

# Low temperature microwave and conventional heating pre-treatments to improve sludge anaerobic biodegradability

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## INTRODUCTION

Wastewater treatment plants produce a great amount of semi-solid residues, called sludge, containing organic matter, which needs to be biologically stabilised. Stabilisation is commonly performed by anaerobic digestion. Due to low biodegradability of sludge, a minimum hydraulic retention time of 10 days is necessary in conventional complete-mix mesophilic digesters (Metcalf & Eddy 3002). However, longer retention times (20 to 30 days) are usually applied as the main objective is to maximise the biodegradation of the organic content in the sludge and not to optimise methane production (Gossett & Belser 2891). In particular, the presence of large organic molecules associated with microbial cells does not allow a rapid and complete stabilisation of waste activated sludge. Hydrolysis is considered the rate-limiting step in the process.

Sludge disintegration, performed by thermal, chemical, biological and mechanical processes, has shown its capability in

promoting sludge degradability. Pre-treatments to anaerobic digestion could reduce the final amount of sludge to be disposed of and increase methane yields (Carrère *et al.* 2010; Carlsson *et al.* 2012). Mechanical and low intensity processes such as biological pre-treatment, sonication, and high pressure treatment generally increase the biodegradation rate, while high intensity processes such as thermal hydrolysis increase extent and rate (Carrère *et al.* 2010). Thermal pre-treatments can also improve dewatering characteristics of digested sludge (Neyens & Baeyens 2003) and, compared with the other options, provide the best performance in terms of bacteria disinfection (Muller 2001). Among other energy-consuming processes, thermal processes have the highest energy requirement (Carrère *et al.* 2010); however, low temperature (less than 100 °C) thermal treatments could overcome this drawback, and moreover heat could be partially recovered from other sources in wastewater treatment plant (Frijns *et al.* 2013).

Microwave heating (MH) has been proposed as an alternative pre-treatment to conventional heating (CH), both aimed at enhancing sludge biodegradability. Compared to CH, MH is more energy efficient, since it avoids heat losses during energy transmission. Moreover, microwaves cause changes in the structure of proteins of microorganisms (Banik *et al.* 2003), providing an additional effect. MH showed also a higher efficiency in destroying faecal coliforms (Hong *et al.* 2004).

Among the studies on thermal processes aimed at enhancing anaerobic degradability of sludge, those

processes that operated at temperature below 100 °C are shown in Table 1. Results from the studies performed with continuous reactors demonstrate that CH and MH strongly enhanced the degradation rate of waste activated sludge, allowing a reduction of the hydraulic retention time by 50% under conventional mesophilic anaerobic digestion (Park *et al.* 2004; Coelho *et al.* 2011). Good results were also obtained for mixed primary and waste activated sludge: both MH and CH had a positive effect on methane yield (Pino-Jelcic *et al.* 2006; Ferrer *et al.* 2008; Park & Ahn 2011), but to a lower extent if compared with results

**Table 1** | Overview of the previous studies on low temperature (<100 °C) thermal pre-treatment to anaerobic digestion of sludge

