

EFFECTS OF THE SOLUBILISATION OF THE COD
OF MUNICIPAL WASTE IN THERMAL DISINTEGRATION

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Corresponding author's e-mail: S.Myszograj@iis.uz.zgora.pl**Keywords:** Biodegradable fraction of municipal waste, thermal disintegration, COD.

Abstract: This paper presents the content changes in the Chemical Oxygen Demand (COD) solubilised in hydrolisates obtained from thermally disintegrated municipal waste biofractions. A series of tests related to biowaste undergoing thermal treatment at the following temperatures: 55, 75, 95, 115, 135, 155 and 175°C were conducted for 0.5, 1 and 2 hours. The highest increase in COD solid fraction solubilisation (238%) was observed for the samples disintegrated at 175°C for 2 hours. The values of the reaction rate coefficient $k_{20} = 0.6 \text{ d}^{-1}$ and temperature coefficient $\theta = 1.023$ were determined. Statistical analysis of the multiple regression (correlation coefficient $R = 0.89$) showed that the temperature has a greater impact on COD solid fraction solubilisation – determined $\beta = 0.66$. The multiple correlation coefficient for the treatment time was $\beta = 0.61$.

INTRODUCTION

The strategic objective for economic development in the years to come is the achievement of a “green economy”, characterized in particular by a low level of greenhouse gas emissions and high energy efficiency. With reference to waste management, this means the maximum reduction in municipal waste landfilling, particularly biodegradable waste. This objective would be accomplished through the legal regulations in force: at EU level – the Landfill Directive (1999/31/EC) and the new Framework Directive (2008/98/EC) [35], and in Poland – The Waste Act and The Ordinance of the Minister of Economy and Labour dated September 7th, 2005 on the criteria and procedures for accepting waste at landfills intended for particular types of waste (Journal of Law no. 186, item 1553 as amended) [33]. The Directive imposes on the member states the necessity of developing a three phase program to reduce the landfilling of organic waste, and explicitly defines the required degree of reduction in the volume of biodegradable waste going to landfills and the timetable for its implementation. The Directive 99/31/EC obliges Poland to reduce its volume of landfilled biodegradable municipal waste in relation to the mass produced in the year 1995 – by about 25% by 2010, 50% by 2013 and 65% by 2020 [34, 35].

The Directive does not specify how to achieve the necessary degree of reduction in the biodegradable fraction of waste.

In Poland, in the year 2009, over 12,100,000 Mg of municipal waste was collected, including about 6.6 million Mg of biodegradable waste (54.6%) [36]. From the total volume of biodegradable waste, only 262 thousand Mg was treated using biological methods. Landfills are still the primary type of facilities for the management of municipal waste (522), and biological waste treatment is carried out in 51 green-waste and biowaste composting plants, 31 MBT plants and 3 waste digestion plants [36].

Data from the National Waste Management Plan indicate that up to 72.4% of the total volume of municipal waste is landfilled, and only about 2.3% of the generated waste volume is subjected to biological treatment processes. The estimated volume of waste will increase from 316 kg/inhabitant (2009) to 377 kg/inhabitant by 2022. It is expected that the volume of biodegradable waste produced in 2022 will amount to 7,761,000Mg of which less than 2,000,000 Mg will be landfilled. This assumption necessitates the development in the biological waste treatment technology [36]. Landfills emit landfill gas and leachate, which constitute an environmental hazard, also the waste deposited in them still remains a problem. Organic waste and sewage sludge can be treated by means of biological or thermal methods, which forces a reduction in its landfilling. The characteristic feature of these raw materials is their high moisture. This property means the use of biological processes to be more expedient than the thermal processes. The biological methods can be implemented in aerobic (composting) and anaerobic (methane digestion) conditions, or such processes that are a combination of these two technologies [2, 13].

The suitability of raw materials for biological treatment, as well as the efficiency of the processes of degradation depend on their chemical composition and structure. Besides organic compounds, the raw materials must also contain the appropriate proportions of other nutrients, and be characterised by adequate hydration and pH level. A large percentage of waste with high fat and protein has a positive impact on the digestion process, while a high content of lignin and hemicellulose reduces the efficiency of mixed waste biodegradation under anaerobic conditions [3, 11, 14, 19, 20]. In addition to the availability of organic carbon, the relationships between the other nutrients also determine the efficiency of the methane fermentation process. Besides the basic elements, the proper course of biochemical transformations, f synthesis and degradation requires access to the macro- and micronutrients such as iron, cobalt, molybdenum, selenium, calcium, magnesium, zinc, copper, manganese, boron and vitamin B₁₂ [9, 12, 16].

