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INTENSIFICATION OF THE BIODEGRADATION OF WASTEWATER SLUDGE BY MICROWAVE IRRADIATION

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Abstract: The applicability of microwave (MW) irradiation for sludge treatment has been intensively investigated in the last 20 years. Studies have focused on the detailed analysis of MW irradiation on waste activated sludge. Results have been derived from batch experiments.

In this study, continuous flow microwave (MW) treatment with different irradiated energy and power intensity was applied in order to improve the chemical oxygen demand (COD) solubilization and biodegradability of thickened primary dairy sludge. The efficiency of MW treatment was examined by batch mesophilic anaerobic digestion (AD) tests, as well. Our results show that the solubilization ratio (soluble to total COD, SCOD/TCOD) and aerobic biodegradability (expressed as the ratio of biochemical oxygen demand to soluble COD: BOD/SCOD) was influenced by energy input, as well as by the MW power. Increased power and/or enhanced energy input resulted in higher solubilization and biodegradability. Nevertheless, the ratios SCOD/TCOD and BOD/SCOD worsened if irradiated energy exceeded the critical value of 90 kJ/L and of 200 kJ/L with power intensity of 600 W, respectively. Biogas production is influenced mainly by the input energy.

Compared to the untreated sludge, the MW energy input of 120 kJ/L and 220 kJ/L resulted in biogas yield increments of 174% and 210%, respectively. Power intensity did not affect the cumulative biogas product, but it can be an influential parameter for the rate of anaerobic digestion. Applying 220 kJ/L energy input with different power intensities (300-600 W), it was found that 300 W power intensity resulted in accelerated anaerobic digestion; approximately 80% of total biogas volume was produced in the first 15 days of the fermentation process.

Keywords: *MW treatment, wastewater sludge, sludge utilization technologies*

1. INTRODUCTION

In the EU approximately 60% of renewable energy generation is based on biomass, but 80% of biomass energy originates from wood utilization [1]. The diversification of bioenergy raw materials should be encouraged and the role and ratio of waste and byproduct in the bioenergy production should be increased, according to the waste-to-energy (W2E) concept. Because of its high organic matter content, the sludge originating from municipal wastewater treatment plants and agrifood industry has high potential for biofuel – mainly for biogas – production.

Sludge generation is continuously rising, because of the growing urbanization and the concentration of industrial activities into industrial parks, respectively. In cities and industrial parks the wastewater purification technology has high removal efficiency; mainly in the mechanical (primary) stage of technology but also in biological wastewater treatment (secondary) stage a huge amount of sludge is produced containing the wastewater pollutants in a concentrated form. During the conventionally used wastewater treatment technologies approximately 8–10 kg/m^3 of waste activated sludge is produced [2]. Furthermore, the specific sludge generation of several types of industry effluents is higher than that for municipal wastewater; therefore, sludge handling has become more and more costly for companies in recent decades. Among the numerous methods availalbe, anaerobic digestion (AD) is often applied for both sludge stabilization and biogas production. Involving sludge in biomass based energy generation has several benefits: it provides facilities to stabilize the sludge with reduced environmental hazard, it makes the utilization of nutrients for agriculture possible, (the main concept of the circular economy), and it reduces the net CO_2 emissions.

In most cases, bioenergy generation based on biotransformation (fermentation) processes needs pre-treatments to make biological degradation and enzymatic hydrolysis possible, or to increase their efficiencies [3]. The direct energetic utilization of biomass needs pretreatment, as well, to increase the energy density of raw materials [4]. Conventional thermal pre-treatments, often in combination with chemical methods, are widely used in practice, but more and more attention is being devoted to finding alternative, non-conventional heating sources that can be applied for enhanced sludge processing [5].

Due to its volumetric and selective heating effect, microwave irradiation provides an energetically effective method for biomass treatment, especially for materials with high water content, such as sludge. Microwave effects on sludge can be explained by three different mechanisms, thermal (heat generation); specific non-thermal effects of the electromagnetic field; and catalytic oxidation [6]. Because of the polar characteristic, water molecules and other polar components have high energy dissipation capability. Therefore, in the processing of high water content sludge by high MW energy intensity the thermal effect dominates over the non-thermal effect [7]. The dielectric behavior of sludge leads to high temperature increase during microwave heating, and the high heat stress and temperature gradient generated inside the processed materials manifests in rapid hydrolysis of macromolecular components, and disruption of cell walls, respectively [8]. It can be noted that the ability of molecules to oscillate is different for the free and bounded water content.

