The influence of energy input on the particle size of disintegrated excess sludge in the ultrasonic disintegration process

Łukasz Skórkowski and Ewa Zielewicz

ABSTRACT

The objective of this research was to examine the influence of energy input on the particle size distribution of disintegrated sludge. The change of particle size distribution indicates the deagglomeration of flocs and disruption of micro-colonies. As the digestibility of sludge increases with dispersion, particle size analysis is an important factor in evaluating the disintegration process. Four different levels of energy input were used in the research: 10–100 kWh·m^{-3}. All samples showed significant changes as far as dispersion ($kd_{CST} = 22.98–74.67$, $kd_{FCOD} = 3.23–18.46$), lysis ($kd_{SCOD} = 4.22–12.09$), acidification ($kd_{VFAs} = 1.78–12.61$), nitrogen release ($kd_{TN} = 4.02–21.61$) indicators were concerned. Results indicate the gradual decrease of measured particle size with increasing energy input. The energy supplied to the disintegration process primarily promotes deagglomeration and with the rise of energy input, the destruction of cells. For $E_V = 50$ and 100 kWh·m^{-3} an increased occurrence of lysis effects and increase in particle fraction <99.9 μm was noted. The highest efficiency evaluated by increase of filtered chemical oxygen demand (FCOD) and soluble COD (SCOD) per unit of volumetric energy – ΔCOD and ΔSCOD (mgO$_2$·Wh^{-1}) was obtained for $E_V = 10$ WhL^{-1}, which corresponds to the most significant change in particle size distribution. The volume of particles <99.9 μm rose from 1.92% for non-disintegrated sludge to 26.62% for volumetric energy 100 kWh·m^{-3}.

Key words | excess sludge, laser diffraction, particle size, sludge pre-treatment, specific energy, ultrasonic disintegration

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INTRODUCTION

Due to the large amounts of sludge generated in the process of wastewater treatment by activated sludge technology, the implementation of techniques reducing the amount of excess sludge and its impact on the environment is essential (Cimochowicz-Rybicka 2013). Anaerobic digestion is the most widespread method of sewage sludge processing. In order to achieve the most favourable effects of stabilization, excess sludge preconditioning is recommended (Zubrowska-Sudol & Walczak 2014; Wang *et al.* 2017; Zhao *et al.* 2017). One of the most commonly used processes of sludge preconditioning is disintegration. As the rate-limiting step in sludge digestion is hydrolysis (Bougrier *et al.* 2006), the main purpose of disintegration is to enhance hydrolysis and consequently achieve enhanced stabilization, defined as improved solids destruction (Zielewicz 2016a) and dewatering of the digested sludge as well as enhanced biogas (methane) production (Tiehm *et al.* 1997; Zawieja & Wolny 2013; Zubrowska-Sudol & Walczak 2014; Zhen *et al.* 2017). Disintegration of excess sludge in wastewater treatment plants (WWTPs) is usually carried out after mechanical thickening and before the digestion tank (Zielewicz 2016a, 2016b; Wang *et al.* 2017; Zhao *et al.* 2017). Disintegration of excess sludge covers a number of processes taking place in sewage sludge due to the application of various types of energy. Disintegration can be divided into mechanical (e.g.: mixing, grinding, ultrasonic disintegration) and non-mechanical disintegration (e.g.: physical, chemical and biological disintegration).

Ultrasonic preconditioning is one of the most widespread mechanical disintegration methods (Tiehm *et al.* 1997; Gogate & Kabadi 2009; Zielewicz 2016a, 2016b). The ultrasonic field of precisely specified parameters influences

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the physical and chemical properties of sludge. Sludge flocs deagglomeration, disruption of microorganisms' cell walls and release of its content into the aqueous phase are the expected effects of disintegration (Neis 2002). Ultrasound cavitation is considered the primary mechanism causing changes in sonicated sludge (Tiehm *et al.* 1997; Zielewicz 2016a, 2016b). The implosion of gas micro-bubbles results in localized increases in pressure and temperature as well as the generation of free radicals. The shear forces generated at low frequency (20–30 kHz) and high intensity are considered the main mechanism of the ultrasonic disintegration process (Pilli *et al.* 2011).

