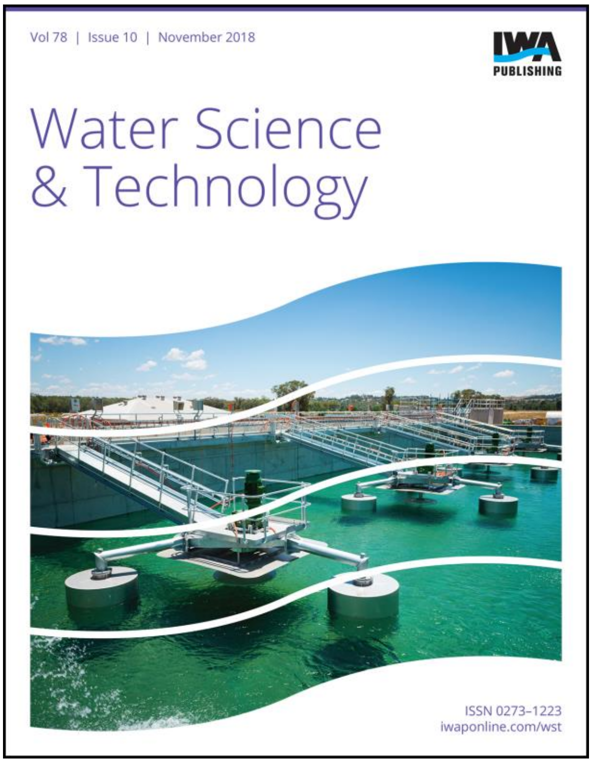

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Detection of the efficiency of microwave–oxidation process for meat industry wastewater by dielectric measurement

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ABSTRACT

Our experimental work focused on the applicability of a quite novel process for wastewater treatment, i.e. a microwave (MW) irradiation-enhanced Fenton-like method. The aim of our research was to detect and evaluate the efficiency of this oxidation process, during the treatment of meat industry wastewater containing a high concentration of organic material. The efficiency was defined by the measurement of the change in COD (chemical oxygen demand, with an initial COD value of 1,568 mg L^{-1}), and with the determination of dielectric parameters during the process. It can be summarized that MW irradiation could assist in a Fenton-like oxidation process to achieve higher organic matter removal. Furthermore, our experimental results and statistical analysis show that there can be found a correlation between the effects of applied MW energy and the dosage of H_2O_2/$FeSO_4$. If the intensity of MW irradiation and the amount of $FeSO_4$ were set higher, the decrease of COD and the increase of tanδ (the dielectric loss tangent) were definitely more significant. With the application of 60 kJ MWE and a 0.14 mgFe^{2+}/mgCOD dosage, the COD removal efficiency was more than 40%, and the increment of tanδ was nearly threefold. Considering the effects of MW-specific process parameters, it can be concluded that the power intensity of MW–oxidation treatment has a significant effect on COD decrease, if the irradiated MW energy was set at lower (30–45 kJ) levels.

Key words | dielectric measurement, microwave, oxidation, wastewater

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INTRODUCTION

Meat industry wastewater contains organic matters and inorganic compounds originating from the meat processing, flushing and cleaning procedures, and from the local wastewater pre-treatment methods. Depending on the food processing technology and product, the amount and the characteristics of the wastewater can vary and are difficult to predict. Therefore, a proper wastewater treatment process is needed before discharging the wastewater into the sewer system or to a natural reservoir. It was shown that specific oxidation processes are suitable to decrease the concentration of organic pollutants. Among these, the Fenton-like reaction, or Fenton's oxidation, seems to be one of the most efficient and promising ways regarding its low expenditure and the ease of operation. Fenton's reagent is a solution of H_2O_2 with ferrous ion as a catalyst, which is used to oxidize organic compounds or other contaminants (Wang 2008). The main advantage of the classical Fenton reaction and photo-Fenton method is the applicability for water and wastewater treatment at ambient temperature and atmospheric pressure, respectively. The equation of the reaction is suggested as follows (Turney 1995):