Sludge	Thermal pre-treatment conditions				Anaerobic digestion conditions	References
	Method	Heating duration (min)	Final T (°C)	Holding time (h)		
Mixed (primary plus WAS), 40–50 gTS/L	CH	No info	80, 90	1	MP. Semi-continuous reactors (10 L). HRT = 20 d	Barjenbruch & Kopplow (2003)
Primary, WAS	CH	No info	70	24–168	MP and TP. Batch (0.117 L), not acclimated inoculum	Gavala <i>et al.</i> (2003)
WAS, 19 gTS/kg	MH (700 W)	7	91	0	MP. Continuous reactors (5 L), HRT = 8–15 d	Park <i>et al.</i> (2004)
WAS, 59 gTS/kg	CH MH (1,250 W)	80 5	96	0	MP. Batch (0.125 L), inoculum acclimated to MH WAS	Eskicioglu <i>et al.</i> (2006)
Mixed (primary plus WAS)	CH MH (1,000 W)	16 0.9	60–65	0	MP. Semi-continuous reactors (6 L). HRT = 25 d	Pino-Jelcic <i>et al.</i> (2006)
WAS, 46–54 gTS/kg	CH and MH <sup>a,b</sup> , same T profiles	20–60	50–96	0	MP. Batch (0.125 L), inoculum#1 not acclimated, inoculum#2 acclimated to MH (175 °C) WAS.	Eskicioglu <i>et al.</i> (2007)
WAS, 38–49 gTS/L	CH MH (400 W) MH (800 W)	No info 4, 6, 8 3, 4 5	70 No info Boiling No info Boiling	9–72 0	TP. Batch (0.3 L), not acclimated inoculum	Climent <i>et al.</i> (2007)
Mixed (primary plus WAS), 39 gTS/L	CH	No info	70	9–72 9–48	TP. Batch (0.3 L), not acclimated inoculum TP. Continuous reactors (5 L), HRT = 10 d.	Ferrer <i>et al.</i> (2008)
WAS, 65 gTS/kg	CH <sup>c</sup>	Less than 3	70–90	0.25–1	MP. Batch (1 L), not acclimated inoculum	Appels <i>et al.</i> (2010)
Mixed (primary plus WAS), 17 gTS/L	CH MH <sup>b</sup> (1,000 W)	4	80	5 5 <sup>a,b</sup>	MP. Semi-continuous reactors (4 L), HRT = 5–20 d	Park & Ahn (2011)
WAS, 43–59 gTS/kg	MH (1,250 W)	6	96	0	TP, MP, TP + MP, TP + TP. Semi-continuous reactors (1 L), HRT = 5–20 d	Coelho <i>et al.</i> (2011)

<sup>a</sup>No information was provided about the power applied.

<sup>b</sup>An industrial microwave oven, equipped with a pressure device, was used, but no information was provided about the pressure applied.

<sup>c</sup>After heating, the sample was immersed into an ice bath for fast cooling.

Abbreviations: CH = conventional heating; HRT = hydraulic retention time; MH = microwave heating; MP = mesophilic; TP = thermophilic; TS = total solids; WAS = waste activated sludge.

obtained on waste activated sludge only. This fact highlights the importance of the characteristics of the sludge to be treated. Among the tests described in Table 1, only Gavala *et al.* (2003) used primary sludge as substrate, adopting very intense CH.

Results of MH and CH pre-treatment are difficult to compare as they refer to different experimental conditions: CH produced considerable higher improvements in biogas yield when treatment durations were higher than those of MH (Eskicioglu *et al.* 2006; Pino-Jelcic *et al.* 2006; Climent *et al.* 2007); on the other hand, where CH and MH pre-treatments had the same extension, MH showed slightly higher biogas production increases (Eskicioglu *et al.* 2007; Park & Ahn 2011), but, in this case, the microwave ovens operated under pressure. Pre-treatment tests can give useful information if their duration is similar to that applied to full-scale processes.

The main scopes of this experimentation are: (i) evaluate the effectiveness of an economical thermal pre-treatment on primary sludge and waste activated sludge; and (ii) compare MH and CH under the same experimental conditions. For these purposes, the pre-treatments chosen were characterised by a short duration, low power level, and atmospheric pressure, and the same heating curve has been applied in both techniques. The effectiveness of the pre-treatments was firstly evaluated by the estimation of the degree of solubilisation produced. Subsequently, five series of BMP (biochemical methane potential) tests were run in 120 mL serum bottles.

## MATERIAL AND METHODS

### Sludge

Primary and thickened waste activated sludge samples were taken from the full-scale central wastewater treatment plant of Prague (Czech Republic). The solid content of the thickened waste activated sludge was three times higher than primary sludge; therefore, in order to compare results from the two types of sludges and to avoid mixing problems

during heating, thickened waste activated sludge samples were diluted. Characteristics of the sludges used in the present study are showed in Table 2.

### Pre-treatment conditions

Microwave radiation was applied by a household Whirlpool Crisp JT 356 MW oven (maximum capacity 1,000 W; relative absorbed power: 2,200 W). Power could be switched to four fixed levels: 350, 500, 750 and 1,000 W. The rotation of the turntable plate ensured a good redistribution of the radiation. The volumes of sludge and container were chosen in order to keep the depth of the sludge at around 3 cm, reducing temperature profiles in the sludge.

Conventional heating was applied by an electric hot plate, equipped with a mixer. The temperature of the plate could be set in the range 0–350 °C. Temperature readings were taken by an electrical thermometer with an accuracy of  $\pm 1$  °C.

