Application of dielectric constant measurement in microwave sludge disintegration and wastewater purification processes

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ABSTRACT

It has been numerously verified that microwave radiation could be advantageous as a pre-treatment for enhanced disintegration of sludge. Very few data related to the dielectric parameters of wastewater of different origins are available; therefore, the objective of our work was to measure the dielectric constant of municipal and meat industrial wastewater during a continuous flow operating microwave process. Determination of the dielectric constant and its change during wastewater and sludge processing make it possible to decide on the applicability of dielectric measurements for detecting the organic matter removal efficiency of wastewater purification process or disintegration degree of sludge. With the measurement of dielectric constant as a function of temperature, total solids (TS) content and microwave specific process parameters regression models were developed. Our results verified that in the case of municipal wastewater sludge, the TS content has a significant effect on the dielectric constant and disintegration degree (DD), as does the temperature. The dielectric constant has a decreasing tendency with increasing temperature for wastewater sludge of low TS content, but an adverse effect was found for samples with high TS and organic matter contents. DD of meat processing wastewater sludge was influenced significantly by the volumetric flow rate and power level, as process parameters of continuously flow microwave pre-treatments. It can be concluded that the disintegration process of food industry sludge can be detected by dielectric constant measurements. From technical purposes the applicability of dielectric measurements was tested in the purification process of municipal wastewater, as well. Determination of dielectric behaviour was a sensitive method to detect the purification degree of municipal wastewater.

Key words | dielectric constant, disintegration, microwave, sludge, wastewater

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INTRODUCTION

The degree of particulate organic matter disintegration and hydrolysis of macromolecular components is one of the key issues for the whole efficiency of sludge utilization processes. Polymeric and insoluble compounds, such as proteins, fats, carbohydrates, and their derivatives, are decomposed into smaller molecules by hydrolysis, such as amino acids, monosaccharides, alcohols, or fatty acids. Due to the complex particle structure and the presence of strong cell membranes, it is difficult for sludge to biodegrade directly under aerobic or anaerobic conditions. The main aim of sludge pre-treatment technologies is to disrupt the cell membranes, leading to lysis of the cells of microorganisms, and to accelerate the hydrolysis of macromolecular components. Methods of sludge disintegration can be based on mechanical, thermal, and chemical processes or a combination of them. Among the different sludge handling methods, microwave radiation is successfully applied in the process of the hygienization, dewatering, drying, and pre-treatment stages of anaerobic digestion (AD). Treatments by microwave irradiation offer advantages such as rapid and direct heating, reduction of energy loss, and better controllability compared to conventional thermal methods. Dielectric heating has a higher energetic efficiency than conventional heating due to the different way of energy transfer (Leonelli & Mason 2010). In conventional heating, the electric

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energy is converted into heat then the heat is transferred to the surface of the material and the heat penetrates from the surface towards the core of materials. During dielectric heating, microwave energy is delivered into the material directly, and heating occurs inside the materials (volumetric heating) due to the molecular interactions with the electromagnetic field (Appleton *et al.* 2005).

A microwave method is suitable to increase the degree of conversion of organic matter in sludge flocs into easily accessible compounds for decomposing microorganisms. Disruption of cell membranes leads to the release of the protoplasm enzymes responsible for increasing the ammonia and phosphate concentrations in sludge liquor. Considering the efficiency of sludge disintegration and the biogas production kinetic, microwave treatments have been found to be superior to conventional thermal treatment processes. The most frequently used method of characterizing the change in the efficiency of disintegration is the measure of the soluble chemical oxygen demand to total chemical oxygen demand (SCOD/TCOD) (Tyagi & Lo 2011). Lysis of microbial cell walls and disintegration of sludge flocs is determined by hydrogen binding, hydrophobic interactions, and the concentration of divalent cations. The increment of Ca^{2+} and Mg^{2+}, as a component of phospholipids, is due to the disruption of cell walls (Ahn *et al.* 2009). Therefore, measurement of the divalent ion concentration in the liquid phase can be a suitable indirect method of determining the degree of disintegration (DD). Furthermore, the strong effect of microwave heating on the cell wall disruption and sludge floc disintegration was confirmed by microscope observations as well (Bohdziewicz & Sroka 2006).

