# Improvement of Active Biological Sludge Quality for Anaerobic Digestion Phase in the Wastewater Treatment Plant by Ultrasonic Pretreatment

### MIHAI STEFANESCU<sup>1\*</sup>, GHEORGHE NECHIFOR<sup>2</sup>, COSTEL BUMBAC<sup>1</sup>, IOANA IONESCU<sup>1</sup>, OLGA TIRON<sup>1</sup>

<sup>1</sup>National Research and Development Institute for Industrial Ecology ECOIND, 71-73 Drumul Podu Dambovitei Str., 060652, Bucharest, Romania

<sup>2</sup>University Politehnica of Bucharest, Faculty of Applied Chemistry and Materials Science, 1-7 Polizu Str., 011061, Bucharest, Romania

This paper emphasized the influence of the main operating parameters of ultrasonic biological sludge pretreatment onto disintegration (DD-COD) and solubilizing degree of organic matter content. The optimal ultrasonic reaction time, energy and amplitude of ultrasonic waves and pH of the sludge (non-modified pH and pH 8.5) were established taking into account their influence on the solubility level of COD, an evolution of particles size. Correlations between disintegration degree and dimensional analysis were performed. Ultrasonic reactor operating to 20 kHz ultrasound frequency was a close system having cooling water jacket. Experimental tests in different operating conditions (reaction time 5 – 60 min., ultrasound energy 2000 – 9000 kJ, pH = 6.5/8.5) demonstrated that alkaline ultrasonic treatment (pH = 8.5, initial homogeneous COD = 72600 mg  $O_z/L$ ) at medium - high ultrasound energy levels led to highest disintegration degree – DD 12% (comparing with pH = 6.5 – DD ~ 5%). Still, the process should be managed carefully because of possibility to have important variation of DD during the ultrasonic treatment.

Keywords: ultrasonic, sludge, disintegration degree, pretreatment

Ultrasonic field has many applications in various domain including water and wastewater treatment especially for removal of organic load. In these cases, the radically sonolysis mechanism has the main role for advanced degradation of specific organic pollutants (e.g. organochlorinated compounds) [1, 2]. Some studies show that sonolysis could remove some inorganic pollutant (e.g. manganese oxidation) [3]. Municipal wastewater treatment generates large quantities of sludge from primary and secondary treatment steps. International legislation has regulations referring both to the quality (dried substance content, organic, inorganic and microbial load a.s.o.) and controlled discharge of the sludge amounts. Some estimates consider that 50 - 60% of wastewater treatment plant capital cost is for sludge management. Anaerobic treatment step of the biological sludge and diminishing water content are efficient classical processes for energy generation (biogas combustion) and increasing of dried substance content. Biogas generation has three main phases: hydrolysis and acidogenesis, acetogenesis and methanogenesis (sludge stabilization). The improving of biogas generation efficiency can be done having in the anaerobic sludge degradation process more organic load in soluble phase [4].

Ultrasonic irradiation effect on biological sludge has two aspects: mechanical action leading to cell bursting membrane and transfer its content into soluble phase and sonolysis process consisting in radical oxidation of organic liquid phase making easier biological degradation of specific organic compounds [5].

The quantification of ultrasonic effect on active sludge can be done based on physical, chemical and biological indicators. Dimensional analysis, microscopic observations, turbidity, dewatering capacity, disintegration degree, solubilizing degree, organic nitrogen and ammonia content and protein amount are the most important indicators of ultrasonic degradation/solubilizing of organic content from homogeneous and soluble phases of the sludge.

Specific operational parameters of ultrasonic pretreatment (ultrasonic reactor design, ultrasonic frequency, ultrasonic energy and amplitude, irradiation time) are determinants for organic matter disintegration during sonication process [5, 6].

Biological sludge characteristics are very important for disintegration/solubilizing of organic load and for the final quality of the sludge before anaerobic digestion.

One of the most important operational parameters is *pH* value of the heterogeneous phase. Many scientifically papers show that low alkaline - alkaline *pH* domain of biological active sludge leads to higher efficiencies of organic load solubilizing and improve the yield of biogas generation in anaerobic sludge treatment phase [7].

