and acidic compounds under these operating conditions (Arya et al. 2023). Another observation was that the zeta potential became increasingly negative with increasing cavitation time (Fig. 2c). This behavior may also be attributed to the release of alkaline substances during HC (Chegukrishnamurthi et al. 2025). In addition, HC disrupts the cellular structures of the orange peels, promoting the release of anionic compounds into the solution. The principal compounds released include pectins, organic acids (e.g., citric or malic acid), and other compounds containing carboxyl groups. In water, these functional groups can dissociate into -COO^- forms, increasing the density of negatively charged groups on the surfaces of colloidal particles (Yapias et al. 2025). Furthermore, fragments of cell walls and polysaccharides form fine colloidal particles whose surfaces are rich in negatively charged functional groups. As a result, the negative surface charge density increases, causing the zeta potential to shift toward more negative values (John et al. 2022).

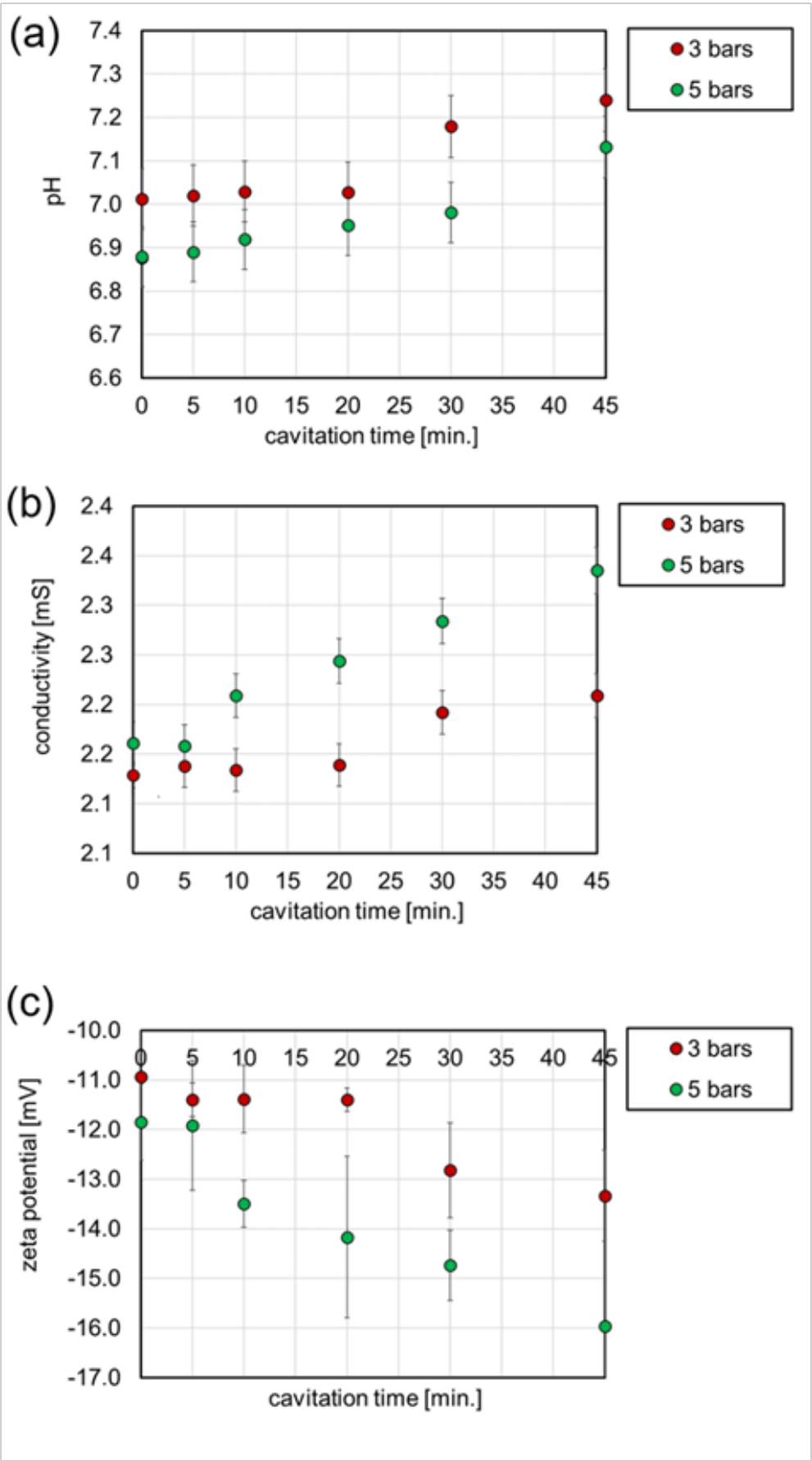


Fig 2. Influence of HC on physiochemical properties: (a) pH; (b) conductivity and (c) zeta potential of the studied samples.