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + \cdot OH + OH^- \quad (1)$$

When comparing the efficiency of the Fenton process to ozonization, it can be concluded that hydrogen radicals produced in the Fenton reaction can degrade both the hydrophobic and hydrophilic dissolved organic matters, but ozone can oxidize mainly the hydrophobic components (Jung *et al.* 2016). However, it can be noted that the main drawback of the application of the Fenton reaction for wastewater treatment in practice is the need for long

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reaction time. Among the available methods to increase the efficiency and shorten the time demand of a given process, microwave (MW) irradiation has been more and more widely used in many fields of technology and engineering. Several studies have shown that MW irradiation can be effectively used in various environmental applications, including extraction processes (Prevot *et al.* 2001), remediation of hazardous and radioactive wastes (Wicks & Schulz 1999) and chemical catalysis (Zhang *et al.* 2003). There are several studies related to the effectivity of MW pre-treatment for enhanced disintegration and biodegradability of sludge (Ahn *et al.* 2009; Jang & Ahn 2013; Yang *et al.* 2013).

Microwave radiation at the frequencies commonly used in industry and laboratory cannot carry enough energy to break the primary chemical bonds (Kappe 2004; Damm *et al.* 2012), but it can be considered as a promising pre-treatment method in biomaterial processing. For wastewater treatment, even standalone MW irradiation can be effectively used (Lin *et al.* 2009), but recent studies show that a quite effective and promising method is to combine MW with other processes and/or materials, such as oxidants and catalysts (Remya & Lin 2011), or to couple it with advanced oxidation processes, like photocatalysis (UV/TiO_2) or Fenton's reaction (Nan *et al.* 2010). Advantages of the special heating mechanism of MWs have been proved in combined microwave–oxidation processes, and it is widely used to enhance the catalytic degradation activity (Jones *et al.* 2002).

It was verified that during MW–oxidation of landfill leachate, increasing the MW power led to higher organic matter removal efficiency, but over a certain value of irradiated energy the performance of pre-treatment was worsened because of radical termination (Chou *et al.* 2013). Time requirement of methylene blue removal could be dramatically decreased by the MW-assisted Fenton process compared to traditional Fenton process (Liu *et al.* 2013). MW irradiation applied for the Fenton-like treatment of high concentration pharmaceutical wastewater improved the organic matter degradation efficiency and settling quality of sludge; it furthermore increased the biodegradability and decreased the sludge product of the process (Yang *et al.* 2009).

Penetration depth and heating efficiency of MW irradiation are determined by the dielectric parameters. The dielectric constant indicates the electric energy storage of materials, and the dielectric loss factor shows the ability of irradiated materials to convert electric energy into heat. Loss tangent is the ratio of dielectric loss factor to dielectric constant (Clark *et al.* 2000). At a given frequency the value of dielectric parameters is determined by the temperature and the physicochemical structure of the materials. Our earlier results verified that physical and chemical changes during the thermal, chemical and biological treatments can be detected by dielectric measurements (Kovács *et al.* 2018).

In our present work we focused on the organic matter removal efficiency of the microwave–oxidation process for meat industry wastewater. Dielectric parameters were also measured to investigate the applicability of dielectric measurements for detection of organic matter removal.

MATERIALS AND METHODS

Meat processing wastewater (MPW) originating from a local meat processing works has been characterized. The main characteristics (chemical oxygen demand, COD; total solids, TS; 5-day biochemical oxygen demand, BOD_5) of MPW are summarized in Table 1.

TS content was determined by drying to constant weight at 105 °C. The COD was measured using the colorimetric method (APHA 5220D 2005). BOD_5 was determined by respirometric method (Lovibond Oxidirect, Germany) at a controlled temperature of 20 °C for a 5-day period. Total organic carbon (TOC) and total nitrogen (TN) contents were determined by a Torch (Teledyne Tekmar, USA) combustion (HTC) type analyzer equipped with pressurized NDIR detector.

