ABSTRACT

Introduction: Rational waste sludge disposal means utilizing its fertilizing and calorific properties to their maximum. The ability to convert sewage sludge depends on many factors. Fermented or aerobically stabilized sludge contains components easily assimilable by plants, but its utility in agriculture or gardening is limited. However, stabilization of sewage sludge in the composting process ensures a beneficial modification of its sanitary and epidemiological condition, as well as significantly improving the organoleptic properties, such as odor, color, and granulation; this stabilization also influences the fertilizing value. Before utilizing the compost created from sewage sludge or other bio-waste, it is necessary to check the degree of its stabilization by evaluating the biological activity. One of the methods of evaluating the biological activity of compost is the respiration test, e.g. using the AT4 test (Static Respiration Index). Literature data of respirometric tests show the availability of substrates for microorganisms; in other words, the susceptibility of a substrate to further biodegradation.

Aims: This article analyzes the results of biological activity tests (AT4) of compost from installations for the aerobic stabilization of sewage sludge and biofractions of municipal waste. The
1. INTRODUCTION

The primary objective of waste disposal is to reduce the amount of waste to landfills, especially when it comes to biodegradable waste. Due to the implementation of Directive 99/31/EC regarding the stockpiling of waste, prohibiting the acceptance by landfills of waste other than hazardous, including sewage sludge, operators of wastewater treatment plants are forced to change the methods of waste disposal applied thus far [1]. The directive imposes on member states the obligation to implement a three-stage program to limit the stockpiling of organic waste, and simultaneously specifies the required reduction rate of the amount of biodegradable waste sent to landfills. Another European Union requirement associated with the construction of new waste treatment plants results in an increase of sewage treatment and the production of sewage sludge [2]. The utilization of sewage sludge in the agriculture of EU states is regulated by the Council’s Directive 96/278/ECC on environmental protection [3]. In Poland, the principles of utilizing sewage sludge are regulated by the Resolution of the Minister of Environment regarding municipal sewage sludge (Journal of Laws of 2015, pos. 257) [4]. In the year 2013, 3344 waste treatment plants servicing 70.3% of the population produced 540.3 thousand tons of d.m. municipal sewage sludge [5]. In Poland the dominant direction of sewage sludge disposal in 2013 was its utilization in agriculture (105.4 thousand tons of d.m.; 19.5%), 29.4 thousand tons of d.m. (5.4%) was used for recultivation of areas and 32.6 thousand tons of d.m. (6%) was used for the cultivation of plants intended for compost production. 13.5% of municipal sewage sludge – 72.9 thousand tons of d.m. – was subjected to thermal recycling, while 31.4 thousand tons of d.m. (5.8%) was stockpiled. It should be noted that according to statistical data, 41% of the produced sludge (219.8 thousand tons of d.m.) was stockpiled at sewage treatment plants [5,6].

Management of sewage sludge disposal is currently aimed towards reducing the amount of stockpiled sludge and increasing its processing rate using thermal recycling methods. Eurostat statistical data regarding the period of 1999-2014 shows an apparent increase in the significance of thermal recycling methods in sewage sludge disposal. Thermal methods of sewage sludge disposal are not, however, broadly and commonly used in all European Union countries [7].

Rational sludge recycling involves utilization of its fertilizing and calorific properties to the maximum extent. The possibilities of processing sewage sludge depend on many factors. These are the physical (moisture, susceptibility to dehydration, calorific value, and combustion heat), chemical (heavy metal, dioxin, and furan content), and sanitary (presence of bacteria, viruses, fungi,

aim of the study was the observation of changes to the degree of stabilization of substrates, during a period of 10 weeks of aerobic processing.

Methodology: The studies on a technical scale were conducted at the mechanical and biological processing of municipal waste (MBP) and selectively gathered green waste and bio-waste processing (composting) installation. The fermented sewage sludge was composted together with the organic substrate acquired from municipal waste. The process was conducted during a period of 10 weeks. Every 7 days samples were taken from the aerated heap in order to determine the moisture, ignition loss, and AT4 parameter. The measurements of the compost’s biological activity were conducted using an OxiTop® Control measurement system.

Results: The mixture of municipal waste biofractions and fermented sewage sludge that made up the heaps turned for the composting process was characterized by the following parameters: moisture – 61%, organic substance content – 58% of dry matter, AT4 – 64.5 gO₂/kg of dry matter. Continued monitoring of temperature changes (average temperature 39°C/61-67°C/55-35°C respectively) in the heaps confirmed the correct course of the composting process in three phases. After 10 weeks of the process a reduction of organic substances by 54% was achieved, while the value of the AT4 parameter in the stabilized compost was 4.9 gO₂/kg of dry matter.