The results of preliminary studies established that the release of organic matter is mainly influenced by the final temperature and temperature ramp of the sample during the process and the duration of microwave radiation. If the final temperature is higher than 80 °C, a partial conversion of NH_4^+ into gas phase as NH_3 can be observed, decreasing the organic matter concentration of the liquid phase (Eskicioglu *et al.* 2007). Furthermore, if the temperature of the sample reaches the boiling point, a slight decrease of COD concentration occurs (Bohdziewicz *et al.* 2011). Because the heat stress is influenced by the temperature ramp, which is dependent on the strength of the electromagnetic field, the power of microwave radiation can also be a determinative process parameter (Beszédes *et al.* 2011). Numerous reports have concluded that microwave pre-treatment is suitable for enhancing the efficiency of the AD process, which lead to the production of a larger amount of biogas product and an accelerated biogas production rate (Bougrier *et al.* 2007). Increments of the biogas production and utilization value of biogas (calorific value and quality determined mainly by the methane, carbon dioxide, and hydrogen sulfide concentrations) are affected by the type of processed sludge.

Thermal and athermal effects of microwave radiation have been investigated for many years. Athermal effects refer to a special effect of the electromagnetic field in the microwave frequency range that is not associated with the temperature increase of irradiated materials. Microwave radiation can polarize the side chains of macromolecules, and a change in the dipole orientation of polar molecules can occur. High-frequency electromagnetic waves induce structural changes and orienting effects within the materials. It can be observed that at frequencies of 915 and 2,450 MHz, which are commonly used for industrial and scientific applications, the energy of a microwave photon is insufficient to break hydrogen and covalent bonds or to induce chemical reactions directly (Kappe 2004).

The heat generation efficiency of microwaves is determined by the frequency, concentration, structure and composition of the irradiated matrix, particle size, viscosity, individual, and integral dielectric properties of compounds, penetration depth, and so on. The heating mechanisms of microwave radiation can be attributed to dielectric polarization and ionic conduction. Depending on the applied frequency, dipolar molecules, such as water, move in time as the polarity of the electromagnetic field varies. During microwave radiation, local charges of dipolar materials tend to move. Dipole lags behind the electromagnetic field, energy dissipation occurs in the material, leading to heat generation. In wastewater and sludge water, moisture can be considered as the main component. Dipole movement is influenced by the strength of the hydrogen bonded matrix of materials. In the case of the free water content, the movement has been found to be in the gigahertz frequency range. Dipolar movement is the dominant mechanism for bound water in the megahertz frequency range and for ice at kilohertz frequencies (Brodie *et al.* 2014).

In heterogeneous and complex systems, the dielectric properties are determined by the molecular structure of compounds and the physico-chemical properties as well. The continuous realignment of polarized dipoles to electromagnetic fields of varying polarities can result in collisions of dipolar molecules and weakening of hydrogen bonds within water molecules (Afolabi & Sohail 2017). The behaviour of molecules in the irradiated materials is characterized by the dielectric parameters, such as the complex dielectric permittivity or its real and imaginary components, namely the dielectric constant and dielectric loss factor. The dielectric loss factor indicates the transmission loss (power dissipation) in the material, that is, the conversion of radiated microwave energy to heat. The dielectric constant influences the heat generation capacity properties of materials, and the ability of the dipolar molecules for polarization at a given frequency, respectively (Jha *et al.* 2011).

Because the dielectric parameters are linked to the molecular structure of materials, changes in physiochemical structure can be detected by dielectric measurements. On the other hand, the change in the dielectric parameters has an effect on the heating efficiency of microwaves, as well. A structural change in the processed materials causes a retroaction toward the efficacy and economy of microwave heating processes. It has been verified elsewhere that microwave treatments are suitable for preliminary hydrolysis of macromolecules before AD of sludge, which leads to an increase in biogas production (Zheng *et al.* 2007; Eskicioglu *et al.* 2009). During sludge treatment carried out by microwave radiation alone, or combining with surfactant dosage, carbohydrates are hydrolysed into sugars, proteins into amino acids, and lipids into short-chain fatty acids (Xiao *et al.* 2017). With decreasing molecular weight and an increasing polar characteristic of hydrolysed monomers, the polarization ability of the processed materials is enhanced, and therefore these changes can be characterized and quantified by the change of the dielectric parameters.