Microwave pre-treatments were carried out in a Labotron 500 professional equipment operated at a frequency of 2,450 MHz with power levels of 250 W and 500 W, respectively, in continuous irradiation operation mode. For the oxidation treatment a mixture of $FeSO_4$ (88%, VWR, Hungary) and H_2O_2 (30%, VWR, Hungary) was used; the pH adjustment to 3.0 and the dosage were applied before MW irradiation. Energy intensity of MW radiation (MWE) was calculated as the product of MW power (W) and time of irradiation (s) (Table 2).

Table 1 | Main characteristics of MPW

Parameter	Unit	Value
pH	[–]	6.8 ± 0.2
TS	[wt%]	2.3 ± 0.15
COD	[mgO_2/L]	1,568 ± 38
BOD_5	[mgO_2/L]	407 ± 53
TOC	[mg/L]	183 ± 17
TN	[mg/L]	95 ± 11

Table 2 | Irradiation time as a function of MWE and microwave power

MW power [W]	MWE [kJ] 30	45	60	75
250	120 s	180 s	240 s	300 s
500	60 s	90 s	120 s	150 s

At each experimental run 100 mL of sample was used. Every wastewater sample was cooled down to a temperature of 25 °C immediately after MW irradiation by a circulating thermostatic bath, and the sampling for COD and dielectric measurement was done after a 120-minute holding time. Microwave power level was controlled at two levels (250 W and 500 W) during the irradiation. Because of the characteristics of the equipment, the temperature of the irradiated samples can be measured after the MW irradiation. Table 3 summarizes the final temperature achieved in the MW treatment procedure.

For dielectric measurement a DAK-3.5 (SPEAG, Switzerland) dielectric probe connected to a ZVL3 (Rohde & Schwarz, Germany) type vector network analyzer was used in a frequency range of 200 MHz to 2,400 MHz. In dielectric measurements a fixed temperature of 25 °C was set; samples were thermostated in a water bath. To minimize the effect of absorption of MWs passing through the wall of sample containers, cylindrical PTFE (polytetrafluoroethylene) bottles were used. Five genuine sample replicates and 10 measurement replicates for each individual sample were used during loss tangent (tanδ) measurement.

To determine the effects of MWE and dosage on COD removal and the change of tanδ, and to investigate the correlation between MWE and dosage in the process, non-factorial response surface methodology with face centered star points was used. In the design of experiments three genuine replicates were applied. The low and high level of MWE, and of dosage, were defined as 30 kJ and 75 kJ, and 0.02 and 0.22 mgFe^{2+}/mgCOD, respectively. COD was measured after dilution using the colorimetric method (APHA 2005).

Table 3 | Final temperature achieved after MW irradiation

MW power [W]	MWE [kJ] 30	45	60	75
250	51 ± 3.2 °C	65 ± 4.1 °C	81 ± 2.2 °C	89 ± 3.4 °C
500	48 ± 2.4 °C	63 ± 1.8 °C	79 ± 3.5 °C	92 ± 1.5 °C

The analysis of variance (ANOVA) was carried out in Statistica 13 (Dell) with a significance level of 0.05. Standardized effects of variances were represented by Pareto charts to show and compare the strength of the effects of MWE, dosage and their interaction.

RESULTS AND DISCUSSION

In the first series of our experiments the effect of MW irradiation on the efficiency of the Fenton process was investigated. Our results verified that MW irradiation assisted in the oxidation treatment of MPW. Application of MW radiation alone did not have significant effect on organic matter removal. It can be concluded that, using a fixed Fe^{2+}/H_2O_2 ratio, the increase of the irradiated MW energy led to higher COD removal efficiency. Effect of MW power level on COD depended on the intensity of irradiated microwave energy. By increasing the irradiated energy, the effect of enhanced MW power level on COD decrease has been weakened (Figure 1(a)).