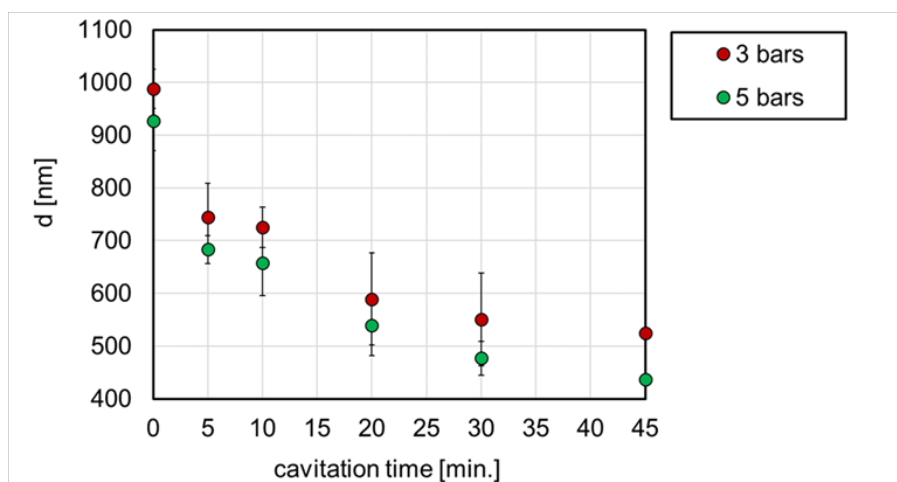


Fig 3. Influence of HC on the particle size of the studied samples.

Figure 3 shows the effect of HC on the particle size distribution of the studied samples. As can be seen from the results, extending the HC treatment time reduced the size of the particles present in the system, which is consistent with previous literature reports (Bałdyga et al. 2009). During HC, intense localized mechanical phenomena, including liquid microjets, shock waves, and high shear stresses, are generated within the system (Azadi and Nobes 2022). These forces disrupt particle aggregates and fragment larger particles into smaller ones. Furthermore, turbulence and intense mixing inhibit particle recombination, resulting in a decrease in the average particle size. A similar effect was observed at both applied pressures, although the higher pressure appeared to be slightly more effective in reducing the diameter of solid particles. The observed structural fragmentation was further confirmed by the changes in the DOC/TOC and sCOD/COD ratios, as well as by the FT-IR/ATR results.

Figure 4 shows the effect of HC on the surface tension of the studied samples. As the results indicate, increasing cavitation time promoted the release of surface-active compounds capable of reducing the surface tension. Consequently, the longer the treatment duration, the greater the concentration of these compounds in the system and the lower the surface tension.

Citrus peels contain essential oils (e.g., d-limonene), phospholipids, proteins, pectin fragments, and other polysaccharides. Many of these compounds are amphiphilic, meaning that they possess both hydrophilic and hydrophobic moieties (Braddock 1999). Consequently, they accumulate at the air-water interface, where they reduce the intermolecular forces between water molecules, thereby lowering the surface tension of the solution. Furthermore, cavitation causes particle fragmentation and better dispersion of the released compounds, increasing their availability at the surface and further enhancing the reduction in surface tension (Zhang et al. 2024). Regarding the effect of pressure, the results suggest that higher pressures are slightly more effective than lower pressures in promoting the release of these compounds.