Realizing the possibilities of short time responding dielectric measurements, the applicability of method was evaluated for wastewater and sludge from different origins. Our present research aimed to determine the dielectric behaviour of municipal wastewater sludge as a function of temperature and total solids (TS) content. Beside this, the follow up of microwave intensified disintegration process for meat industry sludge and purification process of municipal wastewater was also investigated.

MATERIALS AND METHODS

Samples

The municipal wastewater samples were originated from a local wastewater treatment plant (WWTP). In WWTP after bar screening, for the biological treatment an aerated sequencing batch reactor technology was applied. Purified wastewater was sampled after the biological stage of the WWTP. Raw (untreated) food industry wastewater sludge came from sedimentation process in which sample originated form equalization tank of the wastewater line of a meat-processing plant. The main characteristics of the samples are summarized in Table 1.

Analytical methods

The TS content of samples was determined by the drying cabinet method using a temperature of 105 °C to achieve a constant final weight. The organic matter content was measured by the COD fractionation method. SCOD was measured using the colorimetric method (APHA 5220D 2005) from supernatant of samples after centrifugation (10,000 rpm for 15 min) and microfiltration (0.45 μm). The TCOD was measured from the whole matrix after dilution. The disintegration degree (DD) was given as the percentage change in the ratio of SCOD to TCOD.

Experimental setup

Microwave (MW) treatments were carried out in a tailor-made microwave unit equipped with a variable power magnetron (from 100 to 700 W) operating at a frequency of 2,450 MHz. In continuous-flow operation mode, the volumetric flow rate of samples was varied by the revolutions per minute of the peristaltic pump in the range of 10–45 L/h.

In the case where municipal sludge pre-treatment was not used, effects of TS and sample temperature on dielectric constant were examined. MW treatment was applied for meat processing wastewater in order to investigate the effect of MW radiation on disintegration degree and dielectric constant, as well. Samples originated from the purification of municipal wastewater were not subjected to further treatment. Dielectric behaviour was measured as a function of temperature and volumetric flow rate, respectively.

Dielectric constant measurement

The dielectric constant (ε') was determined in a tailor-made dielectrometer equipped with a dual-channel NRVD power

Table 1 | Main characteristics of wastewater samples

Parameter	Unit	Municipal wastewater: raw	Municipal wastewater: purified	Meat industry wastewater sludge
pH	[SU]	7.3 ± 0.3	7.1 ± 0.2	5.9 ± 0.2
TCOD	[mgO_2/L]	986 ± 34	102 ± 2.1	1,697 ± 93
SCOD	[mgO_2/L]	115 ± 22	68 ± 4.1	323 ± 11.8

meter (Rohde & Schwarz, Germany). The magnetron of the dielectrometer operates at a frequency of 2,450 MHz. From the reflection coefficient (Γ) and phase shift (φ) ε' can be calculated by Equations (1) and (2).

$$\delta = \text{arc tg}\left(\frac{|\Gamma|\ \sin\varphi}{1-|\Gamma|\cos\varphi}\right) - \text{arc tg}\left(\frac{|\Gamma|\ \sin\varphi}{1+|\Gamma|\cos\varphi}\right) \tag{1}$$

$$\varepsilon' = \frac{1}{\sqrt{1+\text{tg}\,\delta^2}}\left(\frac{1+|\Gamma|^2+2|\Gamma|\cos\varphi}{1+|\Gamma|^2-2|\Gamma|\cos\varphi}\right) \tag{2}$$

A central composite face-centred (CCF) experimental design and response surface methodology were applied to investigate the effect of variables on the target parameters using Statistica software.

RESULTS AND DISCUSSION

Disintegration of municipal sludge

In the first series of experiments, the effect of TS and temperature on DD and the dielectric constant were investigated for municipal wastewater sludge. The TS content was adjusted by mixing wastewater and sludge originated from municipal wastewater produced in a sedimentation process at a WWTP. The response variables, such as DD and the dielectric constant, were assessed as a function of two first-order effects (x_1: temperature; x_2: TS content), the interaction effect (x_1x_2), and second-order effects, as well. Tables 2 and 3 summarize the results of analysis of variance (ANOVA) for DD and the dielectric constant, respectively.

The results of ANOVA indicated that DD was mainly influenced by the temperature (Table 2), but temperature and TS content had similar strong effects on the dielectric constant (Table 3). The linear term for both temperature and TS content can be considered significant, but quadratic terms of TS content (x_2^2) for DD and the quadratic term of temperature (x_1^2) were not significant at the 95% level. Regression equations for DD and the dielectric constant (DC) with remained significant terms are expressed by Equations (3) and (4), respectively.