Intensification effect of MW irradiation on the oxidation of organic matters depended on the Fe^{2+}/H_2O_2. The change of microwave energy intensity has stronger effect at lower

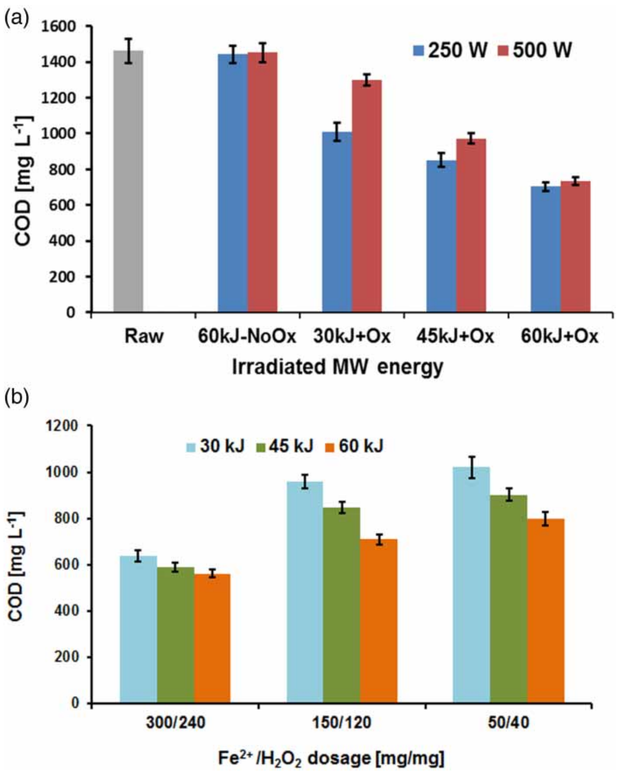

Figure 1 | COD change as function of MW energy (a) and oxidant dosage (b). (a: Fe^{2+}/H_2O_2 dosage fixed at 150/120; MW power of 500 W).

Fe^{2+}/H_2O_2 ratio, than that obtained for higher concentration of Fe^{2+}/H_2O_2 (Figure 1(b)). It can be concluded that, with the application of MW irradiation during the oxidation process, the need for Fenton reagents can be reduced. Membrane separation processes are able to reduce the organic pollutants of food industry wastewater, as well. Microfiltration and ultrafiltration as low-cost membrane separation processes have a COD removal efficiency of 80–90% from dairy wastewater streams (Bennani *et al.* 2015; Kumar *et al.* 2016). Many times in the Hungarian industrial practice the achievable final COD is determined by the threshold limit value for entering the food industry wastewater into sewer system connected to the local municipal wastewater treatment plant. In this respect the high organic matter removal efficiency is just desired when purified wastewater is wanted to be entered to a natural reservoir. It can be noted that, besides the many advantages of membrane filtration, rejected pollutants are concentrated into the sludge phase; therefore an efficient sludge treatment step needs to be applied in the wastewater treatment system. Our results verified that the MW-assisted oxidation process enables the organic matter concentration to be reduced to below the sewer threshold limit (1,000 mg L^{-1} in Hungary) with short process time demand.

Application of the combined biological and chemical treatment process has evidently good organic matter removal efficiency. By the combination of the activated sludge process with filtration and ozonation for the purification of slaughterhouse wastewater, more than 90% reduction of COD and BOD can be obtained, respectively (Alfonso-Muniuzguren *et al.* 2018). Notwithstanding the high efficiency of the laboratory-scale combined process it can be concluded that, mainly due to the low rate of biological degradation, the overall capacity of the technology decreases or the time requirement increases. The purpose of the combined MW–Fenton treatments was the rapid and efficient reduction of organic matter in wastewater. In the next series of experiments the effect of MW energy intensity (in kJ unit) and the specific dosage (mgFe_{2+}/mgCOD) was investigated on the COD of treated wastewater. Our experimental results show that the MW-enhanced Fenton-like process is suitable for reducing the content of organic materials in wastewater. Increasing the MWE by applying longer irradiation time resulted in lower final COD, especially if Fe^{2+} dosage was kept at lower values (Figure 2(a)).