FT-IR/ATR spectral analysis

The FTIR spectra of freeze-dried samples before and after HC are shown in Figures 5 and 6. The FT-IR spectra of the raw mixture (0 min, Figs. 5 and 6) exhibited characteristic absorption bands of a complex lignocellulosic and lipid-rich matrix. In the high-wavenumber region, a broad absorption at 3273 cm^{-1} is attributed to the hydrogen-bonded hydroxyl (O-H) groups in cellulose and lignin (Socrates 2004). Intense

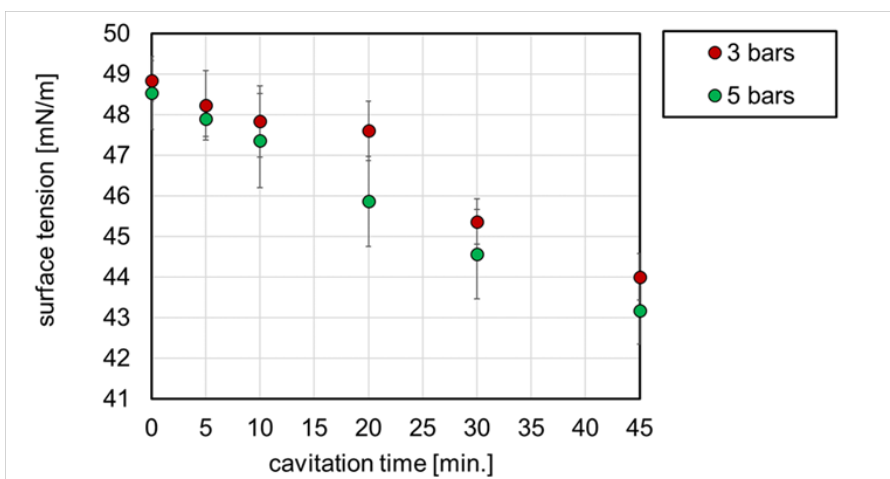


Fig 4. Influence of HC on the surface tension of the studied samples.

peaks at 2920 and 2852 cm^{-1} correspond to the asymmetric and symmetric stretching of aliphatic C-H groups, which are markers for both polysaccharides and terpenoid structures, specifically d-limonene. In the fingerprint region, the band at 1734 cm^{-1} is assigned to C=O stretching in the acetyl groups of hemicellulose or lignin carboxyls (Méndez-Durazo et al. 2025). Aromatic skeletal vibrations and C=C/C=O vibrations from lignin were identified at 1642, 1604, and 1521 cm^{-1} , while the band at 1547 cm^{-1} is associated with the amide II vibrations of proteins. Furthermore, the range of 1239-1010 cm^{-1} reflects C-O stretching and glycosidic linkages in cellulose and hemicellulose (Szaja et al., 2025). Specific peaks confirming the presence of inhibitors included the terminal methylene group of d-limonene at 892 cm^{-1} and phenolic compounds (O-H deformation and C-O stretching) at 1360 and 1239 cm^{-1} (Szaja et al. 2022).

Following 5 min of HC at 3 bar (Fig. 5), the spectra showed a decrease in the intensity of the peaks at 2920, 2852, and 1734 cm^{-1} . Concurrently, there was a significant decrease in the intensity of the lignin-associated bands (1642, 1604, 1547, and 1521 cm^{-1}) and the disappearance of bands at 1360, 1301, and 1276 cm^{-1} . The band at 892 cm^{-1} also decreased, indicating a major loss of d-limonene from the solid phase. Interestingly, a new peak emerged at 865 cm^{-1} , which may indicate the formation of oxidation intermediates, such as peroxy acids or organic peroxides (O-O stretching vibrations

typically found in this region) (Socrates 2004). This emergence could be associated with advanced oxidation processes (AOPs) driven by the postulated generation of hydroxyl radicals ($\cdot\text{OH}$) during the collapse of cavitation bubbles (Thanekar and Gogate 2018; Ozonok 2012). Beyond 10 min of HC treatment, the fingerprint region remained largely unchanged, suggesting that the maximum degree of structural matrix fragmentation was achieved in the early stages of the process.

The FT-IR spectra of the sample treated at 5 bar (Fig. 6) exhibited markedly different behavior from that observed at lower pressures, characterized by the relative structural stability of the insoluble matrix despite the high energy input. Unlike the 3 bar treatment, the characteristic bands in the 1239-1010 cm^{-1} range and the specific d-limonene marker at 892 cm^{-1} remained practically unchanged in both position and intensity throughout the 5-45 minute treatment period.