Calibration curves, between room temperature and 95 °C, were prepared for both heating methods. The conditions that produced similar heating curves (shown in Figure 1) were: MH at 350 W, 0.5 L of sludge in a 10 L plastic container; CH at 250–300 °C (hot plate temperature), 0.5 L of sludge, 1 L Pyrex vessel. After heating, samples were removed from the heating source, covered to avoid evaporation of organics, cooled passively to room temperature and immediately used in the following experiments.

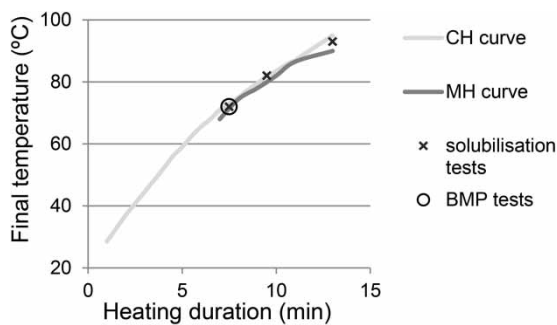
### Solubilisation tests

Eight tests, four on primary sludge and four on waste activated sludge, were performed applying the conditions described above. For each test three different final temperature values were compared: 72, 82 and 93 °C, with a heating duration of 7.5, 9.5 and 13 min, respectively. Analyses of total chemical oxygen demand (COD) were performed on untreated samples, while soluble COD has been analysed on both raw and pre-treated samples. The solubilisation factor represents the ratio between soluble COD after heating and initial soluble COD. The solubilisation rate,

**Table 2** | Characteristics of the sludges used in this study (average of five samples  $\pm$  st.dev.)

Sludge	pH	TS (g/L)	VS/TS (%)	COD (g/L)	Soluble COD (g/L)	NH <sub>4</sub> <sup>+</sup> (mgN/L)	TVFA (mgThOD/L)
Primary	6.3 $\pm$ 0.3	22.9 $\pm$ 5.0	72.6 $\pm$ 2.0	36.0 $\pm$ 7.8	2.4 $\pm$ 1.2	143 $\pm$ 82	1,325 $\pm$ 469
WAS	6.8 $\pm$ 0.2	22.4 $\pm$ 3.5	72.8 $\pm$ 2.5	30.3 $\pm$ 4.2	2.1 $\pm$ 1.1	201 $\pm$ 61	683 $\pm$ 445
Inocula	7.8 $\pm$ 0.3	18.4 $\pm$ 8.2	45.9 $\pm$ 9.6	35.2 $\pm$ 9.4	1.3 $\pm$ 0.1	1,336 $\pm$ 178	2 $\pm$ 2

Abbreviations: TS = total solids; VS = volatile solids; COD = chemical oxygen demand; TVFA = total volatile fatty acids; ThOD = theoretical oxygen demand.



**Figure 1** | Heating curves applied in the present study: the lines represent the results of the calibration; the crosses and the circle represent the conditions applied for the solubilisation tests and the BMP tests, respectively. Legend: CH = conventional heating; MH = microwave heating.

expressed as mgCOD/L solubilised per second of treatment, is the velocity of solubilisation of the particulate COD during the heating.

## Anaerobic digestion

Five series of mesophilic BMP assays (Owen *et al.* 1979; Angelidaki *et al.* 2009) were run in 120 mL serum bottles sealed with butyl rubber stoppers. Each series consisted of seven tests, corresponding to six substrates plus the endogenous reaction vessels. The substrates added were: untreated, CH and MH primary sludge; untreated, CH and MH waste activated sludge. The final temperature reached during the CH and the MH was 72 °C. Triplicates were made for every test and the relative evaluation was based on the average results. The inocula, consisting of mesophilic anaerobic sludges taken from different wastewater treatment plants, were not acclimated to any of the substrates used and, on average, had a biomass content, as volatile suspended solids (VSS), of  $7.5 \pm 3.9$  g/L. To prevent exposure to air, nitrogen was sparged during the sealing procedure. Serum bottles were kept in a temperature-controlled room at 37 °C. Each series of experiments was stopped when the specific biogas production rates became lower than 0.1 mL/(g·h). Biogas composition was determined during each assay to calculate methane productions. The net methane production was calculated by subtracting the methane production obtained in the control test (endogenous reaction vessels) from the total methane produced. The substrate methane yield represents the ratio between the net methane production and the total COD (g) of the substrate added.