Conclusion: The study results show that changes to the content of organic substances in waste subjected to the composting process are directly proportional to changes of the AT4 parameter, which characterizes the respiration activity of substrates. This correlation can be fairly accurately described by a linear equation (R²=0.93).

Keywords: Compost stability; static respiration test; respiration index.
protozoa, and parasite eggs) properties. Equally important are the legal regulations and economic aspects, primarily the acceptable level of processing costs.

The aim of the study was to observe the degree of substrate stabilization during the 10 week composting process. The analysis encompassed study results of the biological activity (AT4) of compost from the sewage sludge and mixed municipal waste biofraction. The AT4 test results may be helpful in the interpretation of the efficiency and optimization of MBP plants.

2. TECHNICAL CONDITIONS OF SEWAGE SLUDGE COMPOSTING

Aerobic or anaerobic biological processing of municipal waste and sewage sludge are the most commonly used methods. Fermented or aerobically stabilized sludge includes contents which are easily digestible by plants, however its use in agriculture or gardening is limited. On the other hand, stabilization of sewage sludge in the composting process ensures, to a certain degree, a positive change of its sanitary and epidemiological condition, as well as significantly improving the organoleptic properties, such as odor, color, and granularity, and has a positive effect on the fertilizer value. The increase of temperature during the composting process contributes to the reduction of pathogenic microorganisms, and primarily bacteria. When highlighting the value of compost as an organic fertilizer, sources list its beneficial, loosening effect on the structure of heavy soils, the binding of light and sandy soils, as well as increasing the water and heat capacity of soils. Additionally, apart from maintaining the agronomical benefits of supplementing soil with organic substances, composting also enables sterilization and odor stabilization. After composting, sewage sludge changes its characteristics. Compost may be stored awaiting more favorable conditions for utilization. Fertilization will take place without onerous odors, which often determine the inability to utilize stabilized sewage sludge that has not been composted.

The correct course of the composting process requires an optimal moisture level of the material and a certain minimum volume of free air space (FAS), in order to maintain the aerobic conditions within the composted material. Factors which directly influence the porosity and permeability of composted materials are the shape and size of particles. Fine, regularly shaped particles fill out the available spaces to a significant degree, thereby reducing the air porosity and permeability. The published studies show, that for most composted waste, the optimal moisture levels of 45% - 65% correspond to FAS values ranging from 36% to 50% [8]. In the case of composting a mixture of municipal waste and sewage sludge, the FAS ratio should reach a value above 30% [8]. Most data available in the literature do not specify whether the optimal FAS value refers to the initial porosity in the heap, or a value that needs to be maintained throughout the duration of the entire composting process [9]. The oxygen and water content in material subjected to the composting process is a determining factor of the biological activity of microorganisms involved in that process. On the other hand, their availability is inseparably connected to the material’s air porosity parameter. The correct content and movement of air within the composted material influences the level of oxygen, the removal of carbon dioxide and excessive moisture, as well as limiting the possibility of excessive accumulation of heat within the material. In situations where excessive water is accumulated, it fills out free spaces, which in turn leads to a decrease in air porosity of the composted material. The limitation of free air spaces leads to decreased air permeability, thereby limiting transport of oxygen and discharge of water and heat from the compost heap [9].

Sludge produced in sewage treatment plants is characterized by a high water content and low porosity, as a result of which the achievement of the correct structure and moisture of the composted mass requires the introduction of structure-forming materials. The air porosity may be increased by the addition of natural structure-forming material into the compost mixture, such as straw, wood shavings, or plastic materials, as well as biofractions of municipal waste or compost. Fermented and dehydrated sludge is safe from a sanitary perspective; however, the methane fermentation process partially removes organic substances from it, which creates the necessity to add easily degradable organic waste. Hydrated sludge intended for composting requires additional material in order to achieve the required water content and create the correct aeration conditions. With a water content above 60%, local anaerobic processes take place during composting. Most frequently raw sludge dewatering to 18-25 % of d.m. is sent for composting [10].

Composting, as any other biochemical process, requires the creation of not only appropriate site
conditions, but also provision of the appropriate substrate amounts required by the microorganisms responsible for the degradation to conduct metabolic reactions. The following elements have to be balanced: carbon, nitrogen, phosphorous, microelements, oxygen (air) delivered to the heap, moisture, heat, and residual (inert) substances. Sewage sludge is characterized by a low C/N ratio (approx. 15), unfavorable to the composting process. The composting technology of sewage sludge itself requires provision of a correct amount of additional organic matter containing organic carbon into the sludge. Data provided in the literature show that this ratio should be 22-35:1 [11]. This optimal value can be achieved by mixing nitrogen-rich sewage sludge with carbon-rich organic waste (grass, straw, paper, wood straw, wood chips, and bark). It also seems beneficial to conduct the composting of sewage sludge together with the organic portion of segregated municipal waste. The addition of a carbon substrate improves the water-air ratios in the heap, regulates the C:N ratio, and in the final composting phase enables the growth of fungi and molds. The type of the additional, organic structure-forming material used influences the compost quality and the composting process speed, and most frequently depends on accessibility and cost. Particular attention should also be given to the amount of microelements which are absorbed and accumulated by plants growing in soils fertilized with compost. These are primarily heavy metals, whose digestibility and toxicity decreases with the increase of pH. For that reason, the compost's reaction should not be lower than 6.5.