A significant phenomenon was observed in the carbonyl region, where the peak at 1734 cm^{-1} (ester linkages) exhibited less attenuation than at 3 bar and instead shifted and sharpened toward 1746 cm^{-1} . This shift is indicative of a change in the microenvironment of the C=O groups, likely reflecting a transition to a non-hydrogen-bonded state or a change in the coplanarity of these groups within the solid matrix without their physical cleavage from the lignocellulosic skeleton (Gregoriou and Braiman 2006). Furthermore, the aliphatic C-H stretching bands at 2920 and 2852 cm^{-1} showed a transient increase in

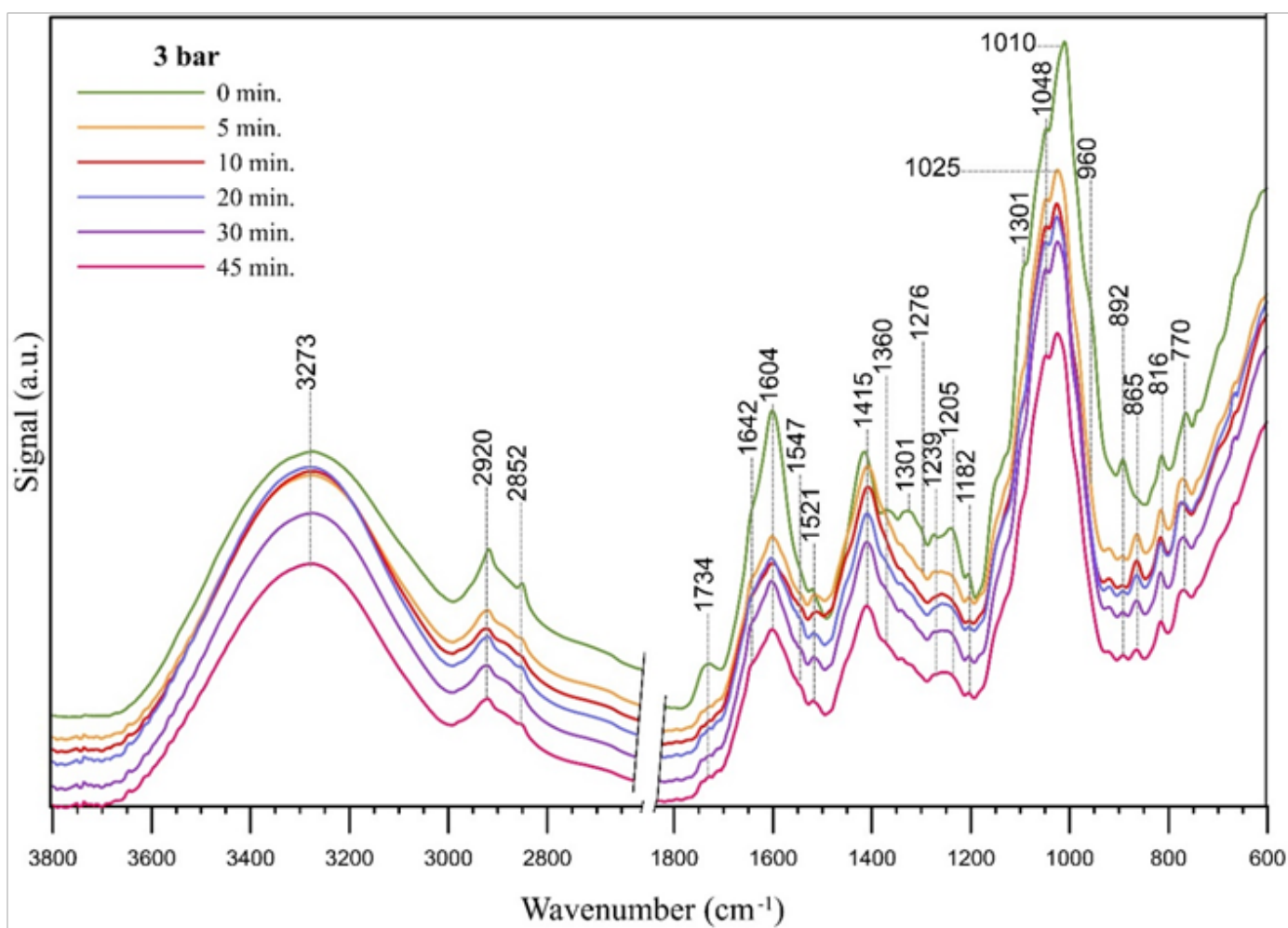


Fig 5. FT-IR/ATR spectra of the raw sample (0 min.) and sample cavitated at a pressure of 3.