According to Bougrier *et al.* (2005, 2006) specific energies between 1,000 and 7,000 $kJ{\cdot}kg_{TS}^{-1}$ were efficient at increasing biogas production from disintegrated sludge, but the increase of specific energy up to 15,000 $kJ{\cdot}kg_{TS}^{-1}$ did not lead to further increases of biogas production. The biogas yield for ultrasonically disintegrated sludge is 24–138% higher than for untreated sludge (Joo *et al.* 2015; Wang *et al.* 2017). Salsabil *et al.* (2009) report using ultrasonic treatment with specific energies between 3,600 and 108,000 $kJ{\cdot}kg_{TS}^{-1}$ and achieving beneficial results of biogas production (increases between 10 and 40%). Carrère *et al.* (2010) put the threshold of effective disintegration between 1,000 and 16,000 $kJ{\cdot}kg_{TS}^{-1}$. Zielewicz (2007, 2016a, 2016b) identifies the energy required for the sludge ultrasonic disintegration as being between 10 and 100 kWh m^{-3}.

The results of ultrasonic disintegration are dependent not only on the amount of energy, but also particle size and total solids (TS) content (Show *et al.* 2007; Zielewicz 2007, 2016a, 2016b; Carrère *et al.* 2010). The TS concentration and the particle size of the thickened sludge (with the possible addition of polyelectrolyte or coagulant) (Chen *et al.* 2018) influences fermentation. As the digestibility of sludge increases with dispersion (Vavilin *et al.* 1996), the particle size analysis of sludge flocs is an important factor in evaluating the disintegration process. Particle size measurements can be conducted using various methods e.g.: direct microscopic observation (Simonetti *et al.* 2014), sedimentation (Hilligardt & Hoffmann 1997), laser diffraction (Bougrier *et al.* 2005; Bieganowski *et al.* 2012; Simonetti *et al.* 2014).

Laser diffraction is a widespread technique in soil science and sedimentology (Molinaroli *et al.* 2011; Bieganowski *et al.* 2012; Vendelboe *et al.* 2012). The use of laser diffraction in the assessment of the sludge flocs has been gaining popularity (Bieganowski *et al.* 2012; Simonetti *et al.* 2014) in recent years, but is still not widely used. Estimation of the particle size distribution utilizing the laser diffraction method is based on the measurement of laser light scattering off the measured particles. The angle of the scattered light reflected off the measured particle defines the size of that particle (Malvern 2015). The recorded energy of the scattered light is calculated by the analysers' software using the Fraunhofer or Mie theory to achieve a particle size distribution of the measured samples (Bieganowski *et al.* 2012; Malvern 2015).

The aim of this research was to examine the influence of energy input on the particle size (measured with the laser diffraction method) of the ultrasonically disintegrated sludge, utilizing a prototype semi-technical scale mosaic head with a short conical emitter.

MATERIALS AND METHODS

Excess sludge used in the research was obtained from a municipal sewage treatment plant located in the Silesian region of Poland. The excess sludge was collected after mechanical thickening.

The analysis of: TCOD (the total chemical oxygen demand of sludge), filtered COD (FCOD) and soluble COD (SCOD) were performed using the dichromate method (ISO 6060-1989); the volatile fatty acids (VFAs) using the esterification method (Hach Lange); the total nitrogen (TN) using Koroleff digestion (peroxodisulphate) and photometric detection with the 2.6-dimethylphenol method (EN ISO 11905-1). The FCOD, SCOD, VFAs and TN evaluation in the aqueous phase was performed after sludge centrifugation for 30 min at a speed of 20,000 rpm in a refrigerated centrifuge (18 °C). For the FCOD test the supernatant was filtered through filter paper (pore size 3 μm); for the SCOD, VFA and TN analysis the supernatant was filtrated through a cellulose acetate membrane filter (Nalgene, pore size 0.45 μm) (Zielewicz 2007, 2016a, 2016b). The chemical tests were performed using the spectrophotometric method (HACH Lange DR5000 UV-Vis spectrophotometer and HACH Lange vial tests). The characteristics of the excess sludge and its supernatant before disintegration are presented in Table 1.