Leifeld *et al.* (2018) investigated the COD removal efficiency of classical Fenton and photo-Fenton processes in a cassava processing wastewater matrix varying the H_2O_2 dosage. It was concluded that a hydrogen peroxide concentration of

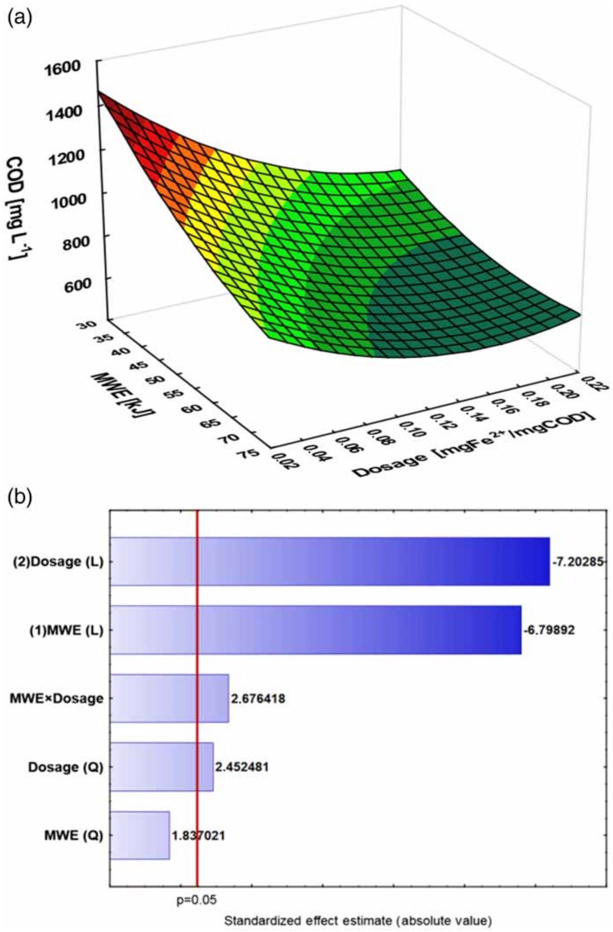

Figure 2 | Response surface (a) for COD and Pareto chart (b) for effects of variables. L – linear term; Q – quadratic term.

5,000 mg L^{-1}, which corresponded to a specific dosage of 1.6 mg $H_2O_{2/mg}$COD, resulted in 50% COD reduction after 120-minute treatment at ambient temperature. COD removal efficiency of 70% can be achieved by the photo-Fenton method. The exact ratio of Fe to H_2O_2 is not known, because the residual iron from the preliminary coagulation stage was used as catalyst. In our experiment lower Fe^{2+} and H_2O_2 dosage was applied. With MWE of 30 kJ, COD removal of only 6% (from the initial COD of 1,568 mg L^{-1} to 1,479 mg L^{-1}) could be achieved when Fe^{2+} dosage ratio of 0.02 was applied (Figure 2(a)), but if the intensity of MW radiation was set higher than 75 kJ, COD removal increased further by nearly 40% (to COD of 940 mg L^{-1}). Using 0.14 or higher Fe^{2+}/COD dosage, the maximum COD removal (higher than COD of 690 mg L^{-1}) was experienced if intensity of MW treatment was set higher than 60 kJ.

The results of ANOVA indicate that both dosage and MWE affect significantly the reduction of COD at a confidence level of 0.95, and when compared to each other, the effect of dosage is stronger (Figure 2(b)). As the response surface plot shows, at low range of MWE the decrease of COD was more influenced by the Fe^{2+} addition than it was at higher intensity MW treatment. Thus, application of MW radiation during the Fenton-like process gives the opportunity to reduce the Fe^{2+} dosage.