The demand for biogenic substances in the methane fermentation process is low, because of small increases in biomass. The basis for assessment of waste biodegradability are the dependencies whose optimal values for methane production are as follows [7, 8]:

– C:N = from 10:1 to 25:1

– C:P = 113:1

– C:N:P:S = (500–1000):(15–20):5:3 or COD:N:P:S = 800:5:1:0.5.

One method of improving the C:N ratio and, therefore, the biodegradability, is the mixing of substrates, for instance in the co-fermentation or composting process, where the volume of one of the substrates is usually greater (> 50%). The disintegration methods also give great opportunities to improve the waste characteristics.

Literature data as well as implementation and research work indicate that the disintegration of complex organic substrates can accelerate their hydrolysis and influence a change in the content of the insoluble fraction and the structure of organic compounds. Disintegration usually consists of the application of high/low temperatures and/or chemical reagents and ultrasounds. Tests on thermal disintegration cover the process optimality and are mainly directed to the acceleration of the hydrolysis of the molecular waste fraction, the production of volatile fatty acids, the biochemical hydrolysis of the remaining non-solubilised molecular fraction and an increase in the quantity of biogas and methane potential in the fermentation process. Hydrolysates can also enrich substrates with a high content of nitrogen compounds with organic carbon.

Sewage sludge is still the basic material in studies of the effectiveness of the various methods of disintegration. Only some of the known methods are currently the subject of interest in research centres implementing high-performance municipal waste treatment technologies. Thermal disintegration is recommended for the treatment of hydrated organic waste, which is not a good material for compost production (food residues, animal by-products, etc) [15, 27]. According to Schieder, preliminary thermal treatment before anaerobic stabilisation of the waste from restaurants and food residues [27] gives a lot of benefits when compared with the conventional digestion process: reduced time, higher load of dry organic matter, a reduction in the volume of the bioreactors, a reduction in the total cost of investment, despite the additional costs of thermal treatment, a reduction in the dry matter up to 70% and a higher recovery of biogas plus less need of hygiene requirements because of required sanitary conditions already guaranteed by the hydrolysis process.

Tsukahara *et al.* [28] carried out research, which tested the efficiency of the methane digestion of mechanically disintegrated kitchen waste and waste subjected to the process of hydrolysis at a temperature of 175°C and a pressure of 4 MPa. He ascertained a greater production of biogas from liquid hydrolysate than from the whole hydrolysed mass. The methane percentage in the biogas was 64%, 62% and 48% respectively for the whole mass of the waste, the liquid hydrolysate and the disintegrated waste. The conversion factor for the carbon compounds contained in the substrate to methane was 16.3%, 15.1% and 8% respectively. Using the same conditions, the experiment was conducted on kitchen waste by Sawayama [26]. The degree of solubilisation of the substrate, measured by an increase in the share of solubilised organic carbon was 75%.

Lissens *et al.* [18] obtained 64% of lignin hydrolysis in the process of the treatment of sorted waste biofraction at a temperature of 185°C (time 15 min.), while Fox *et al.* [11] subjected newsprint to solubilisation at 190°C (time 60 min.) with an effect of 93%. Papadimitriou [23] studied the disintegration of household waste at temperatures of 130°C, 160°C and 200°C. He confirmed the highest degree of hemicellulose and cellulose hydrolysis at 160°C, while the highest loss was determined at 200°C.