The effects of the sludge disintegration were evaluated based on the values of the indicators for dispersion, lysis, acidification and nitrogen release. The dispersion indicators were calculated as the ratio of FCOD (kd_{FCOD}) and capillary suction time (CST; kd_{CST}) before and after the process (Zielewicz 2016a, 2016b):

$$kd_{FCOD} = \frac{FCOD_{UT}}{FCOD_{NT}} \tag{1}$$

Table 1 | Characteristics of excess sludge and its supernatant before disintegration

Parameter							
Sludge				**Supernatant**			
Hydration %	**Capillary suction time (CST) s**	**Chemical oxygen demand (TCOD) $mgO_2 \cdot L^{-1}$**	**pH**	**Chemical oxygen demand ($FCOD_{NT}$) $mgO_2 \cdot L^{-1}$**	**Soluble COD ($SCOD_{NT}$) $mgO_2 \cdot L^{-1}$**	**Volatile fatty acids (VFA_{NT}) as CH_3COOH $mg \cdot L^{-1}$**	**Total nitrogen (TN_{NT}) $mgN \cdot L^{-1}$**
97.46	11.30	17,120	6.9	267.00	117.01	25.30	12.00

$$kd_{CST} = \frac{CST_{UT}}{CST_{NT}} \quad (2)$$

where:

FCOD – of filtered sludge supernatant (fraction $<3\,\mu m$), mg O_2 L^{-1},
CST – s,
$_{NT}$ – before disintegration,
$_{UT}$ – after disintegration.

The cell lysis indicator (kd_{SCOD}), acidification indicator (kd_{VFA}), and nitrogen release indicator (kd_{TN}) were calculated using the following equations (Zielewicz 2007, 2016a, 2016b):

$$kd_{SCOD} = \frac{SCOD_{UT}}{SCOD_{NT}} \quad (3)$$

$$kd_{VFA} = \frac{VFAs_{UT}}{VFAs_{NT}} \quad (4)$$

$$kd_{TN} = \frac{TN_{UT}}{TN_{NT}} \quad (5)$$

where:

SCOD – in supernatant (fraction $< 0.45\,\mu m$), mg O_2 L^{-1},
VFAs – as CH_3COOH, $mg \cdot L^{-1}$,
TN – $mgN \cdot L^{-1}$,

A high-power ultrasonic disintegrator, custom built at Silesian University of Technology, was used in the research. The disintegration set consisted of a WK-2010 ultrasonic generator and mosaic head with a large diameter ($d_E = 120$ mm) and a short conical emitter – Figure 1. The disintegration was performed in the cylindrical vessel (diameter $d_K = 220$ mm), with the distance between the emitter and the bottom of the vessel $h_D = 3.0$ cm. The ratio of the surface area of the emitter and the disintegration vessel was $A_{CH}/A_E = 3.5$. The resonance frequency for the excess sludge used in the studies was established at 21.22 kHz. The disintegration process was performed for the generator power $P_G = 950$ W and head's power $P_{UT} = 400.66$ W.

The energy parameters of the ultrasonic disintegration process were calculated using Equations (6)–(10) (Zielewicz-Madej 2003; Zielewicz 2007, 2016a, 2016b):

$$I_{UD(E)} = P_{UT} \cdot A_E^{-1} \quad (6)$$

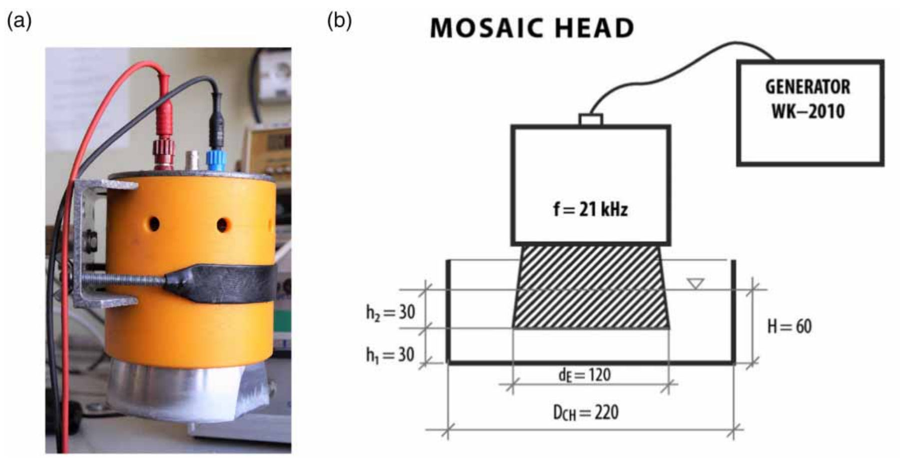

Figure 1 | Ultrasonic disintegrator setup – disintegration vessel, mosaic head.