The effects of MWE and dosage on COD can be given by a regression equation that contains the quadratic, linear and interaction terms, respectively. Considering the *p*-values, the quadratic term of dosage was significant at a confidence level of 0.95 (Figure 2(b)), but the quadratic term of MWE was not significant (Table 4). An interaction effect of MWE with dosage was found; therefore it can be stated that the MW irradiation increases the efficiency of the Fenton process. After removing the non-significant terms the derived regression model was given by Equation (2).

$$y = 2482.1 - 27.98x_1 - 7889.8x_2 + 22031.6x_2^2 + 54.31x_1x_2 \quad (2)$$

where x_1 and x_2 coded the variables of MWE (kJ) and dosage ($mgFe^{2+}/mgCOD$), respectively.

The term of R-squared indicates the explanatory power of the regression model, i.e. shows the ratio of explained variation to the total variation. As the value of R^2 – 0.8802 – indicates, the developed model can be considered adequate. Intensification and acceleration effects of MW irradiation on the heterogeneous Fenton reaction between iron oxide and hydrogen peroxide, and precipitation effects of iron-based colloids were verified in concentrated landfill leachates, as well (Chen *et al.* 2018). Similar to our observations, this former study concluded that the application of MW irradiation as sole treatment did not result in remarkable COD reduction, but in presence of H_2O_2 MW irradiation promoted the production of hydroxyl radicals.

Table 4 | Results of ANOVA for COD

Factor	SS	DF	MS	*F*-value	*p*-value
MWE (L)	207,477.5	1	207,477.5	46.225	0.00047
MWE (Q)	15,146.7	1	15,146.7	3.374	0.09607
Dosage (L)	232,862.2	1	232,862.2	51.881	0.00003
Dosage (Q)	26,996.2	1	26,996.1	6.014	0.03411
MWE by dosage	32,151.3	1	32,151.3	7.163	0.02323
Error	44,883.8	10	4,488.4		
R^2	0.8802				

SS: sum of squares; DF: degrees of freedom; MS: mean square (L – linear term; Q – quadratic term).

Our earlier experiences verified that dielectric parameters are suitable to indicate the change of disintegration degree during MW sludge treatment (Lemmer *et al.* 2017). Dielectric behaviour of materials is influenced by the frequency, temperature and the physico-chemical structure of the materials (Jha *et al.* 2011). When the particulate organic matter of wastewater transforms into soluble form during the chemical and/or thermal treatment, the dielectric parameters are supposed to be changed. In order to test the hypothesis, i.e. the dielectric parameters are connected to the organic removal in the wastewater purification process, the tanδ was measured for samples treated by the microwave–oxidation process.

At the first stage of experiments, during the dielectric measurements the frequency range of 200 MHz to 2,400 MHz was swept. In static measurement mode, with the temperature set to 25 °C, the higher difference between the dielectric parameters of different pre-treated samples was found at lower frequencies. Among the dielectric parameters (dielectric constant, loss factor, tanδ, reflexion coefficient etc.), tanδ was proved to be the most sensitive to quantify the change of organic matter concentration in the meat industry wastewater. Therefore, tanδ was measured at the frequency of 200 MHz after the pre-treatment of samples.

Results of dielectric measurements verified that the tanδ is suitable to detect the organic matter removal efficiency of MW–oxidant treatments. With the decrease of COD tanδ increased, and the effect of condition of treatment (MW power and energy intensity) shows similar tendencies to that obtained for COD values (Figure 3).

The results show that both MWE and dosage affect the value of tanδ, and with respect to *F*-values the effect of dosage proved stronger.

