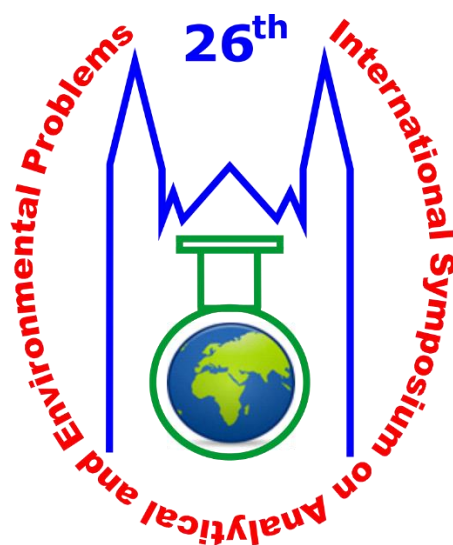




***PROCEEDINGS OF THE***

***26<sup>th</sup> International Symposium***  
***on Analytical and Environmental Problems***

***Szeged, Hungary***  
***November 23-24, 2020***



**University of Szeged**

**Edited by:**

Tünde Alapi

Róbert Berkecz

István Ilisz

**Publisher:**

University of Szeged, H-6720 Szeged, Dugonics tér 13,  
Hungary

**ISBN 978-963-306-771-0**

**2020.**

**Szeged, Hungary**

***The 26<sup>th</sup> International Symposium on Analytical and  
Environmental Problems***

**Organized by:**

SZAB Kémiai Szakbizottság Analitikai és Környezetvédelmi Munkabizottsága

**Supporting Organizations**

*Institute of Pharmaceutical Analysis, University of Szeged  
Department of Inorganic and Analytical Chemistry, University of Szeged*

**Symposium Chairman:**

*István Ilisz, DSc*

**Honorary Chairman:**

*Zoltán Galbács, PhD*

**Organizing Committee:**

*István Ilisz, DSc*

*professor of chemistry*

*University of Szeged, Institute of Pharmaceutical Analysis*

*Tünde Alapi, PhD*

*assistant professor*

*University of Szeged, Department of Inorganic and Analytical Chemistry*

*Róbert Berkecz, PhD*

*assistant professor*

*University of Szeged, Institute of Pharmaceutical Analysis*

**Scientific Committee:**

*István Ilisz, DSc*

*Tünde Alapi, PhD*

*Róbert Berkecz, PhD*

*Daniela Sojic Merkulov, PhD*

*associate professor*

*University of Novi Sad, Faculty of Sciences, Department of Chemistry, Biochemistry and  
Environmental Protection*

## DETECTION OF CHANGES IN SOLUBLE ORGANIC MATTER CONTENT IN MUNICIPAL THICKENED SLUDGE BY DIELECTRIC MEASUREMENTS

**Laura Haranghy, Zoltán Jákó, Cecília Hodúr, Sándor Beszédes**

*University of Szeged Faculty of Engineering, Department of Process Engineering, Moszkvai  
krt. 9, H-6725 Szeged, Hungary.  
e-mail: haranghylaura@gmail.com*

### Abstract

The dielectric measurement is a rapid and non-destructive method to investigate the physico-chemical change in the examined materials. Many researches observed the dielectric properties of pure water, different materials from the food industry, but there is less information about the dielectric characteristic of wastewater and sludge. Our study aims to detect correlation between dielectric constant and SCOD/TCOD parameter, to verify if different microwave irradiation treatments can enhance the solubilization of organic matter in municipal sludge. With the determination of soluble chemical oxygen demand (SCOD) we detect the effects of different microwave irradiation treatments on the sludge's organic matter solubility. Our results show dielectric measurement is a suitable method to detect the changes in the soluble organic matter content, since there is a strong linear correlation between the dielectric constant ( $\epsilon'$ ) and the SCOD/TCOD parameter, which indicates the the solubility of the organic matter.

### Introduction

Due to the urbanization, the increasing amount of consumed water quantity, even though the developing wastewater treatment plants, the amount of municipal sludge is increasing. Because of the high cost of operating and treatment processes, it should be taken into consideration to utilize the arising amount of sludge. For economic reasons the application of different pre-treatment technologies are required.

Microwave (MW) irradiation is a promising pre-treatment method in sludge treatment, as a result of its unique heating mechanism which provides a fast and selective heating ability [1]. Due to its thermal effect it can disrupt the cell wall of pathogene microorganisms, thereby enhance the disposal of sludge. The released cellular fluid contains proteins, carbohydrates and lipids originated from the bacteria presented, which increases the organic matter content of the sludge, therefore it becomes more available for the degrading microorganisms [2]. The SCOD/TCOD parameter describes the ratio between the amount of organic matter in the soluble phase and the total chemical oxygen demand of the observed sample.