$$I_{UD(CH)} = P_{UT} \cdot A_{CH}^{-1} \quad (7)$$

$$E_V = P_{UT} \cdot t_{UT} \cdot V_{CH}^{-1} \quad (8)$$

$$E_S = P_{UT} \cdot t_{UT} \cdot TS^{-1} \cdot V_{CH}^{-1} \quad (9)$$

$$PD_{UD} = P_{UT} \cdot V_{CH}^{-1} \quad (10)$$

where:

A_E – surface area of the emitter, cm^2,
A_{CH} – surface area of the disintegration vessel, cm^2,
P_{UT} – workstation power transmitted by the transducer, measured during sonication, kW,
t_{UT} – sonication time, h,
$I_{UD(E)}$ – the intensity of the ultrasound field in relation to the surface area of the emitter, Wcm^{-2},
$I_{UD(CH)}$ – the intensity of the ultrasound field in relation to the surface area of the vessel, Wcm^{-2},
V_{CH} – sludge volume, m^3,
E_V – consumption of energy per unit volume corresponding numerically to the energy density, W_{UP} kWh m^{-3},
E_S – consumption of energy per unit dry weight (TS) of sludge, so-called specific energy, kWh kg_{TS}^{-1},
PD_{UD} – power density, kWL^{-1}.

The parameters of ultrasonic disintegration are presented in Table 2.

The other parameters of ultrasonic disintegration were: the intensity of the ultrasound field in relation to the surface area of the emitter $I_{UT(E)} = 3.54$ W cm^{-2}, the intensity of the ultrasound field in relation to the surface area of the vessel $I_{UT(CH)} = 1.05$ Wcm^{-2}, power density $PD_{UT} = 572.34$ W L^{-1} (Eder & Günthert 2002; Zielewicz 2007, 2016a, 2016b).

Particle size analysis was performed using a Malvern Mastersizer 3000 laser diffraction particle size analyzer with the measuring range of 0.01–3,500 μm equipped with a Hydro EV flexible volume wet dispersion unit. The Mastersizer analyser uses the full Mie theory which completely solves the equations for interaction of light with matter (Malvern 2015).

Table 2 | Parameters of ultrasonic disintegration

$E_{V(UT)}$ Consumption of energy per unit volume – volumetric energy (kWh m^{-3})	t_{UD} sonication time (s)	$Es_{(UT)}$ Consumption of energy per unit dry mass (TS) – specific energy (kWh kg_{TS}^{-1})
10	63	0.39
20	126	0.79
50	315	1.97
100	630	3.93

The volume of sludge used in the research was determined by obscurance (Malvern 2015). The optimum results were achieved for 10% obscurance (Malvern 2015). The working parameter of the pump and stirrer were experimentally established to provide stable results during the procedure. The measurements were performed in four repetitions.

The samples collected for measurement from the sonication vessel were averaged (the volume of the sludge from the chamber was slowly stirred after disintegration, as the design of the prototype head and disintegration vessel did not allow for mixing during sonication (Figure 1(b)), thus the obtained results include the effects of the vessel's dead volume.

RESULTS AND DISCUSSION

The results of the CST, FCOD, SCOD, VFAs and TN after disintegration are presented in Table 3.

The changes of the FCOD, SCOD, VFAs and TN in the form of sludge disintegration indicators were calculated according to Equations (6)–(10) and are presented in Table 4.

The results show a gradual increase in the measured parameters with increasing energy input. It should be noted that the increase in the $E_{V(UT)}$ value from 10 to 20 kWh m^{-3} did influence the dispersion measured as the changes in FCOD and CST values but did not heavily influenced parameters connected with cell lysis. The increase of the dispersion indicators is also noticeable in particle size distribution, with the surge in the results for $Ev_{(UT)} = 0$ and 10 kWh m^{-3} (Figure 2 and Table 5).

Further increases in energy input resulted in the increase of all of the measured parameters (all the indicators rose significantly). It should be noted that there was a surge in the lysis and nutrient release indicators between $E_{V(UT)}$ 20 and 50 kWh·m^{-3}. For low volumetric energy levels (10, 20 kWh·m^{-3}) the rise of the kd_{SCOD} and kd_{VFAs} was comparable – kd_{SCOD} rose to 4.22 and 4.62, kd_{VFA} to 1.78 and 1.75, respectively. The results for $E_{V(UT)}$ 50 and 100 kWh·m^{-3} were much higher (kd_{SCOD} rose to 9.67 and 12.09, kd_{VFA} to 3.01 and 12.61 respectively). The nutrient release indicator kd_{TN} reached similar values for volumetric energy 10 and 20 kWh·m^{-3} 4.02 and 4.43, respectively. For $E_{V(UT)} = 50$ and 100 kWh·m^{-3} the kd_{TN} was considerably higher and reached 10.63 and 21.17, respectively.