Table 2 | Analysis of variance for DD

Source	SS	Df	MS	F-value	*p*-value
x_1	1,536.0	1	1,536.0	781.95	0.00001
x_1^2	168.52	1	168.52	85.79	0.00003
x_2	57.04	1	57.04	29.04	0.00031
x_2^2	0.0003	1	0.003	0.0013	0.97191
$x_1\,x_2$	13.69	1	13.69	9.69	0.02474
Error	19.64	10	1.964		
R^2	0.984				

SS, sum of squares; Df, degrees of freedom; MS, mean square.

Table 3 | Analysis of variance for dielectric constant

Source	SS	Df	MS	F-value	*p*-value
x_1	366.79	1	366.79	29.18	0.00361
x_1^2	1.19	1	1.29	0.182	0.67894
x_2	330.38	1	330.38	55.14	0.00023
x_2^2	170.04	1	170.04	26.01	0.00045
$x_1\,x_2$	156.25	1	156.25	23.91	0.00064
Error	65.38	10	6.54		
R^2	0.917				

SS, sum of squares; Df, degrees of freedom; MS, mean square.

$$Y_{DD} = 0.4554 + 0.962\,x_1 + 0.1236\,x_2 + 0.0115\,x_1x_2 - 0.014\,x_1^2 \tag{3}$$

$$Y_{DC} = 56.77 - 0.469\,x_1 + 6.868\,x_2 + 0.039\,x_1x_2 - 0.359\,x_2^2 \tag{4}$$

Generally, the increment of temperature causes an enhancement of DD, but over a certain value the higher temperature does not result in a further enhancement of disintegration (Figure 1(a)). Due to the thermal effects, the high molecular-weight macromolecular components are hydrolysed, and sludge floc and cell walls start to decompose, which increases the organic matter content in the liquid phase, as indicated by the higher DD (Yang *et al.* 2013). Structural change and hydrolysis of macromolecules lead to an increment of the concentration of lower weight and polar components. Due to the higher mobility and better polarization ability of smaller and polar components, the dielectric constant of MW treated wastewater and sludge tends to increase, as well. Therefore, the disintegration mechanisms can be detected well by dielectric measurement.

Considering the effects of variable TS content, different behaviours of the dielectric constant can be found as a function of temperature. As the temperature increased, the dielectric constant showed a decreasing tendency for samples with a lower TS content, but an increasing tendency was detected for wastewater sludge with a higher solid content (Figure 1(b)). A possible explanation for the adverse effect