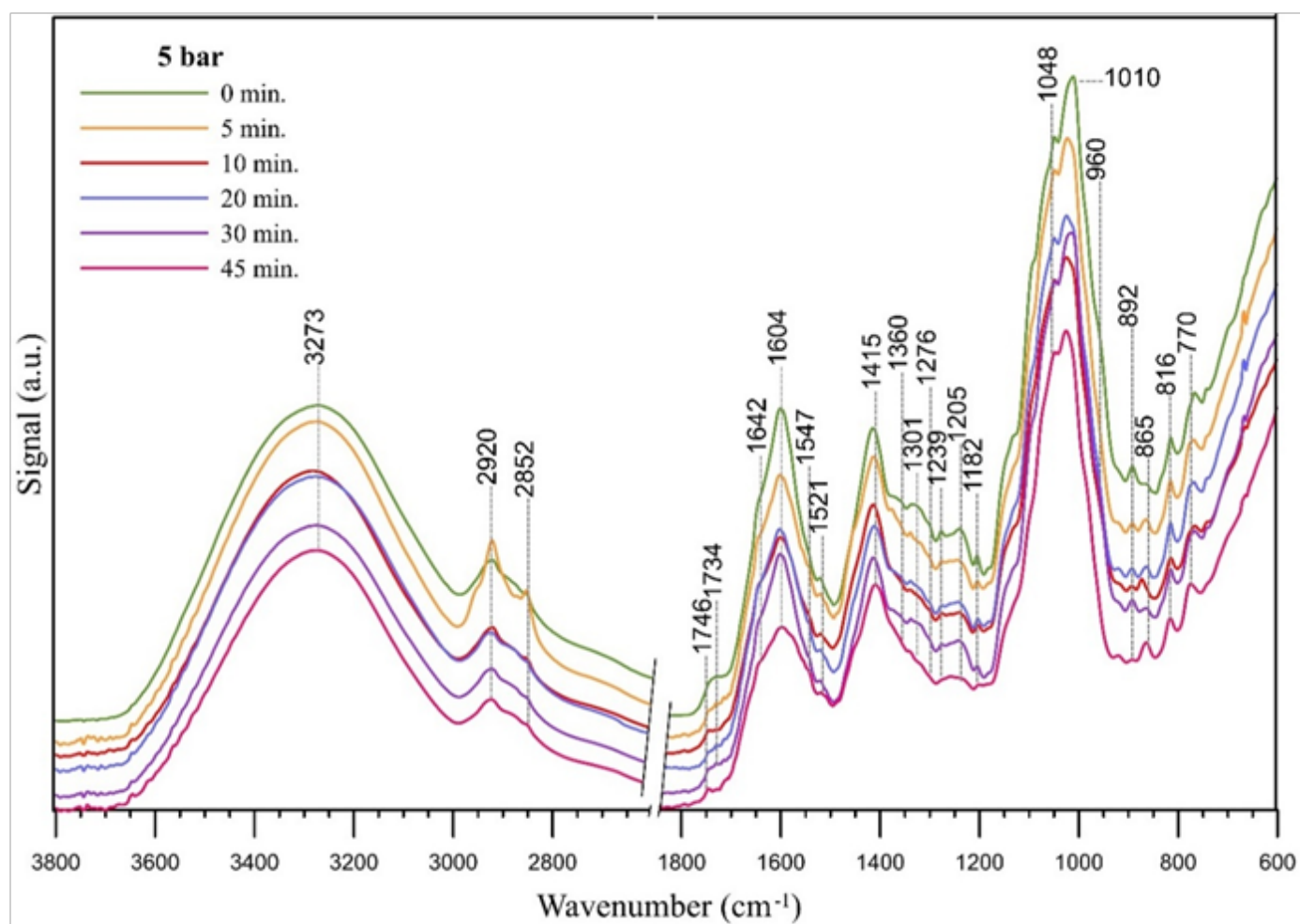


Fig 6. FT-IR/ATR spectra of the raw sample (0 min.) and sample cavitated at a pressure of 5 bar (5-45 min.).

intensity in the 5-minute sample before stabilizing. This apparent increase in the solid-phase spectrum suggests rapid surface erosion of the particles, which probably increases the specific surface area and exposes previously shielded aliphatic groups to the IR beam rather than reflecting a true increase in concentration. The most prominent spectral change over time was the growth of a band at $\sim 865\text{ cm}^{-1}$, which reached its maximum intensity at 45 min. Since this peak is attributed to O-O stretching vibrations in peroxides and intermediate oxidation products, its growth suggests a possible advanced chemical oxidation occurring on the matrix surface. Although radical species were not measured directly, the increase in this band, coupled with the observed decline in soluble d-limonene at 5 bar, is consistent with the oxidative mechanisms described in the cavitation literature (Ozonek 2012).