Summarizing the results available in the scientific literature, it can be concluded that microwave (MW) treatment is suitable to increase the soluble chemical oxygen demand (SCOD) of sludge. The increment in the solubility of organic matters, disruption of cell walls and extracellular polymeric network, furthermore the destruction of original aerobic microbial community leads to intensified anaerobic digestion process of sludge. Increment of solubility degree is influenced by the type (origin) of sludge, time of MW irradiation, microwave power, physicochemical and dielectric properties of components, and total and volatile solid content of materials. In MW treatment, higher initial volatile solid concentration resulted in a higher degree of solubilization [9].

The majority of research papers deal with relatively low total solid (TS) content sludge. Very little research has focused on the efficiency analysis of the MW treatment of thickened sludge, or sludge cake from dewatering processes (sludge belt and screw press, sludge filtering press, etc.). Sludge with a high content of solid and organic matter is sensitive to the use of an appropriate temperature range. In research practice, the temperature range over the boiling point (120–200 °C) is used for sludge treatment, mainly on the lab scale, with batch pressurized reactors. But in sludge with a high protein and carbohydrate content (from the primary dairy industry or canning industry, for instance) the macromolecular components are rapidly decomposed to free amino acids and reducing sugars, which contribute to the occurrence of Maillard-type reactions [10] , whose products are resistant to biodegradation.

Unfortunately, studies available in the field of microwave irradiation of sludge have focused mainly on batch treatment with small sludge samples (10–200 mL). Therefore the irradiated MW energy range was varied widely in the range of 50–80,000 kJ/g VS, depending on the quantity of the sample, irradiation time and the power of microwave equipment. Considering the type of processed sludge, it can be concluded that mainly the effects of MW irradiation on waste activated sludge have been investigated; very few papers have been concerned with primary sludge treatments. Therefore, our research focused on the investigation of the applicability and efficiency of microwave pre-treatment on the biodegradability of primary, thickened sludge from the food industry.

2. MATERIALS AND METHODS

The thickened wastewater sludge samples originated from a dairy in Szeged, Hungary. The main characteristics of the sludge are summarized in *Table 1*. Sludge samples were stored refrigerated (+4 °C) in closed plastic containers before processing to avoid dehydration and the solubility change from freezing-thawing.

Table 1

Main characteristics of sludge

TS [%]	pH [-]	COD [mg/L]	SCOD/TCOD [%]	BOD_5 [mg/L]
4.9±0.22	6.2±0.32	23910±421	23±1.8	1424±63

TS-total solids; SCOD and TCOD-soluble and total chemical oxygen demand; BOD_5-5 day biochemical oxygen demand

Microwave pre-treatments were carried out in a semi-pilot, specially-made microwave unit equipped with a magnetron with variable nominal power in the range of 200–650 W at an operating frequency of 2,450 MHz. In continuous flow operation mode the sludge flows through a toroidal pipe (inside diameter 10 mm) placed inside the microwave cavity. The flow rate was varied in the range of 5–60 L/h by the revolution of a peristaltic pump. Energy input (kJ/L) was calculated from the magnetron power, volumetric flow rate and residence time of sludge in microwave reactor.

The total solid content (TS) of sludge was measured by drying cabinet method at 105 °C. Chemical oxygen demand (COD) was determined by photometric method according to ISO 6060-1989 with HACH cuvette tests. To separate the soluble organic matters (expressed as SCOD) sludge samples were centrifuged (RCF of 10,000 G for 20 minutes) and filtered (0.45 mm syringe filter), after dilution (dilution factor was 10). Biochemical oxygen demand (BOD_5) was measured in a respirometric BOD meter (Oxidirect) at 20 °C for 5 days (according to the DIN 38409H51 method). Each analytical test was carried out three times.

Batch mesophilic anaerobic digestion (AD) test was applied to determine the biogas production of sludge at 37 °C for 30 days. AD tests were carried out in lab-scale, continuously stirred glass reactors (volume of 250 mL with sample volume of 100 mL, head space of 150 mL), applying adapted mesophilic municipal sludge as inoculum at a dosage of 10 vol%. The pH of sludge samples was adjusted to 7.2 and nitrogen purging was used before the tests to ensure the anaerobic condition. The volumetric biogas production was determined by manometric method, pressure increment was detected by Oxitop-C measuring heads, gas volume was calculated by ideal gas law. AD tests were triplicated; biogas product was given as the mean of the calculated volumes.