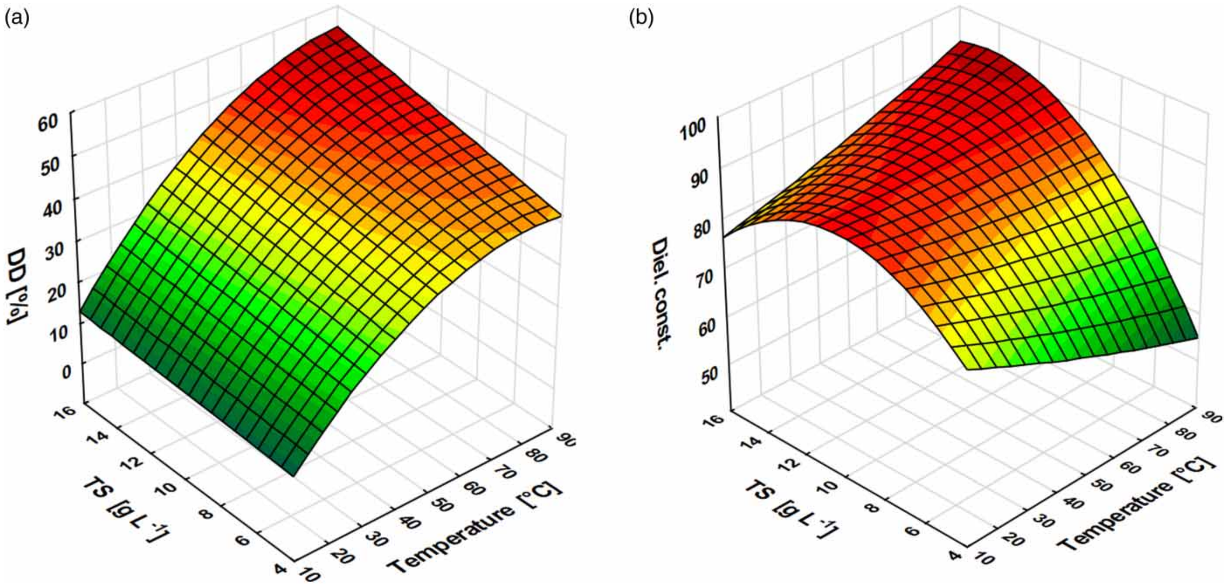

Figure 1 | Disintegration degree (a) and dielectric constant (b) as a function of TS and temperature.

of temperature on the dielectric constant is the different composition of samples, which determines the dielectric behaviour. In wastewater with a lower TS content, the ratio of free water to solids is higher; and, therefore, the dielectric behaviour is determined mainly by the relaxation mechanisms of water molecules. At the given frequency (2,450 MHz), the dielectric constant of water decreased with increasing temperature. If the TS content increases, the dielectric behaviour starts to be more influenced by the dipolar relaxation shift of smaller organic molecules and ionic conduction of ionic compounds. Due to thermal effects, the hydrolysis of macromolecules and the release of intracellular substances occur (Bohdziewicz *et al.* 2011). These mechanisms cause an increase in the ratio of free to bound water content and enhance the mobility and polarization ability of compounds in the electromagnetic field, leading to an increase in the dielectric constant (Brodie *et al.* 2014).

Continuously flow microwave treatments of meat-processing sludge

In order to investigate the relationship between the change of the disintegration degree and dielectric parameters, continuous-flow microwave treatments were carried out for primary sludge with a high organic matter content that originated from a meat-processing plant.

As the results show, applying continuous-flow microwave treatment decreased volumetric flow rate (results in higher residence time in the microwave reactor) and increased microwave power leading to an increased disintegration degree (Figure 2(a)). The Pareto chart indicates that the flow rate and microwave power also had a significant effect on the DD, but the quadratic terms of variables and interaction of two variables were not significant at the 95% level (Figure 2(b)).

The dielectric constant showed a main tendency similar to that of DD. Variation of the dielectric constant indicates a physicochemical change of treated sludge (Figure 3(a)).

The results of response surface modelling and ANOVA show that the microwave power, flow rate, and their interaction also had a significant effect on the dielectric constant of meat-processing effluents (Figure 3(b)), and the behaviour of the dielectric constant can be described by a quadratic regression model. The regression model for the DD and dielectric constant is given by Equations (5) and (6), where x_1 and x_2 coded the variables for the microwave power level (MWP-W) and the volumetric flow rate (Flow-L/h), respectively.

$$Y_{DD} = 43.84 + 0.053\,x_1 - 0.913\,x_2 \quad (5)$$

$$Y_{DC} = 76.12 + 0.03\,x_1 + 0.106\,x_2 + 0.002\,x_1x_2 - 0.0002\,x_1^2 - 0.007\,x_2^2 \quad (6)$$

Dielectric constant measurement for wastewater purification process

In order to investigate the suitability of dielectric measurement for detection of the changes in wastewater