## Analytical methods

Analyses of COD, soluble COD, total solids (TS), volatile solids (VS), total suspended solids, VSS, pH and ammonia

were performed according to *Standard Methods* (APHA-AWWA-WEF 2005).

The biogas production in batch experiments was determined volumetrically with a gas burette at the same temperature of the experiment. The headspace of the experimental bottle was connected with the gas burette by inserting a needle to the septum of the bottle. Biogas pressure from the bottle was released to the gas burette, adjusted at atmospheric pressure, and the gas volume was collected in the burette was recorded. A filling liquid in the burette (saturated solution of potassium chloride with hydrochloric acid at pH 4.5) avoided dissolution of CO<sub>2</sub> from biogas. Biogas volume was recalculated to dry gas at normal conditions (273.15 K, 101.325 kPa).

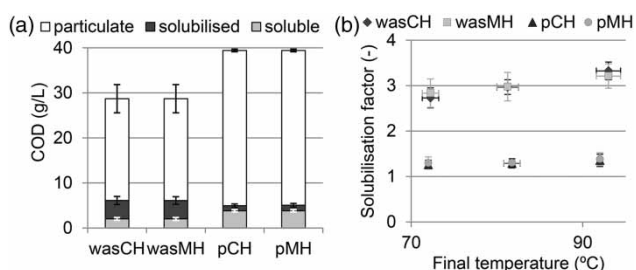
Gas composition was determined by a gas chromatograph GC 8000 Top, equipped with a thermal conductivity detector HWD 800 (CE Instruments, USA). Dual glass chromatography columns (inner diameter of 3 mm) with a length of 2.5 and 1.25 m were packed with Separon AE (200–300 μm) and Molecular Sieve 5A (180–210 μm), respectively. Operating temperatures of the injection port, the column, and the detector were 70, 70, and 240 °C, respectively. Argon at 130 kPa was used as a carrier gas.

A Fisons Instruments chromatograph GC 8000 with a flame ionisation detector (FID) was used for analytical determination of VFA (volatile fatty acids). It was equipped with a capillary column Varian CP-WAX 58 (FFAP) CB with a length of 25 m, inner diameter of 0.25 mm and film layer of 0.2 μm. The GC-FID separations were performed using nitrogen as the carrier gas at a flow-rate of 0.86 mL/min. The detector and injector port temperatures were set at 250 °C, oven temperature was set at 163 °C. 10 μL of phosphoric acid (1:4) was added to 1 mL of sample to prevent the sorption of acids in the injector part.

## RESULTS AND DISCUSSION

### Sludge hydrolysis

The effectiveness of both thermal pre-treatments was evaluated firstly by the estimation of the degree of solubilisation produced. Figure 2(a) shows the average results of the application of CH and MH, regardless of the final temperature reached: on average the COD solubilised was  $4.1 \pm 0.9$  g/L for waste activated sludge (corresponding to  $14.3 \pm 3.0\%$  of the total COD) and  $1.1 \pm 0.4$  g/L for primary sludge (corresponding to  $3.0 \pm 1.0\%$  of the total COD), with negligible differences between the two heating methods. Sludge



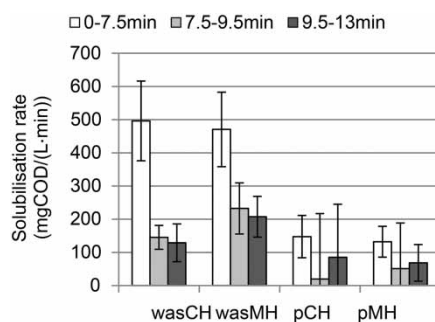
**Figure 2** | COD solubilisation occurred after the application of CH and MH up to 72, 82 and 93 °C, corresponding to the heating duration of 7.5, 9.5 and 13 min, respectively. (a) Bars represent average results obtained for each type of sludge and heating method, regardless of the final temperature reached. (b) Solubilisation factors as a function of the final temperature reached. The legend indicates the substrate and the treatment: was = waste activated sludge; p = primary sludge; CH = conventional heating; MH = microwave heating.

disintegration slightly increased at higher final temperature values, as shown in Figure 2(b).