Dielectric materials, like the municipal sludge can absorb the microwave interval of the electromagnetic spectrum, and its extent depends on the material properties, structure, temperature and frequency. The dielectric behaviour of a material can be described with certain dielectric properties.

Complex relative permittivity ( $\epsilon$ ) includes the characteristics that affect the reflection of electromagnetic waves from the material interface, as well the energy loss that occurs with the absorption of the electromagnetic wave [3]:

$$\epsilon = \epsilon' - j \cdot \epsilon'' \quad (1)$$

The dielectric constant ( $\epsilon'$ ) represents the electrical energy absorption capacity of the dielectrical material, the dielectric loss factor ( $\epsilon''$ ) describes the dissipation ability of the dielectric material and  $j$  is the imaginary factor.

The product of free space permittivity ( $\epsilon_0$ ) and the relative permittivity ( $\epsilon_r'$ ) gives the dielectric constant:

$$\epsilon' = \epsilon_0 \cdot \epsilon_r' \quad (2)$$

It has been proven that measuring dielectric properties is a capable way of determine structural and/or molecular changes in the raw material matrix, and such, it is a suitable method to detect the organic matter removal efficiency of wastewater treatment processes [4]. By the application of this rapid, non-destructive measurement method the efficiency of different pre-treatment processes could be estimated.

### Experimental

Municipal thickened sludge samples were being used in our experiments. The samples were pre-treated by microwave irradiation with a total energy intensity of 30, 45 and 60 kJ at two different power levels (250 and 500 W), with the corresponding the irradiation time of 1-4 minutes. For the microwave treatments a Labotron 500 laboratory scale microwave equipment was used.

For the dielectric measurements a DAK 3.5 (SPEAG) open-ended coaxial dielectric probe was being used, connected to a ZVL-3 vector network analyser (Rohde&Schwarz). The dielectric properties were measured in the frequency range of 200-2400MHz.

Based on previous studies focusing on the determination of soluble chemical oxygen demand (SCOD), we applied the organic matter fractionation method, i.e. sedimentation, centrifugation (RCF=6000 for 10 minutes) and filtration (0,45 $\mu$ m pore sized PTFE disc filter), which was followed by a colorimetric method (Hanna, COD cuvet test, after 2 hours thermodigestion at 180 °C ) to determine the exact values of SCOD

### Results and discussion

The aim of our work was to investigate the correlation between the dielectric constant and an indirect parameter, the soluble chemical oxygen demand (SCOD) which expresses the quantity of biodegradable organic matter content in the soluble phase of wastewater sludge for microorganism.

In the first series of the experiments, after the different energy intensity microwave irradiation treatments, we measured the dielectric constant ( $\epsilon'$ ) in each sample to investigate its frequency dependence:

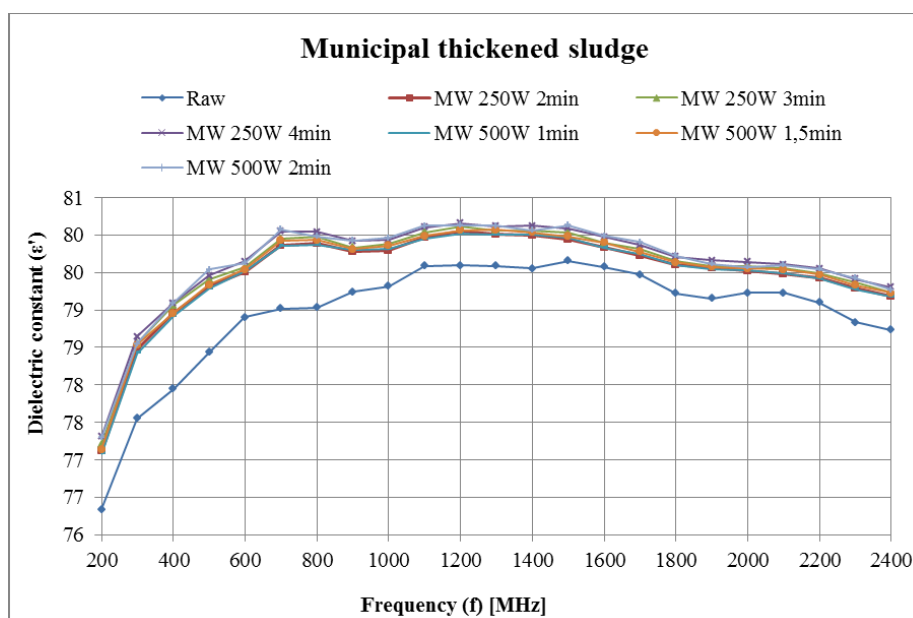


Figure 1. The frequency dependence of the dielectric constant on different MW treatments

In comparison with the raw sample, all different energy intensity MW treatments resulted in higher dielectric constants. The treated samples follow the same tendency and show similar  $\epsilon'$  values on most of the measuring frequencies. Despite similarity it can be stated that the MW

treatments cause deviation in the dielectric constant. To investigate the extensive effects of the different sludge treatment possibilities on the dielectric constant, further researches are suggested in this topic.