Table 3 | Characteristics of excess sludge and its supernatant after disintegration

Parameter			Volumetric energy – $E_{V(UT)}$ (kWh m^{-3})			
			10	20	50	100
Sludge	CST	s	259.70	276.75	385.50	843.73
	pH		6.72	6.62	6.70	6.84
Supernatant	$FCOD_{UT}$	$mgO_2 \cdot L^{-1}$	862.01	1,102.00	2,199.02	4,930.09
	$SCOD_{UT}$	$mgO_2 \cdot L^{-1}$	494.00	539.97	1,131.01	1,415.00
	$VFAs_{UT}$ as CH_3COOH	$mg \cdot L^{-1}$	42.80	42.20	72.51	304.00
	TN_{UT}	$mgN \cdot L^{-1}$	48.20	53.20	127.61	254.00

Table 4 | Characteristics of excess sludge disintegration indicators

$E_{V(UT)}$ Consumption of energy per unit volume (kWh m^{-3})	Disintegration time (s)	Sludge disintegration indicators				
		Sludge dispersion indicator		Cell lysis indicator	Acidification indicator	Nitrogen release indicator
		kd_{FCOD}	kd_{CST}	kd_{SCOD}	kd_{VFA}	kd_{TN}
10	63	3.23	22.98	4.22	1.78	4.02
20	126	4.13	24.49	4.62	1.75	4.43
50	315	8.24	34.12	9.67	3.01	10.63
100	630	18.46	74.67	12.09	12.61	21.17

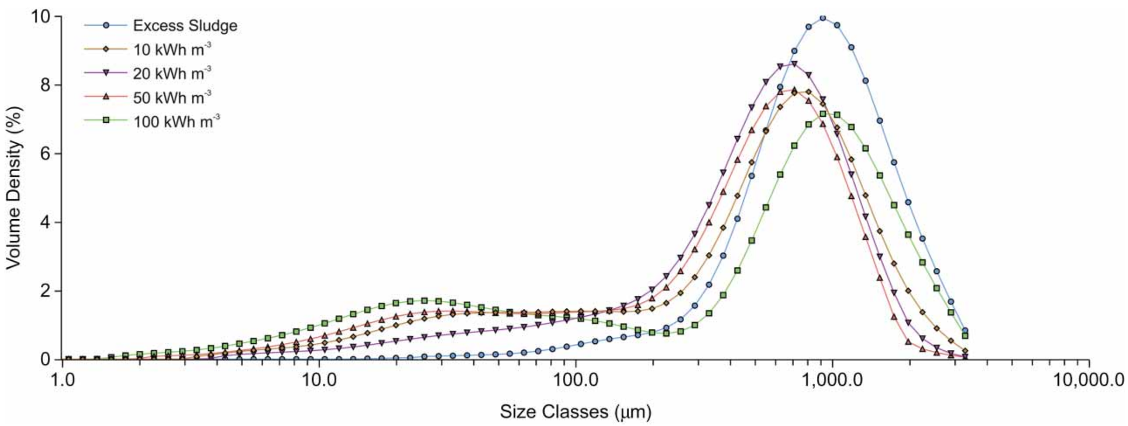

Figure 2 | Particle size distribution of disintegrated sludge for $E_{V(UT)}$ 10–100 kWh·m^{-3}.

The results monitored as kd_{TN} are associated with the release of organic substances, in the form of extracellular polymeric substances (EPS) and intracellular material, into the aqueous phase. EPS are found in the interior of microbial aggregates – sludge flocs – and consists mainly of proteins and carbohydrates, but nucleic acids, lipids and other polymeric compounds are also present (Sheng *et al.* 2010).

The particle sizes presented in Table 5 indicates d_1–d_{90} cut diameters (d_x are standard percentile readings from analysis, i.e. d_{10} – the size in microns at which 10% of the sample is smaller and 90% is larger). D [4,3] is the mean diameter of the sphere, equivalent in respect of volume or mass (Malvern 2015)), and particle fraction <99.9 μm.