In practice, the high-temperature methods enjoy a good reputation, whereby it is necessary to mention the process of thermal-hydrolysis conducted in the technology provided by Cambi as one of them. In addition to several thermo-hydrolysis installations for sewage sludge, the installation of pretreatment of municipal waste was completed in the Cambi technology (Lillehammer, Norway 1999, 14 000 Mg of waste). The Cambi Technology involves the destruction of cells of excessive sludge and primary sludge thickened to about 12% D.M., which is based on heating the sludge to a temperature of 165–170°C at a pressure of 3 bar, followed by a decrease in pressure. The process time

is 30 minutes. The reduction in the dry matter of thermally disintegrated sludge in the digestion process is above 65%, while an increase in the biogas production compared with the conventional systems is about 30%. On top of this, a better final dehydration of sludge is obtained. Waste with the following morphological composition is subjected to thermal treatment in the installation in Lillehammer: 70–82% – kitchen waste, 2–7% – garden waste, 3–7% – paper, 8–12% – diapers, 1–4% – soil, small plastics, broken glass [25]. Electricity obtained from the biogas is 50% greater than in the classic non-hydrolysed waste digestion system, and the biodegradability of hydrolysates is over 70%. The use of the disintegration methods creates a chance to change the waste management system, based, so far, mainly on landfilling.

The article discusses the changes in the content of solubilised COD in hydrolysates obtained from the thermally disintegrated municipal waste fraction.

METHODOLOGY AND SCOPE OF STUDIES

The scope of the tests covered the performance of waste thermal disintegration. The municipal waste used in the studies was collected from an area of new buildings. The representative sample was collected in accordance with the guidelines included in PN-93/Z-15006. The test material consisted of segregated waste constituting fractions from 0 to 80 mm.

The morphological composition of waste used for testing, after the segregation of glass, plastics, metals, textiles, inert materials was as follows: kitchen waste (20–80 mm) – 61%, 0–20 mm fraction – 26%, paper – 12%, tiny sticks – 1%. The chemical composition of waste used in the studies is presented in Table 1.

Table 1. The chemical composition of wastes used in the studies

parameter	value
Moisture	59.8 %
Organic mass	62.0 % DM
C:N	18.1
Total Kjeldahl Nitrogen (TKN)	1.4 % DM
Protein	8.75 % DM
Total Organic Carbon (TOC)	25.4 % DM
Total phosphorus	7.1 % DM

The waste was subjected to treatment at the following temperatures: 55, 75, 95, 115, 135, 155 and 175°C for 0.5, 1 and 2 hours. The samples for disintegration were prepared by weighing 50 g of waste and adding 200 ml of distilled water. The thermal treatment was conducted in an autoclave Zipperclave 1.0. manufactured by Autoclave Engineers. The autoclave warm-up time lasted 10–20 mins depending on the required temperature, and the maximum cooling time was 15 mins. Evaluation of the impact of waste thermal disintegration on the change in the hydrolysate profile was performed on the basis of physico-chemical analyses of raw samples as well as samples that had undergone

treatment. The determinations of COD and its fractions were performed according to the current methodology.

RESULTS OF THE STUDIES

In order to determine the efficiency of thermal disintegration, an analysis of the chemical composition of water extract from the tested waste was performed. The value of COD in the water extract was 2120 g/m³, and COD of the molecular fraction was 951 g COD/ kg DM.

Fig. 1 illustrates changes in soluble COD (SCOD) and particulate COD (PCOD) which depend on the temperature and disintegration time.

Analysis of the results indicates the important effect of the temperature and disintegration time on the obtained value of soluble COD in hydrolysate. An increase in COD solid fraction solubilisation and a decrease in the TCOD value were observed after an increase in the treatment temperature during each series of tests. The lowest value for SCOD (2540 g/m³) was observed in the sample disintegrated at 55°C for 0.5 hour, and the highest value – 33,600 g/m³ for the sample treated at 175°C for 2 hours. Regardless of the disintegration time, a significant increase in the SCOD values was observed in samples exposed to treatment at a minimum of 135°C for 1 hour and at 115°C for the 2-hour treatment.

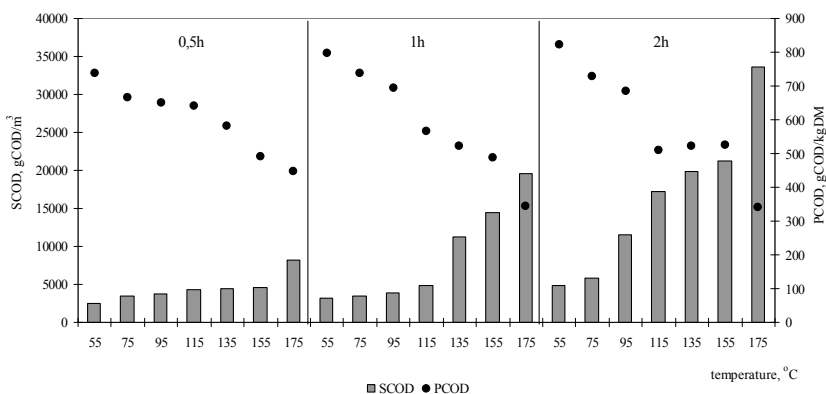


Fig. 1. Changes of soluble COD (SCOD) and COD bounded in suspension (PCOD) depending on temperature and disintegration time