All these experimental aspects have been taken into consideration before starting the ultrasonic pretreatment tests of biological active sludge, from a Romanian modern municipal wastewater treatment plant having tertiary treatment step and advanced conditioning step of the sludge after biogas generation phase.

# **Experimental part**

Ultrasonic pretreatment of biological active sludge was performed in the followings conditions: constant ultrasonic frequency for all experimental tests - 20 kHz, ultrasonic energy 2000 - 9500 kJ, ultrasonic wave amplitudes 20 - 80%, irradiation time 5 - 60 min., pH 6.5 - 8.5 (NaOH 10% was used for pH correction).

SONICS VIBRACELL 500 ultrasound source with ultrasonic close system reactor having cooling jacket was the system used for experimental tests and MALVERN

<sup>\*</sup> email: mihai.stefanescu@incdecoind.ro; Phone: (+40)214100377

MASTERSIZER 2000 equipment was used for dimensional

analyze of initial and treated sludge.

Initial biological sludge had pH 6. Organic load of homogeneous and centrifuged liquid phases as CODCr were 72600 mg O<sub>2</sub>/L and 9020 mg O<sub>2</sub>/L respectively. The same centrifugation parameters were for all sludge samples (8000 rpm, 15 min., 20 °C). COD determination of untreated (initial) biological sludge was performed for each experimental test category because sludge characteristics are changing in time. Disintegration (DD) and solubilizing (SD) degrees were based on these data:

$$SD = \frac{(\textit{soluble COD after US-soluble COD before US})}{\textit{homogeneous phase COD before US}} x 100$$

$$DD = \frac{(\textit{soluble COD after US-soluble COD before US})}{\textit{homog. COD before US-soluble COD before US}} \, x 100$$

Correlations between SD, DD and dimensional analysis were performed for each initial and treated sludge sample. The main resulted information is about the variation domain of particles maximal dimension  $(D_{\max})$  with maximal volume  $(V_{\max})$  content (%) from the total sludge sample volume and the specific surface  $(S_s)$  of the particles.

## **Results and discussions**

The influence of ultrasonic energy was studied for 15 minutes ultrasonic irradiation on 20% amplitude. The energy variation domain was 2000 - 9500 kJ. Organic load expressed as COD had values in the range of 96980 mg  $O_2/L$  (energy - 3000 kJ) - 12100 mg  $O_2/L$  (energy - 7000 - 9000 kJ);

In case of high ultrasonic energies 7000/9000 kJ solubilizing and disintegration degrees had maximum values of 4.8% (increase with 34% of dissolved COD) and 5.9% respectively comparing with those established for 3000 kJ energy (SD = 0.9%, DD = 1%) (fig. 1). This evolution is confirmed by the diminishing of particle size and increasing of specific surface: D = 45 - 52  $\mu$ m, S = 0.545 m²/g corresponding to 9000 kJ energy and D = 60 - 69  $\mu$ m, S = 0.237 m²/g for 3000 kJ ultrasonic energy.

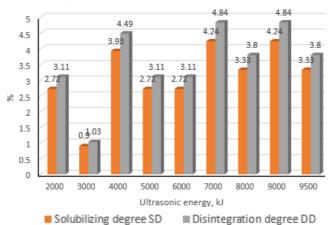


Fig. 1. Effect of ultrasonic energy to solubilizing and disintegration degrees

The influence of ultrasonic waves amplitude was established in case of experimental disintegration tests for biological sludge with 9000 kJ ultrasonic energy during 15 minutes. The amplitude waves domain was 30÷80%. The rise of amplitude led to the increases of dissolved organic load with 32 for 80% amplitude, solubilizing and disintegration maximum degrees being 4.06 and 4.63 respectively (fig. 2). Maximal specific surface 0.355 m²/g was determinate for maximal amplitude of 80%.