3. RESULTS AND DISCUSSION

Studies have reported that the divalent cations can bind to extracellular polymeric substances (EPS), which produce larger and more compact flocs. MW treatment is suitable to disrupt the polymeric matrix formed by polysaccharides, proteins, and products of microbial metabolism. Microwave induced disintegration of sludge flocks assists in releasing organic matter from i) the polymeric network, and ii) intracellular space due to the cell wall disruption effect, therefore the solubility of COD (given by the SCOD parameter) increases. In the MW sludge treatment process the COD solubilization is influenced by the pH, TS content, and energy input. Our preliminary results show that in batch processing, beside the irradiation time and energy, the specific MW power intensity also has an effect on the disintegration degree [11].

The results of our continuous flow treatment show that the solubilization ratio (SCOD/TCOD) of thickened primary dairy sludge was influenced by both energy input and the MW power. At lower power intensity (in the range of 300–500 W), increasing energy input resulted in the enhancement of SCOD/TCOD. Solubility of organic matters of raw sludge (23%, data not shown in figures) improved by over

40% after MW treatments. But a worsening effect of energy input increment was found for MW treatment at 600 W, if energy input exceeded 90 kJ/L (*Figure 1*).

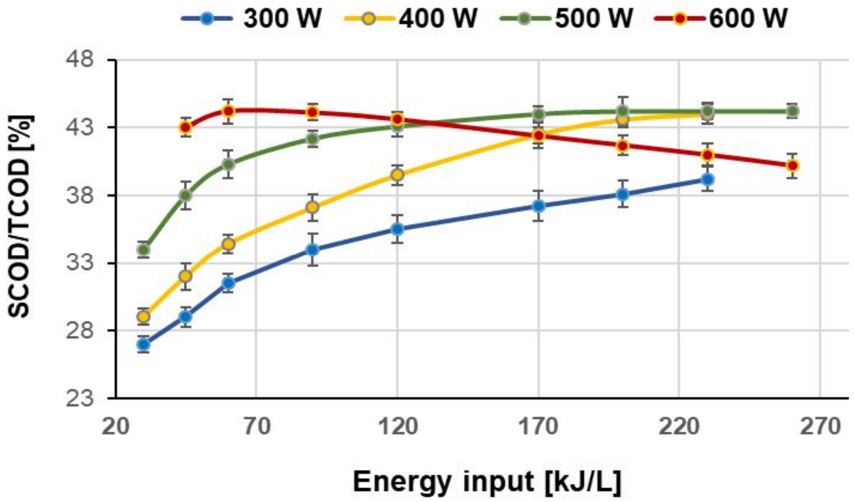

Figure 1

Organic matter solubility (SCOD/TCOD) as a function of MW energy input (kJ/L) and power intensity (W)

(SCOD-soluble chemical oxygen demand, TCOD-total chemical oxygen demand)

Ahn et al. [12], Uma et al. [9], and Ebenezer et al. [13] reported the specific irradiated MW energy (energy input) to be the main factor in organic matter solubilization. Their batch MW waste activated sludge disintegration tests verified that over a certain value energy input (14,000–82,000 kJ/kg TS, depending on the type of sludge) the SCOD/TCOD worsens. In our experiments a lower amount of energy was irradiated, but it has been clearly verified that the MW irradiation applied to continuously flowing sludge was suitable to increase the COD solubilisation. However, because the increment of organic matter solubility is limited, no further enhancement in SCOD/TCOD was observed when the sample was MW irradiated at 200 kJ/L or higher *(Figure 1).*

Increased organic matter solubility is reported to be advantageous because it stimulates improvement in biodegradability [14], [15]. MW pre-treatment for sludge, applied prior to biological transformation processes (such as anaerobic digestion or composting), has great potential because enhanced solubility makes the biodegradation and enzymatic hydrolysis processes faster. In order to quantify the efficiency of MW treatment of sludge on the change of biodegradability, the ratio of biochemical oxygen demand (BOD) to soluble chemical oxygen demand (SCOD) was calculated. This parameter indicates the bioavailability (in this study, the ability to undergo aerobic microbial degradation in a 5-day period) of organic matter in the soluble phase of sludge.