The analytical approach in this study targeted both the solid and liquid phases of the treated waste. FT-IR measurements were conducted on the freeze-dried matrix to evaluate its structural integrity, whereas physicochemical parameters were determined for the soluble fraction in order to quantify the release of organic matter and inhibitory compounds. Correlating these two phases reveals a highly efficient HC-driven solubilization mechanism. The sharp reduction in FT-IR peak intensities associated with d-limonene (2920 , 2852 , and 892 cm^{-1}) and phenolic compounds (1360 and 1239 cm^{-1}) in the solid matrix subjected to HC cavitation at a pressure of

3 bar (Fig. 5) directly corresponds to their rapid release into the liquid phase. Physicochemical data confirm this stripping effect: within the first 5 min of treatment at 3 bar, the soluble d-limonene concentration increased from 17.1 to $148.5\text{ }\mu\text{g/ml}$, while the phenol concentration increased from 15.87 to 27.20 mg/L (Table 2). These results indicate that HC effectively disrupts the organic skeleton, liberating the inhibitors previously entrapped within the lignocellulosic structure. The efficiency of this redistribution is further supported by the increase in the sCOD/COD ratio from 0.376 to 0.656 in only 5 min, providing chemical evidence of the structural fragmentation (e.g., breakdown of ester and glycosidic linkages) observed in the FT-IR spectra. The concurrent increase in the DOC/TOC ratio further corroborates this interpretation.

The analytical approach adopted in this study targeted both the solid and liquid phases of the treated waste. FT-IR measurements were conducted on the freeze-dried matrix to evaluate its structural integrity, while physicochemical parameters were determined in the soluble fraction to quantify the release of organic matter and inhibitory compounds.

The relatively unchanged FT-IR fingerprint of the solid matrix at 5 bar, together with the physicochemical data, indicates a mechanism dominated by chemical oxidation (AOP) rather than physical solubilization. This is supported by the sCOD/COD ratio, which reached only 0.595 after 45 min at 5 bar, significantly lower than the value of 0.853

achieved at 3 bar (Table 2). This resistance to disintegration at higher pressure is likely attributable to the cushioning effect, whereby an excessive density of cavitation bubbles (super-cavitation) reduces the intensity of individual bubble collapses, thereby reducing the mechanical shear forces required for matrix fragmentation (Brennen, 2014). However, the 5 bar treatment showed a unique capacity for the extensive chemical degradation of inhibitors. Although the soluble d-limonene concentration increased sharply during the first 5 min (112.9 µg/ml), it subsequently declined to 78.4 µg/ml after 45 min. This decrease in the liquid phase coincided with the rising intensity of the 865 cm⁻¹ (O-O bond) oxidation band in the FT-IR spectra. These observations suggest that the inhibitors released into the solution are further oxidized by hydroxyl radicals (·OH), leading to the formation of oxidation products that were subsequently re-deposited on the matrix.

Comparison of the two pressure regimes demonstrates that 3 bar is the optimal operating pressure for enhancing the biodegradability of OW. At 3 bar and short treatment times, HC acts as a liquefying pressure, maximizing solubilization, as reflected by the DOC/TOC and sCOD/COD ratios, and efficiently releasing inhibitors from the matrix within 10 min (Szaja et al. 2025). In contrast, treatment at 5 bar is dominated by oxidative process. Although this pressure promotes the secondary chemical degradation of inhibitors such as d-limonene, it is less effective for matrix disintegration due to the cushioning effect and potential super-cavitation that suppress the mechanical shear forces (Brennen 2014, Thanekar and Gogate 2018). Moreover, it requires substantially greater energy input (0.531 kWh vs. 0.369 kWh) while producing inferior solubilization. Overall, these findings indicate that HC treatment at 3 bar provides the best compromise between enhanced organic matter availability and reduced energy demand.