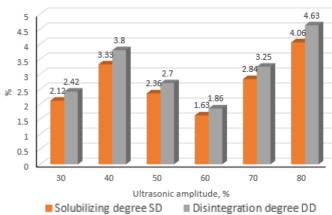


Fig. 2. Effect of ultrasonic amplitude to solubilizing and disintegration degrees

The influence of ultrasonic treatment time was studied in case of hardest treatment conditions: 9000 kJ energy and 80% amplitude. Ultrasonic irradiation time variation was in the range of 5 - 60 min. The dissolved organic load concentrations were 11440 - 13200 mg O<sub>2</sub>/L. The maximum increase of 60% was for 45 min ultrasonic degradation time. In this case the SD and DD were 6.8 and 7.7 respectively (fig. 3);

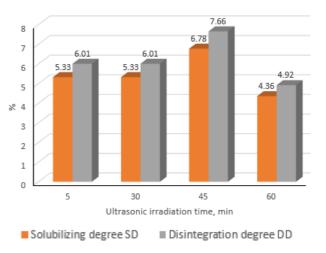


Fig. 3. Effect of ultrasonic irradiation time to solubilizing and disintegration degrees

Good results were reported even for 5 min but dimensional analysis has shown that sludge particles had bigger size in case of 5 min irradiation time being in the domain of 45 - 52  $\mu m$ . For higher disintegration time - 45 min - particle size dimensions decrease to 26 - 30  $\mu m$ , S being 1.5 times higher.

The influence of pH disintegration is very important because alkaline ultrasonic treatment is known as a more efficient process for disintegration of organic matter. Comparative tests to 6.5 and 8.4 pH values were performed.

The dissolved organic load raised together with pH increasing for 5 min of ultrasonic irradiation (9000 kJ energy, 80% amplitude) as followings: pH 6.0 - COD = 12144 mg  $O_2/I$ , pH 6.5 - COD = 21120 mg  $O_2/I$ , pH 8.5 - COD = 21200 mg  $O_2/I$ ; disintegration degree had maximum value in case of pH = 8.5, DD = 10%, specific surface of the particles being the biggest one - 0.98 m²/g. In case of 15 min ultrasonic irradiation time DD = 12% for 7000 - 8000 kJ ultrasonic energy (fig. 4).

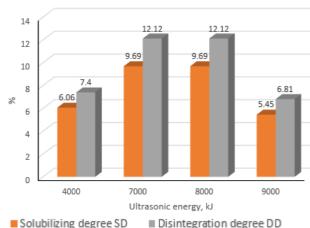


Fig. 4. Effect of ultrasonic energy to solubilizing and disintegration degrees in alkaline pH = 8.5, US time = 15 min., 80% amplitude

### **Conclusions**

Systematic study of determinant parameters for ultrasonic degradation of biological active sludge emphasized that increasing of disintegration and solubilizing degrees together with improvement of dimensional analysis parameters are advantaged by high ultrasonic energies (7000 - 8000 kJ), minimum 80% ultrasonic waves amplitude, 5 - 45 ultrasonic irradiation time,  $pH \sim 8.5$ , disintegration degree being 12 - 17%. These results have become the base for other bioreactor tests in order to establish the amount of biogas generating after ultrasonic pretreatment of biological sludge.

Acknowledgement: This work was supported by a grant from the Ministry of Research and Innovation, Program Nucleu, contract no. 38N/2016 (Project code PN 16-25 03 09).

### References

1.PATROESCU, V., JINESCU, C., COSMA, C., CRISTEA, I., BADESCU, V., STEFAN, C.S., Rev. Chem. (Bucharest), **66**, no. 4, 2015, p 537 2.STEFANESCU, M., BADESCU, V., COSMA, C., International Symposium The Environment and the Industry, Book of Abstracts, 2015, p. 54 3.STEFANESCU, M., COSMA, C., CRISTEA, I., IONESCU, I., BUMBAC, C., Rev. Chem. (Bucharest), **67**, no. 8, 2016, p. 1458 4.BRISOLARA, K.F., Water Environ. Res., **10**, no. 83, 2011, p. 1351 5.WANG, F., WANG, Y., JI, M., J. Hazard. Mater., **B123**, 2005, p. 145 6.ZHANG, P., ZHANG, G., WANG, W., Bioresour. Technol., **98**, no. 1, 2007, p. 207

7.KIM, D.H., JEONG, E., OH, S.E., SHIN, H.S., Water Res., **44**, 2010, p. 3093

Manuscript received: 11.11.2017