The effectiveness of the disintegration process is evaluated by *inter alia*, an increase in the content of soluble COD in hydrolysate in relation to the initial value of COD solid fraction, bearing in mind the following dependency:

$$\Delta \text{SCOD} = \frac{[\text{SCOD}_t - \text{SCOD}_o]}{[\text{TCOD}_o - \text{SCOD}_o]} \cdot 100\% \quad (1)$$

SCOD_t – COD solubilised in the hydrolysate after thermal disintegration;

SCOD_o – COD solubilised in the sample of water extract;

TCOD_o – total COD in the raw waste sample.

Fig. 2 illustrates the efficiency of COD solid fraction solubilisation. Thermal treatment for a period of 0.5 hour resulted in an increase in the SCOD content from 3.2% (55°C) up to 45.6% (175°C). An increase in the disintegration time to 1 hour at temperatures ranging between 55°C and 115°C resulted in obtaining COD in hydrolysates comparable to that obtained during the 0.5-hour treatment. It was only at 135°C that the share of the solubilised COD was increased four times in the hydrolysate in comparison with treatment at the same temperature and a time of 1 hour.

The prolongation of the disintegration time to 2 hours caused the achievement of much better effects, even at lower treatment temperatures. For example, an increase in the content of soluble COD by 28.5% was obtained during the 2-hour treatment at 75°C while the increase in the solubilisation of the COD solid fraction was 71.4% at 95°C. The best effect was obtained for waste disintegrated at 175°C for 2 hours. In this sample, the increase in the SCOD content amounted to almost 238%.

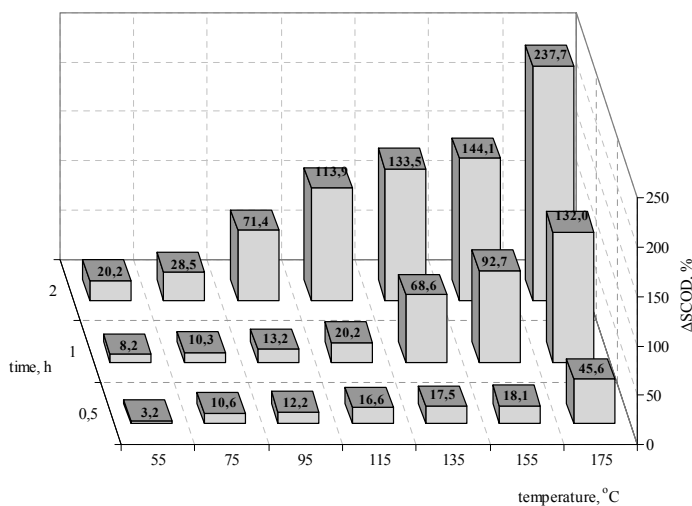


Fig. 2. Effectiveness of organic compounds liquidation (Δ SCOD,%) depending on temperature and disintegration time

DISCUSSION OF RESULTS

The process of thermal waste treatment in every sample caused the degradation of organic substances, and consequently resulted in a reduction in the total COD value. The values of SCOD depending on the degree of TCOD value reduction are illustrated in Fig. 3. Analysis of the results indicates that an increase in the soluble COD content in hydrolysates at a specific treatment temperature is related to a proportional decrease in the total COD.

At treatment temperatures in the range of 115–175°C, a total COD decrease from 20% to 45% was observed, whereby a smaller degree of mineralization (max. 23%) was obtained at a disintegration time of 1 hour and a higher share of soluble COD in the hydrolysates was found when compared with the treatment for 2 hours.