Results show the same tendency which has been revealed for SCOD/TCOD, i.e. the increment of irradiated energy and MW power had a positive effect on biodegradability, but over a certain energy input (200 kJ/L), application of the higher power intensity range (500–600 W) led to a decrease in the ratio of biodegradable organic matter *(Figure 2)*.

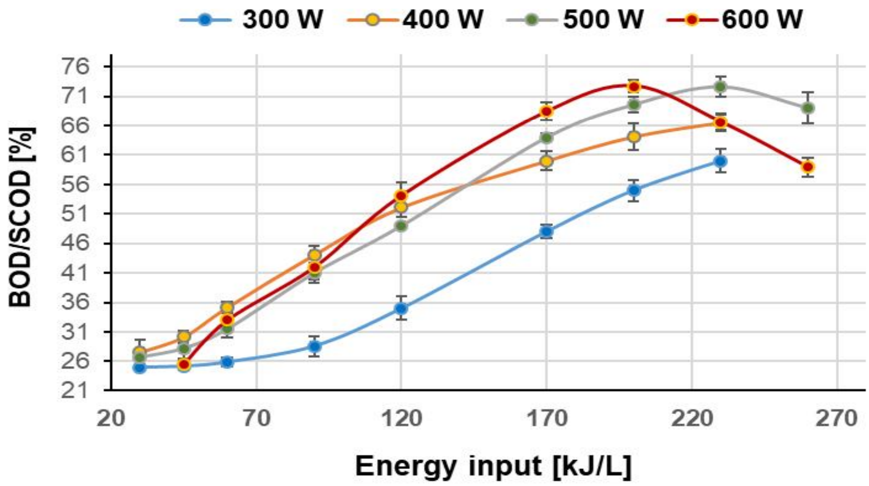

Figure 2

BOD/SCOD of sludge pre-treated by MW
with different power intensity (W) and energy input (kJ/L)
(BOD-biochemical oxygen demand, SCOD-soluble chemical oxygen demand)

Similarly to the change of organic matter solubility, aerobic biodegradation is supposed to be inhibited by compounds formed in high energy and/or high temperature MW treatment [16]. It should be noted that there was no significant difference in BOD/SCOD if the power intensity was varied in the range of 400–600 W when energy input of 50–120 kJ/L was applied.

For the evaluation of the efficiency of MW treatment for enhanced biodegradability, mesophilic AD tests are the most frequently used method. In general, MW irradiation is suitable to intensify the anaerobic digestion process. The main effect of MW irradiation, applied as a pre-treatment step in biogas technology, is attributed to the hydrolysis stage of anaerobic digestion [17]. Hydrolysis is considered the main rate-limiting step of AD; therefore, MW pre-treatments are suitable to increase the specific biogas yield (on TS, i.e. total solid or VS, i.e. volatile solid basis), and also accelerate the digestion process.

Sludge contains different microbial cells and organic and inorganic components that work together with extracellular polymeric substances to build up a special physicochemical structure that is resistant to anaerobic digestion. Kavitha et al. [18] concluded that the energy efficiency of MW treatments aiming at biomass lysis and enhanced hydrolysis (and therefore increasing the methane production) was higher if

EPS and sludge flocs are disrupted in a preliminary stage using mechanical (ultrasonication) and/or chemical (alkali dosage) methods. These methods are suitable for decreasing the average size of solid particles and increasing the specific surface area of particles and flocs. These effects result in easier penetration for electromagnetic waves (higher penetration depth) and higher dissipation of irradiated MW energy.

The release of divalent cations from sludge flocs, which is due to i) the deflocculating effect of MW and ii) release of ionic components from intracellular space from cell wall disruption, has a synergetic effect that leads to an increase in ionic strength. Therefore, further research is needed to investigate the effect of the change of ionic strength on the microbial activity in the different stages of the anaerobic digestion (AD) process [2]. Effects of high-intensity microwave irradiation on contaminants of emerging concern (CECs) have not yet been examined in detail. Further research is needed to explore the pathways of transformation of CECs exposed to a high-intensity electromagnetic field, mainly in real, multicomponent, and biological active systems such as sludge.