Despite many positive aspects and benefits associated with the use of compost, its sales markets are still limited. The barrier to utilizing compost is the difficulty in achieving a constant and objective evaluation of its quality and process control. Assessment of the composting process and its effectiveness is possible primarily through the analysis of the organic substance content. Additional information on the degree of compost stabilization is provided by the activity tests.

In EU countries, there are studies currently being conducted on consolidating the limits and testing methods for determining the biological activity of waste. In reference to the correct assessment of the loss of the capacity of the waste for further biodegradation, in Germany, Austria and Poland, static respiration test (AT4) was considered the most appropriate parameter. The AT4 index provides a visualization of not only the loss of total organic substance matter as a result of mineralization processes, but also of the decreased ability of organic matter to undergo further degradation in the aerobic process.

The aim of conducting the tests is the monitoring of the biological activity of waste decomposition during composting, the assessment of the waste’s biological activity decrease before stockpiling, and the control of processes which take place in landfills.

3. MATERIALS AND METHODS

3.1 Scope of the Test

The studies on a technical scale were conducted at mechanical - biological pretreatment (MBP) installation of municipal waste, selectively gathered green waste and bio-waste. The applied technological processes and utilized facilities enable the segregation, from the entire stream of gathered municipal waste, of a range fractions, depending on their characteristics and morphology, in order to further direct them to the corresponding recycling process. The municipal waste treatment technology covers the division of the stream of municipal waste into organic waste, raw material waste, waste with energy properties, as well as separation of the hazardous waste fraction and ballast. It also allows the composting of the biofraction, compost purification as well as treatment and conditioning of secondary raw materials. The first fraction separated at the plant is the so called subscreen fraction with a diameter of 20 mm, which is used as the transfer material at the landfill. The remaining part of the waste is divided into organic fraction, also called biofraction, and oversize fraction (>80 mm). The bio-fraction is cleansed from minor elements such as: plastics, glass and batteries, and then it is directed to the composting hall together with the contaminated waste cardboard. The resulting organic matter was subjected to composting using the aerated heap method during a 10 week period. The organic substrate acquired from municipal waste was composted together with fermented sewage sludge from the waste water treatment plant, operating in a system of mechanical and biological treatment involving nutrient removal. The finished compost was further purified in the compost refinement line, consisting of a screening drum, ballistic table, cyclone, and a system of belt conveyors. The studies were
conducted in an aerated heap of 70m in length, 6m in width and 1.8m in height (Fig. 2). The substrate for composting was prepared by mixing 94 Mg of dehydrated, fermented sewage sludge and 165 Mg of municipal waste biofractions with the parameters stated in Table 1. The morphological characteristics of municipal waste after mechanical processing and being sent for composting are shown in Fig. 1.

A measurement of moisture and temperature was performed in the heap every day during the entire study cycle. The waste was turned with a frequency of once per week. Moisture, ignition loss, and the AT4 parameter were determined in the waste sampled from the heap every 7 days. The general waste sample taken during the stabilization process was prepared by taking 10 increment samples with a minimum mass of 10kg each, at regular intervals during a typical working day. The gathered increment samples were mixed thoroughly. Measurements of the compost’s biological activity were conducted using an OxiTop® Control measurement system.

### Table 1. Characteristics of substrates used in the studies

| No | Parameter       | Unit | Bio-fraction of municipal waste | Sewage sludge |
|----|-----------------|------|---------------------------------|---------------|
| 1  | Moisture        | %    | 46.9                            | 85.7          |
| 2  | Organic matter  | % d.m.| 59.6                           | 44.5          |
| 3  | AT4             | gO₂/kg d.m. | 69.7                      | 30.8          |

![Fig. 1. Morphological characteristics of municipal waste after mechanical processing](image1.jpg)

![Fig. 2. Aerated heaps](image2.jpg)
3.2 Methodology of Calculations

The method of determining the respiration activity serves to assess the biological reactivity or maturity of stabilizers (composts) in aerobic conditions. It specifies the mass amount of oxygen used by microorganisms within a specific time. In this method, the oxygen requirement may be determined by measuring the amount of oxygen used or carbon dioxide produced, and is expressed in g O₂/kg of dry matter waste [12].