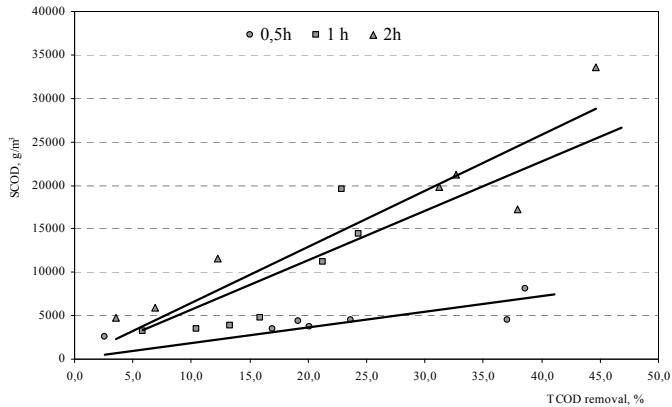


Fig. 3. Value of soluble COD (SCOD) depending on the level of total COD (TCOD) value decrease

Statistica 9.1 software was used in order to determine the correlation between the changes in the soluble COD values and the time and temperature of disintegration.

Partial correlations between the SCOD values in hydrolysates and the process parameters are positive, and amount to: $R = 0.82$ for the temperature and $R = 0.80$ for the treatment time. Multiple regression analysis (correlation coefficient $R = 0.89$) showed that the temperature has a greater impact on COD solid fraction solubilisation – determined $\beta = 0.66$. And the multiple correlation coefficient for the treatment time is $\beta = 0.61$.

On the basis of analysis of the data and dependences in pairs: COD/time/temperature, their inter-dependence of these parameters was confirmed according to the model equation [4]:

$$\text{SCOD} = \text{SCOD}_{\max} - [(\text{SCOD}_{\max} - \text{SCOD}_0) \cdot e^{-k \cdot t}] \quad (2)$$

Because the soluble COD value in hydrolysates for the invariable composition and fixed content of disintegrated waste dry matter depends exclusively on two parameters: temperature and time, the process effectiveness can be described with a mathematical model (2) taking into account the Van't Hoff-Arrhenius equation. [4, 21, 22]:

$$\text{SCOD} = \text{SCOD}_{\max} - [(\text{SCOD}_{\max} - \text{SCOD}_0) \cdot e^{-(k_{20} \cdot \Theta^{(T-20)}) \cdot t}] \quad (3)$$

SCOD – SCOD value (g/m^3);

SCOD_{\max} – max. value of SCOD (g/m^3);

SCOD_0 – initial value of SCOD in water extract from waste (g/m^3);

t – disintegration time (d);

T – temperature of disintegration ($^{\circ}\text{C}$);

k – first order reaction rate coefficient (d^{-1});

k_{20} – reaction rate coefficient in 20°C (d^{-1});

θ – temperature coefficient.

The determined value of the reaction rate coefficient k_{20} is 0.6 d^{-1} , and in the case of the temperature coefficient, it is equal to $\theta = 1.023$. The matching area of the measurement data determined by the following equation ($R = 0.94$) is illustrated in Fig. 4.

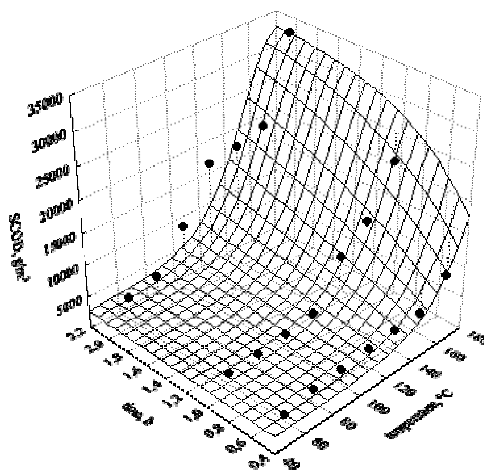


Fig. 4. The non-linear regression model fitting area to measurement data

CONCLUSIONS

The effect of thermal municipal waste biofraction disintegration on the content change in the obtained hydrolysates was tested in order to check the possibilities of accelerating the hydrolysis process which limits biodegradation under both the aerobic and anaerobic conditions. Analysis of the results obtained from the performed tests shows that the method of thermal waste treatment has a positive impact on those characteristics which increase its potential susceptibility to biodegradation. The series of performed tests demonstrated an increase in the content of SCOD in TCOD in all samples after disintegration. The tests confirmed the literature data on the high effectiveness of biowaste hydrolysis at treatment temperatures exceeding 135°C and the possibility of solid fraction solubilisation. Statistical analysis of multiple correlations between parameters showed that the temperature, and not the treatment time, has a higher effect on the disintegration process.