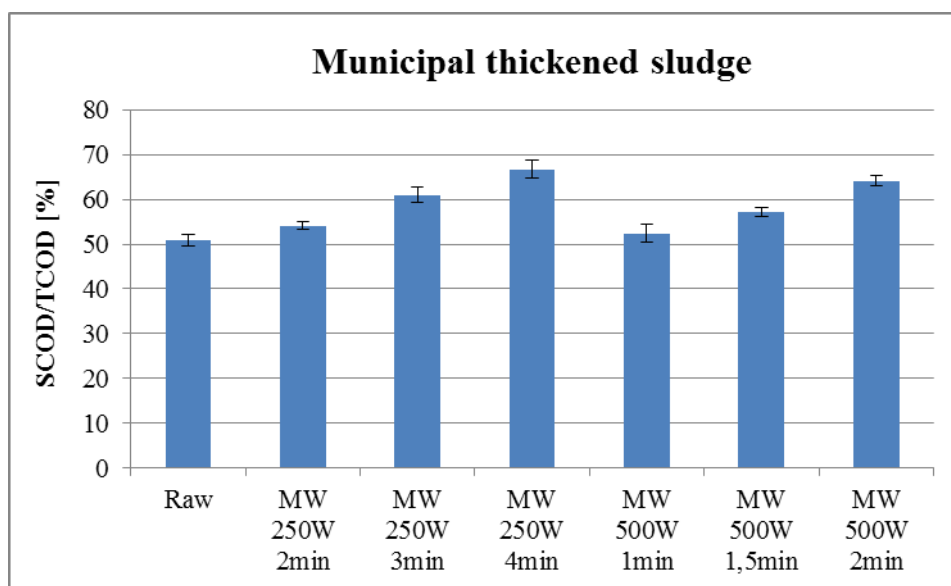


Figure 2. The effect of different MW treatments on SCOD/TCOD.

The SCOD/TCOD results show that the higher energy intensity treatments cause higher solubilization of the organic matter, the efficiency grows with the increase of the irradiated energy (Figure 2.). Treatments where lower power level (250W) was applied were more effective than those where it was set at 500W.

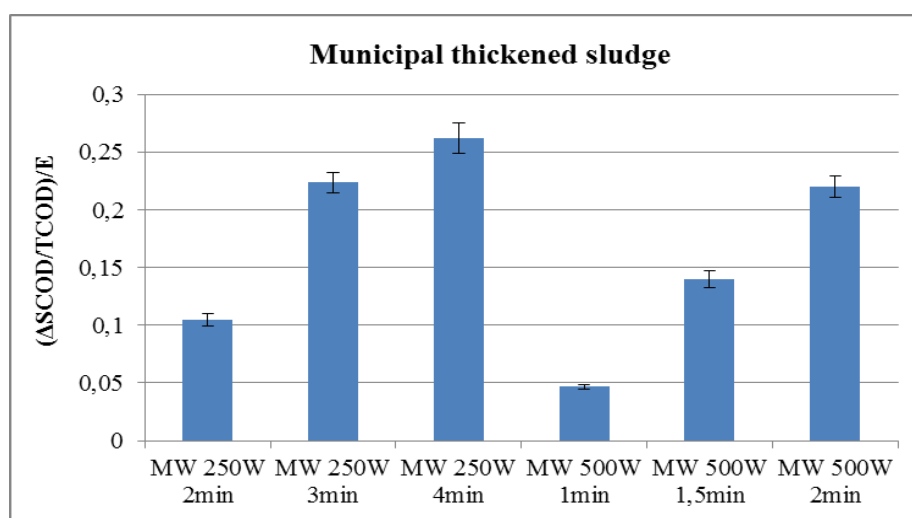


Figure 3. Increase of SCOD/TCOD compared to control sample.

Figure 3. depicts how different treatments affect the SCOD/TCOD parameter in comparison with the raw control. The values was divided by the irradiated energy to create a specific parameter to show the real tendencies of certain treatments. It shows that lower power level (250W) treatments – considering the SD range as well - were observably more effective than the ones which were carried out with the higher level of power (500W) The 250W-4 minutes MW irradiation turned out to be the most efficient operational setup, as it resulted in an increase

of more than 50% in regards of the solubility of the organic compounds, compared to the sample which was irradiated for half the time at the same power level.