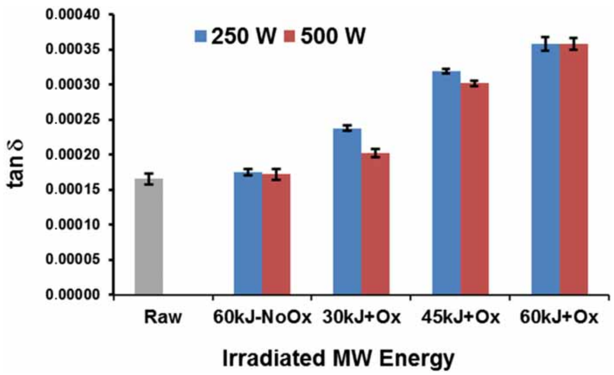

Figure 3 | Change of tanδ after MW–oxidant pre-treatments (frequency of 200 MHz, temperature of 25 °C, for oxidation treatment: Fe^{2+}/H_2O_2 fixed at 150 mg/120 mg).

Considering the tendency of COD reduction, the decrease of COD causes the increase of tanδ. It can be seen that the optimal range of process parameters to achieve the lowest COD (MWE higher than 60 kJ, and Fe^{2+} dosage higher than 0.14 mgFe^{2+}/mgCOD) agreed with the process parameter ranges for highest tanδ (Figure 4(a)).

Based on the results of analysis of variances, it can be established that the linear and quadratic terms of dosage and MWE can be considered significant, but the interaction of these two variables is not significant, at a confidence level of 0.95 (Table 5).

After removing the non-significant terms the constructed regression model for tanδ was given by Equation (3)

$$y = -0.000039 + 0.000007x_1 - 0.000000005x_1^2 + 0.002082x_2 - 0.005389x_2^2 \quad (3)$$

The main reason for the difference in dielectric parameters of raw and pre-treated samples is presumed to be the change of polarization ability of the molecules. During the MW-intensified oxidation process the organic macromolecules hydrolysed to lower molecular weight components (Dogan & Sanin 2009; Yu *et al.* 2010); moreover, the oxidation mechanisms coupled with MW irradiation assist the transformation of remaining organic matters into soluble forms. Increased solubility and decreased average molecular weight of organic components led to enhanced ability for polarization in the electromagnetic field (Brodie *et al.* 2014).

It can be noted that MPW contains organic matters in particulate form. Compared to the experiences related to MW processing of sludge, MW irradiation used as solely pre-treatment could not increase the organic matter solubility; the COD measured from soluble phase was not changed (Figure 1(a)), because the wastewater has lower COD than sludge. But MW irradiation used together with the Fenton-like treatment was theoretically suitable to decrease the integrity of organic matter particles. If ions and smaller molecular components are released, the polarizability of components enhances, which is indicated by the increasing tanδ. If oxidant is added, the thermal effect of MW irradiation can be observed by using the same dosage and by increasing MWE (Figure 4(a)). In that case the increment of tanδ originated solely from the MW effects; the effect of increased Fe^{2+} dosage on dielectric parameters can be excluded.

Figure 5 shows the tendency of the change of dielectric loss tangent (Δtanδ) and final COD of MW- and combined MW/Fenton-treated meat industry wastewater samples. Regarding the effects of treatment, the change of concentration of organic materials and the change of the dielectric parameter show a similar tendency.