The values of the reaction rate coefficient $k_{20} = 0.6 \text{ d}^{-1}$ and temperature coefficient $\theta = 1.023$ were determined for the analysed tests results. The value of the reaction rate coefficient for the solubilisation of the solid waste fraction (hydrolysis) at 37°C calculated on the basis of designated parameters k_{20} and θ amounts to 0.88 d^{-1} .

For comparison, the values of the reaction rate coefficients k for the biochemical degradation reaction given in the literature amount to, e.g.:

- for kitchen waste from $0.12 (35^{\circ}\text{C})$ [17] to $0.55 \text{ d}^{-1} (37^{\circ}\text{C})$ [30],
- for municipal solid waste from 0.1 to $0.34 \text{ d}^{-1} (37^{\circ}\text{C})$ [17, 24, 30, 31],
- mono-substrates: for carbohydrates $0.025\text{--}0.200 \text{ d}^{-1} (55^{\circ}\text{C})$ [6], $0.5\text{--}2.0 \text{ d}^{-1}$ [12]; for proteins $0.015\text{--}0.075 \text{ d}^{-1} (55^{\circ}\text{C})$ [6]; for fats $0.005\text{--}0.010 \text{ d}^{-1} (55^{\circ}\text{C})$ [6], for cellulose $0.04\text{--}0.13 \text{ d}^{-1}$ [13], $0.066 \text{ d}^{-1} (35^{\circ}\text{C})$ [17].

The higher the value of this coefficient, the higher the substrate hydrolysis rate and its assimilation. The value of the coefficient θ which describes the temperature effect on the reaction rate is presented in the literature in the range between 1.013 (sewage sludge) and 1.066 (hydrocarbons) [3, 20, 21]. The higher the value of this coefficient, the higher the temperature effect on the decomposition rate, and thus the substrate biodegradability.

The availability of hydrolysed, easily assimilable organic compounds, especially volatile fatty acids for microorganisms, is one of the factors that determines the efficiency of the methane digestion process, aerobic degradation or denitrification and dephosphatation.

Therefore, the speeding up of the solubilisation of the organic compounds contained in the biowaste into soluble forms results in a significant improvement in the efficiency of the progress of these processes [10].

Not only does the solubilisation level of solid sludge fraction, but also the form of organic compounds [4, 21] arising from the disintegration process determines the biodegradability. As many authors have proved in their studies, the established optimal conditions for performing the process are not identical for all substrates. Barlindhaug and Odegaard [1], Wilson and Novak [32] and Bougrier [5] came up with the hypothesis that hydrocarbons are more susceptible to mineralisation than proteins during the thermal treatment process. However, proteins can undergo solubilisation more easily. Therefore, precise evaluation of the substrate quality before initiating the disintegration process is desirable [1]. As a result of thermal treatment, hydrolysate with a characteristic "strong tea" colour is formed. As well as other components, such as Amadori products, which are potential inhibitors of methane fermentation it also contains biodegradable compounds.

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EFEKTYWNOŚĆ UPŁYNNIENIA ChZT ODPADÓW KOMUNALNYCH W DEZINTEGRACJI TERMICZNEJ

W artykule omówiono zmiany udziału ChZT rozpuszczonego w hydrolizatach uzyskanych z dezintegrowanej termicznie biofrakcji odpadów komunalnych. Wykonano serie badań, w których bioodpady poddawano obróbce w temperaturach 55, 75, 95, 115, 135, 155, 175°C w czasie 0,5, 1 i 2 godzin. Największy wzrost upłynnienia frakcji stałej ChZT (238%) uzyskano dla próbek dezintegrowanych w temperaturze 175°C w czasie 2 godzin. Wyznaczono wartość współczynnika szybkości reakcji $k_{20}=0,096 \text{ h}^{-1}$ i współczynnika temperaturowego $\theta=1,053$. Analiza statystyczna wyników badań wykazała, że większy wpływ na efekt dezintegracji ma temperatura, niż czas obróbki.