Multi-criteria decision support

HC generated a dataset, making it difficult to identify the most advantageous operating conditions because numerous performance indicators were obtained for different combinations of process variables. Selecting the HC parameters that maximize biodegradability while minimizing energy consumption therefore required a multi-criteria decision analysis (MCDA). The analysis was performed for the 12 experimental variants listed in Table 2. The Pareto-optimal set, consisting of 7 elements, included variants specified for a pressure of 3 bar, whereas the variants assigned to a higher pressure were excluded based on the multivariate dominance cone analysis. Assuming equal importance for all criteria ($w = 0.25$ for each criterion), variant 6 (pressure of 3 bar, HC time of 45 min) was identified as the optimal solution according to the min-max criterion.

In practice, however, the evaluation criteria do not have equal importance, and their weights should reflect their relative significance. Because criterion 1 DOC/TOC ratio and criterion 4 (energy consumption) were considered the most important, they were assigned higher weights than the remaining criteria. Four weighting scenarios were evaluated (w_1, w_2, w_3, w_4 , the sum of the weights is always equal to 1): (1) 0.5, 0.1, 0.1 and 0.3; (2) 0.4, 0.1, 0.1 and 0.4; (3) 0.5, 0.1, 0.15 and 0.25, and (4) 0.5, 0.1, 0.2 and 0.2. Under all weighting scenarios, the

first stage of the analysis identified variant 4 (3 bar, 20 min) as the optimal solution. Notably, this variant also achieved the highest DOC/TOC ratio. In the second stage, the optimal subset consisted of variants 5 and 3, corresponding to HC treatment at 3 bar for 10 and 30 min, respectively.

Conclusions

The results of this study indicate that HC can be applied as an effective pre-treatment strategy to enhance the solubilization of organic matter in orange waste. Multi-criteria analysis showed that HC treatment at a pressure of 3 bar for 20 min can be considered as the optimal operating condition, providing the best balance between enhanced biodegradability and low energy consumption. This selection was further supported by the physicochemical characterization and FT-IR/ATR spectral analyses. Under these operating conditions, the DOC/TOC ratio increased by a factor of 2.45 compared to the untreated waste, while the corresponding energy consumption reached 0.164 kWh. In the second stage of the multi-criteria analysis, the optimal sub-set included HC treatment at 3 bar for 10 and 30 min, respectively, which produced only slightly lower DOC/TOC ratios than the optimal variant. Importantly, all operating conditions resulted in the release of both d-limonene and phenolic compounds, which may adversely affect the subsequent anaerobic digestion (AD) process. Therefore, further research in this area should be carried out.

Acknowledgements

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Kawitacja hydrodynamiczna jako metoda poprawiająca rozpuszczalność materii organicznej w odpadach pomarańczowych przed procesem fermentacji metanowej

Streszczenie: W niniejszym badaniu oceniono skuteczność zastosowania kawitacji hydrodynamicznej (KH) jako metody wstępnej obróbki odpadów z pomarańczy. W przeprowadzonych badaniach zastosowano dwa ciśnienia 3 i 5 barów oraz następujące czasy 5, 10, 20, 30 i 45 min. Jako induktor kawitacji zastosowano płytę kryzową z jednym stożkowym otworem koncentrycznym (3/10 mm). Spośród wszystkich analizowanych przypadków wyniki analizy wielokryterialnej wykazały, że najkorzystniejszym wariantem pod względem poprawy solubilizacji materii organicznej i zużycia energii jest ciśnienie 3 barów i czas kawitacji 20 minut, w tym przypadku stosunek rozpuszczonego do całkowitego węgla organicznego (RWO/WO) wzrósł 2.45-krotnie, dodatkowo uzyskano dwukrotny wzrost sChZT/ChZT (rozpuszczonego do całkowitego chemicznego zapotrzebowania tlenu) przy zużyciu energii 0,164 kWh. Jako kolejne korzystne warianty analiza wielokryterialna wykazała ciśnienie 3 bar i czasy kawitacji wynoszące odpowiednio 10 i 30 min. Co ważne, we wszystkich przypadkach zaobserwowano uwalnianie zarówno d-limonenu, jak i fenoli, co potencjalnie może hamować późniejszą fermentację metanową tych odpadów.