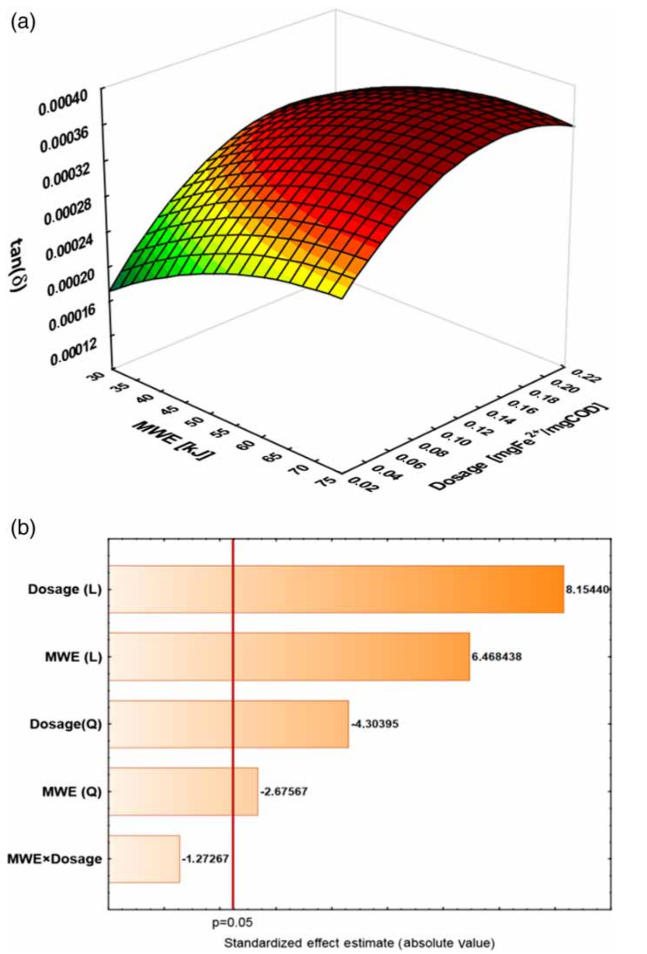

Figure 4 | Response surface (a) for loss tangent (tanδ), and Pareto chart (b) for effects of variables. L – linear term; Q – quadratic term.

Table 5 | Results of ANOVA for dielectric loss factor (tanδ)

Factor	SS	DF	MS	*F*-value	*p*-value
Dosage (L)	$1.476*10^{-8}$	1	$1.48*10^{-8}$	66.495	0.00002
Dosage (Q)	$4.112*10^{-9}$	1	$4.11*10^{-9}$	18.524	0.00155
MWE (L)	$9.289*10^{-9}$	1	$9.29*10^{-9}$	41.841	0.00007
MWE (Q)	$1.589*10^{-9}$	1	$1.59*10^{-9}$	7.159	0.02327
MWE by Dosage	$3.601*10^{-10}$	1	$3.60*10^{-10}$	1.061	0.23193
Error	$2.221*10^{-9}$	10	$2.22*10^{-9}$		
R^2	0.9107				

SS: sum of squares; DF: degrees of freedom; MS: mean square (L – linear term; Q – quadratic term).

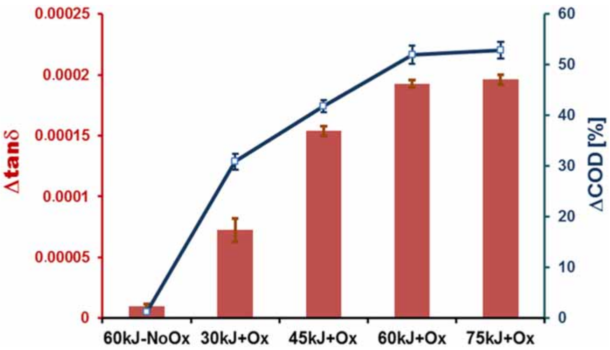

Figure 5 | Correlation between COD change and tanδ (for oxidation treatment: Fe^{2+}/H_2O_2 fixed at 150 mg/120 mg).

CONCLUSION

The effects of a MW radiation-enhanced Fenton-like process have been investigated. Our experimental results showed that organic matter removal efficiency is influenced by the irradiated MW energy and by the $H_2O_2/FeSO_4$ as well. The combined MW–oxidation treatments efficiently removed the organic content of the meat industry wastewater samples, especially if the MWE and dosage were set to >60 kJ and 0.14 mgFe^{2+}, respectively, with a COD decrease of more than 40%. The statistical analysis suggests that, for both the COD removal and the change of tanδ, the effect of dosage can be considered stronger than the effect of MWE. Significant interaction effect between MWE and COD, as variables, could only be found in the case of tanδ ($p = 0.05$).

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