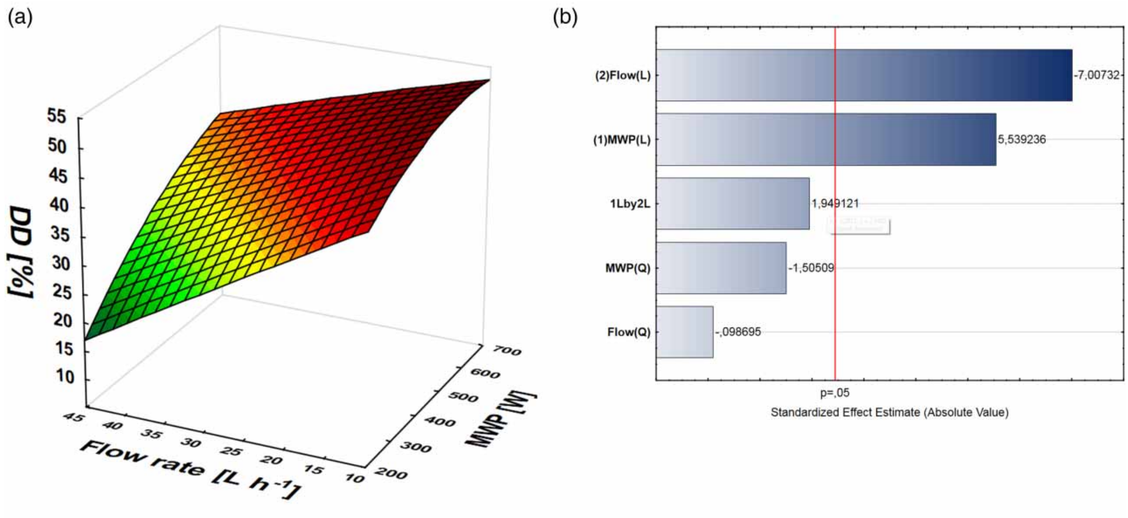

Figure 2 | Change of disintegration degree of meat-processing sludge during MW treatments (a) and Pareto chart of effects (b). (The dielectric constant was measured after cooling to a temperature of 25 °C for all samples.)

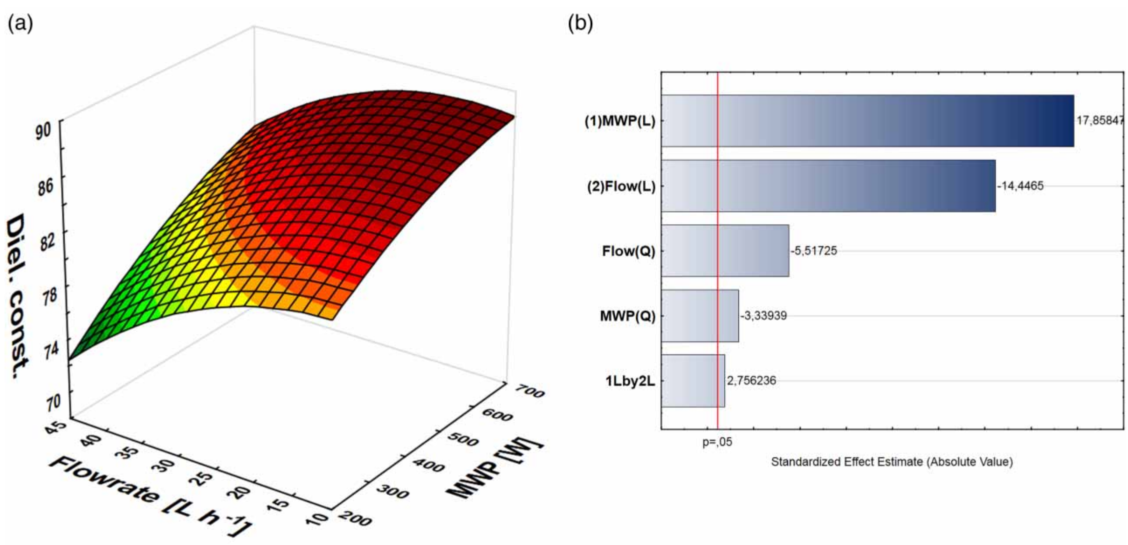

Figure 3 | Dielectric constant of MW irradiated meat-processing wastewater sludge (a) and Pareto chart of effects (b). (The dielectric constant was measured after cooling to a temperature of 25 °C for all samples.)

quality parameters, the method was tested using municipal wastewater, as well. Municipal wastewater samples were obtained from different stages of the purification process at a local WWTP. Figure 4 shows the temperature and flow rate dependency of the dielectric constant for raw wastewater (a) and purified wastewater (b). The dielectric constant measurements were carried out in continuous flow mode to investigate the effect of flow rate on the dielectric parameters and to test the sensitivity of the measurement method to sample homogeneity.