Beyond the promising results related to MW irradiation when using it as a pre-treatment method for AD, it should be highlighted that the production of wastewater sludge in sewage treatment plants is continuous, therefore the detailed investigation of continuous flow microwave sludge processing is needed. In our research the mesophilic biogas production of sludge samples treated at different MW power and specific MW energy input (determined from volumetric flow rate, residence time in microwave reactor, sample volume and magnetron power) was measured in a 30-day fermentation period. The specific volumetric biogas product was given on a TS basis *(Figure 3).* It can be concluded that continuous flow MW irradiation of sludge increased the biogas product. Application of MW treatment with an energy input of 120 kJ/L and 220 kJ/L resulted in biogas yield increments of 174% and 210%, respectively, compared to the biogas product of raw sludge (118 mL/g TS, data not shown in figures).

Results of the AD tests show that at higher levels of irradiated energy (120–220 kJ/L) the power intensity of MW treatment has no significant effect on the biogas production *(Figure 3)*. If irradiated energy was in the range of 45–60 kJ/L the power intensity has a slight effect on the biogas yield. The difference between the biogas yield of sludge exposed to 300 W and 600 W MW power with energy input of 45 and 60 kJ/L was 15% and 21%, respectively. Microwave treatments applied prior to AD are reported as a suitable method to increase the biogas production. Several studies highlighted that microwave irradiation alone – or in combination with chemical methods – under optimized conditions resulted in a higher methane ratio in the produced biogas [19], [20]. There are no known sludge pre-treatment methods especially designed and optimized to produce substrate exclusively to methanogenic archaea.

Microwave irradiation is widely used in the industry, but there is not enough experience derived from semi-pilot and pilot systems to implement directly to industry scale sludge processing. Among many factors of scale-up, one difficult question that arises is how to define control parameters. In lab-scale studies the biogas yield is given as the maximum achievable cumulative values in 30–60 days anaerobic digestion process. The AD process operating on the industrial scale through continuous flow and one-stage digester in the mesophilic temperature range is decidedly not controlled to achieve the maximum biogas yield. On the other hand, the economic motivations are to minimize the capital costs for installing digesters and other service units and to minimize the operation expenditures of AD plants, while at the same time the technology should remain 'flexible' to variable feed quantity and quality. From this aspect the biogas production rate is considered as an important parameter [21].

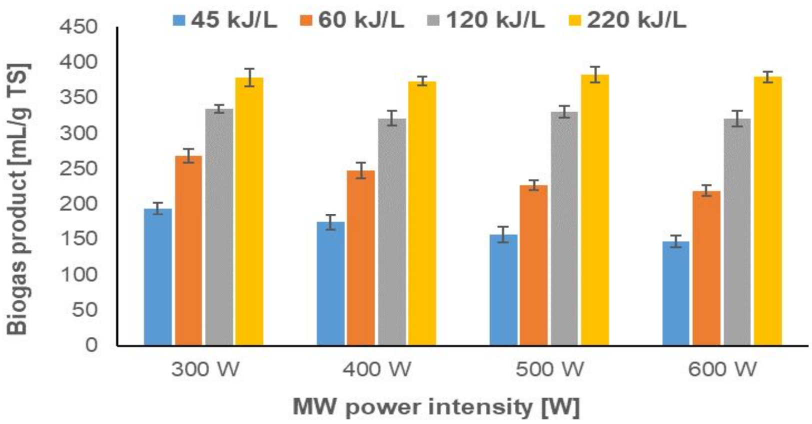

Figure 3
Biogas product of MW pre-treated sludge
(biogas product is given on total solid basis)

In order to quantify the effect of MW treatment on the rate of anaerobic digestion the cumulative biogas production of the first period (0–15 days) and second period (16–30 days) of the AD process was calculated from the daily volumetric biogas production. Results related to the contribution of the two periods to total biogas yield show that MW pre-treatment can accelerate the anaerobic digestion process. Compared to the raw sludge (Cont), the MW pre-treated sludge produced a greater amount of biogas in the first 15 days of fermentation *(Figure 4)*.