With the increase of volumetric energy, the particle size distribution also changes significantly. The mean diameter of disintegrated sludge (when compared to that of non-disintegrated sludge) decreases with the increasing volumetric energy. The content of smallest particle fraction, below 99.9 μm, also increases with the rise of the $E_{V(UT)}$ – Table 5. The high values of kd_{FCOD} and kd_{CST} indicators for all disintegration times can be attributed to the

Table 5 | Cut diameters of sludge for specific disintegration times

	Volumetric energy $E_{V(UT)}$ (kWh m^{-3})				
Particle size (μm)	**0**	**10**	**20**	**50**	**100**
d_1	70.4	6.81	9.42	5.49	3.57
d_{10}	361	37.4	80.5	27.9	18.0
d_{25}	580	197	289	149	81.6
d_{50}	891	579	552	482	677
d_{75}	1,340	958	870	800	1,180
d_{90}	1,910	1,410	1,240	1,140	1,770
D [4,3]	1,030	669	627	541	778
Volume below 99.9 μm (%)	1.72	18.78	11.58	21.31	26.62

Table 6 | The indicators of energy consumption

	$E_{V(UT)}$ (Wh·L^{-1})			
Increase of substances per unit of energy	**10**	**20**	**50**	**100**
ΔFCOD (mgO_2·Wh^{-1})	74.5	41.75	38.64	46.63
ΔSCOD (mgO_2·Wh^{-1})	22.7	13.65	17.28	11.48

moderately low TS content of the excess sludge used in this research (2.54 g·L^{-1}). According to Shaw *et al.* (2007), Carrère *et al.* (2010), the optimal TS content for US disintegration is between 2.3 and 3.2 g·L^{-1}; similar results for TS between 2–4 g·L^{-1} were also reported by Zielewicz (2016a).

As presented in Table 5 and Figure 2, the increase in energy input corresponds to the decrease of sludge floc diameters, i.e. for volumetric energy $E_{V(UT)}$ 100 kWh m^{-3} the cut diameter of more than 26.62% particles is lower than 99.9 μm, for non-processed sludge it is only 1.72%. The D [4,3] mean diameter reduction also corresponds to the increase in the energy input, except for the highest E_V value. This result can be attributed to the stronger influence of non-disintegrated sludge (dead volume) and susceptibility of very small particles on reagglomeration (products of effective cell lysis can behave as polyelectrolytes). In the literature (Simonetti *et al.* 2014) the sludge is often mixed during the process, which reduces the influence of the disintegration vessel's dead volume on the particle measurement results. Thus the results of the sludge floc particle measurement differ, depending on the mixing conditions during the disintegration process. In this research the sludge was gently mixed after disintegration, thus the dead space volume was taken into account. As shown in Tables 3–5, the 10 and 20 kWh m^{-3} energy input produced similar results as far as kd_{SCOD}, kd_{VFA}, kd_{TN} indicators and D [4,3] diameter are concerned. The dispersion indicators were however slightly higher for the 20 kWh m^{-3} energy input. The 50 and 100 kWh m^{-3} energy input levels produced significantly better results, however the results did not justify much higher energy use (the efficiency decreases). As is presented in Table 6, the highest efficiency evaluated by increases of FCOD and SCOD per unit of volumetric energy – ΔCOD and ΔSCOD (mgO_2·Wh^{-1}) was obtained for Ev = 10 Wh L^{-1}, which corresponds to the most significant change in particle size distribution (between results for $E_{V(UT)}$ 0 and 10 kWh m^{-3} – Figure 2, Table 5).

CONCLUSIONS

The results of the experiment conducted show the connection between the sludge floc diameter and energy input – the measured particle sizes decrease with the increase of energy input. The analysis of the disintegration indicators and particle size distribution suggests that energy supplied to the disintegration process primarily promotes deagglomeration of larger flocs and with the rise of energy input, the destruction of cells. The surge in the kd_{SCOD} and kd_{TN} indicators for volumetric energy $E_{v(UT)}$ 50 and 100 kWh m^{-3} suggests an increased occurrence of lysis effects. The influence of prolonged sonication time ($E_{V(UT)}$ rise) can be especially observed in the increase of particles in the 'smallest' fractions of the disintegrated sludge (mainly <100 μm). The high values obtained for the disintegration indicators can be attributed to the favourable properties, mainly the low concentration of TS (2.54%) of the thickened excess sludge used in the research. Disintegration of the excess sludge in the ultrasonic field was the most economically feasible for the 10 kWh m^{-3} energy input, which was especially visible in the dispersion monitored as kd_{FCOD}, kd_{CST} indicators and the growth of the particle fraction <99.9 μm by 17%.

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