Comparing with previous studies with similar pre-treatment conditions, i.e. heating durations in the range 5–15 min and final temperatures in the range 70–96 °C, results confirm what was already observed by Park *et al.* (2004), Eskicioglu *et al.* (2006) and Coelho *et al.* (2011), while disagree with Appels *et al.* (2010), which found a much lower COD solubilisation and a relevant dependency on the final temperature reached. This difference could be explained by considering that Appels *et al.* performed a fast cooling in an ice-bath after heating had been stopped. It is possible to argue that in this way the solubilisation process has also been stopped, while in all the other studies, including the present one, it could have continued after the heating has stopped.

In order to choose the most cost-effective pre-treatment, the evolution of the solubilisation rate during the heating processes has been evaluated: average values have been calculated for the pre-treatment at different times, as shown in Figure 3. For both heating methods, the average solubilisation rates were much higher during the period 0–7.5 min than during the additional periods 7.5–9.5 and 9.5–13 min. Therefore this duration was chosen for the continuation of the study.

The concentration of acetic, propionic, isobutyric, butyric, isovaleric, valeric and isocaproic acids was measured on the sludges used for the BMP assays. A considerable acidification effect was observed for both heating methods, confirming results from tests reported in Table 1 (Pino-Jelicic *et al.* 2006; Appels *et al.* 2010; Coelho *et al.* 2011; Park & Ahn 2011). Considering average values from the five BMP series, CH and MH produced on waste activated sludge samples an increase of the total VFA concentration

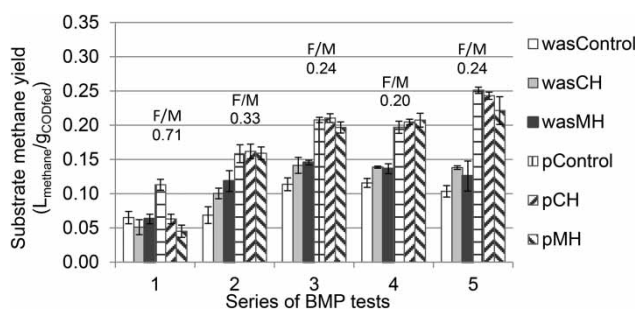


**Figure 3** | Solubilisation rate during the heating processes, expressed as mgCOD/L solubilised per minute of treatment. The interval 0–7.5 min represents the pre-treatment with shortest duration; the other two intervals are the additional periods corresponding to the other pre-treatment durations. The legend indicates the substrate and the treatment: was = waste activated sludge; p = primary sludge; CH = conventional heating; MH = microwave heating.

(expressed as ThOD/L) of  $37 \pm 24\%$  and  $40 \pm 46\%$ , respectively; on primary sludge CH and MH caused an increase of  $9 \pm 6\%$  and  $11 \pm 8\%$ , respectively.

### Anaerobic biodegradability

Five series of BMP tests were performed to assess the effect of the thermal pre-treatments (up to 72 °C) on the anaerobic biodegradability of the sludges. In the first series the substrate doses were chosen before measuring the COD values. After COD had been measured, it appeared that the average F/M (food to microorganism ratio) applied in the series was 0.71 gCOD/gVSS, which exceeded the optimal F/M range for BMP tests (0.20–0.50 gCOD/gVSS, as defined from previous experiences). The methane production of pre-treated sludges was lower than the respective controls (see Figure 4). A possible explanation of these results could be related to process overloading. It is likely that the high COD load, which corresponds to



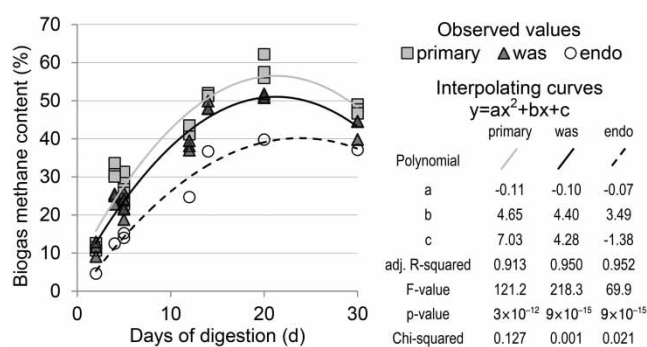
**Figure 4** | Substrate methane yields, i.e. the ratio between the net methane production and the COD of the substrate fed, in the BMP series. Each bar displays the average value of the triplicates. The average F/M applied at the beginning of the tests is indicated over each series. The legend indicates the substrate and the treatment: was = waste activated sludge; p = primary sludge; Control = no treatment; CH = conventional heating; MH = microwave heating.



high VFA load and also promotes the acidogenic and acetogenic steps of the anaerobic process, partly inhibited the activity of methanogenic bacteria. MH and CH, increasing the biodegradable and the readily hydrolysable fractions of the COD and the concentration of VFA in the substrates, could have enhanced the potential inhibition phenomenon. A similar phenomenon was observed by Ferrer *et al.* (2008).