A study by Gil et al. [16] verified the positive effect of MW irradiation on methane production rate for mixed sludge (primary and secondary sludge, originating from 80% municipal and 20% agrifood-industry wastewater). The results of our AD tests show that the total cumulative biogas product is mainly influenced by the irradiated energy; in the range of energy input of 120–220 kJ/L the power intensity has no significant effect on biogas production. But considering the daily volumetric biogas production, our results reveal that the power intensity applied has an effect on the rate of anaerobic digestion process. As *Figure 4* shows, applying the same energy input of 220 kJ/L with different power intensity (300–600W), the lower power intensity resulted in accelerated anaerobic digestion, with approximately 80% of the total biogas volume being produced in the first 15 days of the fermentation process.

Therefore, in continuous flow MW treatment of sludge, the MW power intensity does not affect the cumulative biogas yield, but can be an influential parameter for the rate of anaerobic digestion.

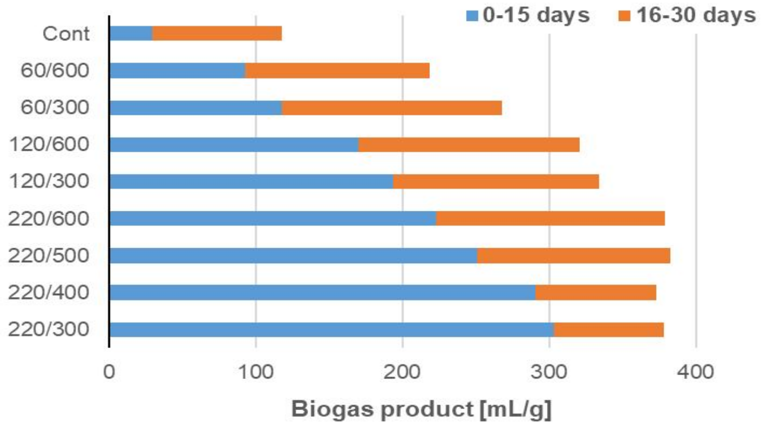

Figure 4

Cumulative biogas product in the 1st (0–15 days) and 2nd (16–30 days) period of AD process

(Cont- non treated sample; MW pre-treatments are encoded as energy input [kJ/L] / power intensity [W])

4. CONCLUSIONS

Many studies have verified the rapid and strong sludge disintegration effect of microwave (MW) irradiation, applying it as stand-alone method or coupled with conventional chemical method, advanced oxidation processes, enzymatic treatment, or ultrasonication. Increased organic matter solubility is induced by disintegration, which is also manifested in higher biogas production. Most studies available present results obtained from batch MW treatment of municipal waste activated sludge; however, detailed information about the efficiency and applicability of continuously flow MW treatment for enhanced biodegradability is missing. In our earlier research the advantages of batch MW pre-treatment for enhanced disintegration of sludge and accelerated anaerobic digestion were verified. In the present research, the efficiency of continuous flow MW treatment of thickened primary dairy industry sludge was investigated. Our experimental results verified that MW pre-treatment was suitable to increase the SCOD/TCOD, which indicated an enhanced degree of solubilization. Furthermore, the aerobic biodegradability (expressed in BOD/SCOD) and the biogas production improved by 170–210% (depending on the MW power and irradiated energy). Results revealed that the power intensity of MW irradiation has an effect on COD solubilization and irradiated energy affects biogas yield. Increasing the irradiated energy (energy input) and microwave power intensity resulted in higher organic matter solubility. However, irradiated energy at levels over 90 kJ/L had a negative effect on COD solubility, and 200 kJ/L at 600 W power intensity, worsened aerobic biodegradability. Results of mesophilic AD tests revealed that the biogas yield increased due to the use of MW irradiation as a pre-treatment; furthermore, the rate of anaerobic digestion process accelerated. Analysis of the effects of MW-related process parameters on the efficiency of AD shows that the cumulative biogas production is influenced mainly by the energy input. The power intensity of MW irradiation has only a slight effect on biogas yield, if energy input exceeds 120 kJ/L, but it can be an influential parameter for the rate of anaerobic digestion/biogas production. Our experiments verified that continuous flow microwave treatment can assist in achieving higher utilization of organic matter in primary food industry sludge in the form of biogas. We conclude that MW processing technology – under optimized conditions – can be an answer to the challenge of achieving higher utilization of waste and byproduct towards the valorization of secondary resources. With well-designed MW pre-treatments the availability of organic matter fraction of sludge can be increased, which will lead to accelerated anaerobic digestion and higher biogas yield. These results contribute to achieving higher valorization of sludge with reduced environmental impact of digestate and higher energy efficiency of sludge utilization technologies.

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