Despite of the different origin and characteristics of municipal wastewater from meat industry effluent, similar to that obtainable for meat-processing wastewater sludge, the flow rate can influence the measurable dielectric constant depending on the organic matter content of municipal wastewater. The results indicated that both the temperature and the flow rate had significant effects on the value of the dielectric constant, but the quadratic term of the flow rate for raw (untreated) and purified wastewater was not significant at the 95% level. The change of the dielectric constant versus temperature (x_1) and volumetric flow rate (x_2) was

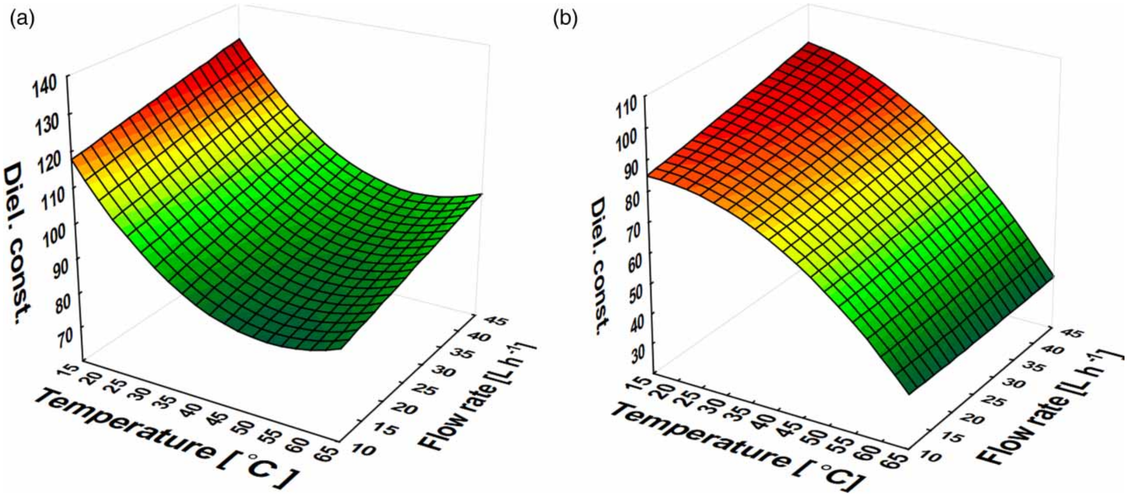

Figure 4 | Dielectric constant of raw (a) and purified (b) municipal wastewater.

different for raw and purified municipal wastewater, as given by Equations (7) and (8), respectively. The response surfaces of the dielectric constant for municipal wastewater are given in Figure 4.

$$Y_{raw} = 149.4 - 2.719\,x_1 + 0.335\,x_2 + 0.026\,x_1^2 \quad (7)$$

$$Y_{purif} = 75.74 + 0.667\,x_1 + 0.044\,x_2 - 0.007\,x_1x_2 - 0.019\,x_1^2 \quad (8)$$

Because of the high particulate matter content of raw wastewater, especially in a higher temperature range, the flow rate has stronger effect on the dielectric constant. Due to the higher temperature, structural changes, such as hydrolysis of macromolecules, occur in raw wastewater; furthermore, the flow of wastewater assists to achieve higher homogeneity for MW radiation, which enhances the efficiency disintegration of particulates. These effects lead to a higher value of the dielectric constant.

The increment of the dielectric constant above a critical temperature range (50–55 °C) was due to thermal hydrolysis of the macromolecular components and release of intracellular substances into the intercellular space. These effects can be expressed more if the organic matter content of wastewater is higher (see Figure 1(b)). In the case of purified water with a lower organic matter content, the effect of flow rate on the dielectric constant at a given temperature was smaller and the temperature dependency had a tendency similar to the one known for 'pure' water (Holtze *et al.* 2006). In summary, the measurement of the dielectric constant is a suitable method to detect changes in the organic matter removal efficiency during the use of conventional wastewater purification technology.

CONCLUSION

Our research was focused on the investigation of the applicability of dielectric constant measurement in sludge disintegration and in the wastewater purification process. Furthermore, dielectric measurements were implemented in continuous-flow microwave treatments of meat industry sludge. Results support the finding that dielectric measurements are suitable to detect and quantify the physicochemical changes of sludge structure and organic matter removal during the wastewater purification process. Although the main component of wastewater is the water, the dielectric behaviour of raw wastewater as a function of temperature or the volumetric flow rate was different from that obtained for purified water. Furthermore, our results indicated that thermal treatments have an effect on the disintegration of organic matter particulates in municipal and food industry wastewater, and these changes have an effect on the dielectric constant as well. Our preliminary results suggest that the method is suitable for developing real time and in-line dielectric measurement methods to detect the disintegration and removal efficiency of the wastewater purification process.

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