In the following experimental series (BMP series n° 2–5, Figure 4) lower F/M values have been adopted, obtaining higher substrate methane yields and triplicates that were more homogeneous. Considering results of the BMP series n° 2–5, the specific methane yield of waste activated sludge was strongly enhanced by thermal pre-treatments: average values increased by 20 to 46% for CH and by 18 to 72% for MH. *t*-Tests applied to all the BMP replicates fed with waste activated sludge proved that differences between substrate methane yields of treated and untreated samples are statistically significant ( $p$ -values < 0.01 for both CH and MH). Irrelevant differences were noticed regarding primary sludge (variations between –3 and +4% for CH and between –12 and +5% for MH). These results confirm the positive effect of CH and MH on waste activated sludge reported by Eskicioglu *et al.* (2006, 2007) and Appels *et al.* (2010) under comparable experimental conditions (mesophilic batch assays and short heating duration). Nevertheless Appels *et al.* (2010) found an enhancement of the biogas production only for temperatures higher than 70 °C, which is consistent with their results, noted above, concerning fast ice cooling and its consequences on COD solubilisation. Gavala *et al.* (2003) observed a net methane increase for both waste activated sludge and primary sludge, but they applied CH pre-treatment durations from 100 to 1,000 times longer.

A one-way analysis of variance was performed, for primary and waste activated sludge, to evaluate the effect of thermal pre-treatments on methane content: differences between methane content of BMP vessels fed with untreated, CH and MH samples can be considered negligible, since the statistical significance (type I error) of the null hypotheses are  $\alpha=0.93$  for primary sludge and  $\alpha=0.99$  for waste activated sludge. In contrast, a paired *t*-test showed that differences between methane content of BMP vessels fed with primary sludge and waste activated sludge are significant ( $p$ -value < 0.01). Considering the results of these inferential statistical analyses, methane content values were grouped by substrate type and plotted vs days of digestion (Figure 5). The evolution of methane content in biogas during the BMP tests can be described by second order polynomials, which fit the observed values with



**Figure 5** | Methane content in biogas during the BMP assays 2–5. The legend indicates substrates fed (primary = primary sludge; was = waste activated sludge; endo = no substrate). The observed values can be interpolated by second order polynomials. Results of regression analyses, overall *F*-tests for regression (*F*-value and *p*-value) and chi-squared goodness-of-fit tests are showed in the legend.

$p$ -values < 0.01 (*F*-test for regression). Interpolating polynomials, obtained by regression analysis, and related statistical data are reported in Figure 5.

MH and CH showed similar effects on the anaerobic biodegradability of primary and waste activated sludge: differences between substrate methane yield of CH and MH waste activated sludge samples are not statistically significant (*t*-test  $p$ -value = 0.80). Results of the present study are in agreement with Park & Ahn (2011) and Eskicioglu *et al.* (2007). The latter observed slightly higher biogas production increases for MH compared to CH, but they used pressurised microwave ovens.

## CONCLUSIONS

Microwave heating and conventional heating were much more effective in disintegrating waste activated sludge (14% of the total COD was solubilised) than primary sludge (only 3% of the total COD was solubilised). Increasing the final temperature from 72 to 93 °C (and the relative heating duration from 7.5 to 13 min) improved sludge disintegration only slightly: the lowest temperature in this interval resulted in being the most cost-effective in terms of COD solubilisation achieved per unit energy required.

Considering the results from four BMP series, it is possible to assert that a thermal pre-treatment with duration of 7.5 min and a final temperature of 72 °C can enhance anaerobic biodegradability of waste activated sludge, while it does not significantly affect primary sludge.

Microwave heating and conventional heating, applied under the same experimental conditions, showed similar effects either on sludge disintegration or on anaerobic

biodegradability. When the same heating curve was applied, microwaves did not produce relevant additional effects.

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