After the MW irradiation treatments each sample was cooled to the temperature of 28°C to carry out the dielectric measurements. The results of dielectric measurements show that the dielectric constant at the frequency of 2400 MHz has a linear correlation with the SCOD/TCOD (Figure 4):

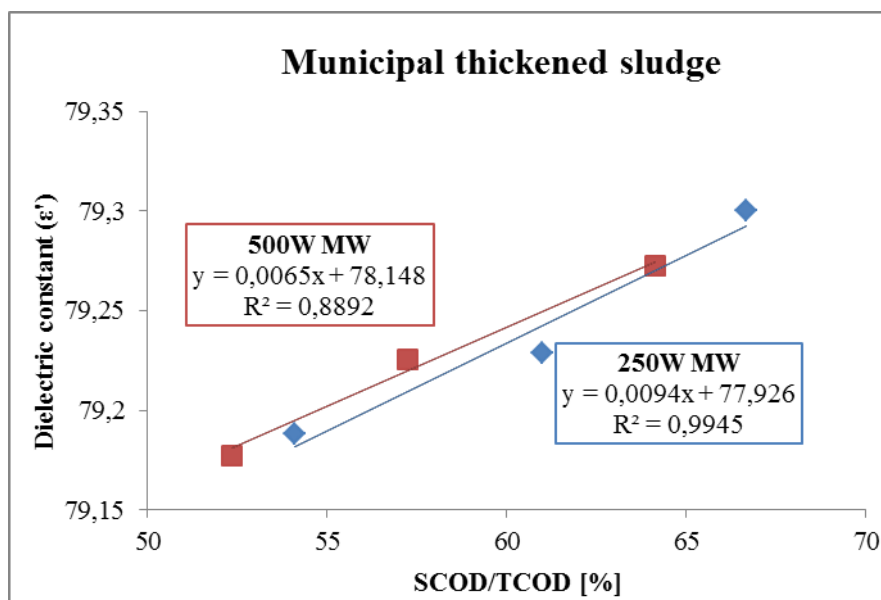


Figure 4. The correlation between SCOD/TCOD and dielectric constant ( $f = 2400$  MHz, temperature of 28°C).

The higher organic matter solubility can be traced back to the degradation effect of the microwave irradiation on the solid particales and the macromolecules of the sludge. By detecting the change of the dielectric constant, it can be stated that the irradiation caused flake desintegration in the treated sludge samples [5].

### Conclusion

In this study we compared the efficiencies of different energy and power intensity MW treatments on the solubility of organic matter in wastewater sludge samples. Based on our results, microwave irradiation is proved to be an applicable sludge pre-treatment method, since it affects the SCOD/TCOD parameter in a way which indicates that the solubility of the organic compounds of the municipal thickened sludge is enhanced in the soluble phase. In order to gain a more detailed understanding of how microwave irradiation affects the material matrix of sludge, i.e. whether a correlation between biodegradability indicators (e.g. BOD/SCOD) and the MW-enhanced solubility is presented, further researches are recommended.

Our research also focused on to investigate a possible correlation between the dielectric constant ( $\epsilon'$ ) and a solubility indicator (SCOD/TCOD). It can be stated that with the application of dielectric measurement the change in the solubility of organic matter can be detected, since the latter and the change of dielectric constant and shows a linear correlation between each other. The application of open-ended coaxial dielectric probe proved to be a promising detection method to estimate the efficiency of various sludge pre-treatment methods.

### Acknowledgements

The work was created as part of the project "Sustainable Raw Material Management Thematic Network—RING 2017" EFOP-3.6.2-16-2017-00010 within the Programme SZECHENYI2020, supported by the European Union, co-financed by the European Social Fund. The authors thank the financial support provided by NKFIH/OTKA K-115691 project.

### References

- [1] Appels L., Houtmeyers S., Degrève J., Impe J.V., Dewil R: Influence of microwave pre-treatment on sludge solubilization and pilot scale semi-continuous anaerobic digestion. *Bioresource Technology*, 2013, 128, 598-603.
- [2] Ahn, J.H., Shin, S.G., Hwang S.: Effect of microwave irradiation on the disintegration and acidogenesis of municipal secondary sludge. *Chemical Engineering Journal*, 2009, 153, 145-150.
- [3] S. Chandrasekaran, S. Ramanathan, T. Basak: Microwave Material Processing—A Review. *AIChE Journal*, 2012, 58, 2, 330-363.
- [4] Jákói Z., Hodúr C., László Z., Beszédes S.: Detection of the efficiency of microwave-oxidation process for meat industry wastewater by dielectric measurement, *Water Science & Technology*, 2018, 78, 10, 2141-2148.
- [5] Kovács V.P., Lemmer B., Keszthelyi-Szabó G., Hodúr C., Beszédes S.: Application of dielectric constant measurement in microwave sludge disintegration and wastewater purification processes, *Water Science & Technology*, 2018, 77, 9)ó, 2284-2291.