The amount of used oxygen may be specified manometrically, based on the measurement of the carbon dioxide produced during the biodegradation process. The carbon dioxide is bonded by the absorbent agent (e.g. sodium hydroxide, caustic soda solution, soda lime or potassium hydroxide) and does not appear in the form of free gas.

As a consequence, the change in pressure is assigned only to oxygen consumption. In order to be able to measure the oxygen consumption by means of a manometer, the following conditions must be fulfilled:

- A biologically active sample must be placed in a vessel which is impermeable to gas,
- A sufficiently large air space must be maintained above the sample, which will ensure the unlimited access of oxygen to the biodegradation processes,
- The measuring vessel must contain the agent that absorbs carbon dioxide, but the sample may not come into contact with the agent.

AT4 is a parameter that determines the demand for oxygen necessary for the decomposition of waste within four days.

In order to carry out the respirometric test, a set including the OxiTop® OC 110 controller and MG 2.5 measuring vessels was used. The OxiTop apparatus consists of reaction vessels, adapters with fixing clamps and measuring heads. The stabilised material samples are placed in reaction vessels, which are closed tightly using an adapter with a measuring head fixed outside which contains an electronic vacuum pressure sensor. Inside each vessel, there is an attachment secured to the adapter, on which the CO₂ absorbent (Fig. 3) is placed. The measurement was performed at a temperature of 20±1°C for samples with a humidity ranging from 40 to 50%.

Despite the fact that the AT4 parameter presents the 4-day demand for oxygen by microorganisms, the test itself must be carried out longer – typically for 7 days. This is related to the potential occurrence of the so called lag-phase during the test. 4 phases of microorganism development can be distinguished:

- Phase 1 - Primary inhibition phase, also called the resting or adaptation phase. It occurs during the transfer of the bacterial colony into a new environment and during changes in the environmental conditions (as, for instance, at sample defrosting).
- Phase 2 - Intensive development phase, also called the logarithmic growth phase. The cells divide intensively, taking advantage of the favourable conditions.
- Phase 3 - Equilibrium (stationary) phase, in which equilibrium is established between the newly formed cells and the dead cells. The quantity of available nutrients is limited then by the number of cells.
- Phase 4 - Death (decline) phase. This occurs when the sources of nutrients begin to deplete and/or the concentration of metabolism products increases to a level which is harmful to the bacteria themselves.

During the AT4 measurement, the impact of the first phase and partly the second phase, when the amount of microorganisms is too small to accomplish the full “metabolic efficiency”, is responsible for the lag-phase. An assumption has been made that the lag-phase ends when the mean oxygen demand after 3 hours reaches 25% of the 3-hour mean values, occurring at the time of a maximum increase in the oxygen demand, determined for the first four days. The oxygen mass used during the lag-phase is deducted from the oxygen mass taken up during the whole test (lag-phase + 4 days). The oxygen mass from the lag-phase may not exceed 210% of its total demand during the first 4 days.

The basis for calculations of the AT4 parameter is the ideal gas law. It follows from the general gas equation that each pressure change is related to a change in the quantity of substance at the constant vessel volume and constant temperature. The quantity of substance is a result of the m/M quotient form [13]:

\[
\text{m} = \frac{pV}{RT}
\]
Fig. 3. Oxi-Top set

\[ \Delta p = \Delta m \cdot R' \cdot T \cdot V^{-1} \cdot M^{-1} \]  

where:
- \( M \) – molar mass of substance,
- \( \Delta p \) – pressure difference,
- \( R' \) – gas constant for CO\(_2\) in normal conditions,
- \( T \) – temperature,
- \( V \) – volume of the measuring vessel.

Equations 1 present the correlation between the pressure change and the oxygen consumption.

During measurement, the test vessel should be located in a place characterised by a constant temperature. Changes in the temperature lead to changes in pressure, which gives incorrect the measurements of oxygen consumption. At an initial pressure equal to 1000 hPa and temperature of 20°C (293 K), an increase in temperature by 1°C causes an increase in pressure to 1003 hPa. With the measuring system sensitivity equal to 1 hPa, this means an error which cannot be ignored.

The biological activity (respiration) of waste samples has been calculated on the basis of the following equation:

\[ AT4 = \frac{M_{R}(O_2) \cdot V_{fr} \cdot \Delta p}{R \cdot T \cdot m_{Bi}} \]  

where:
- \( AT4 \) – biological activity of a sample (g O\(_2\)/kg of dry mass),
- \( M_{R}(O_2) \) – molar mass of oxygen: 32000 (mg/mol),
- \( V_{fr} \) – free gas volume (dm\(^3\)),
- \( R \) – general gas constant: 83.14 (dm\(^3\)•hPa•(K•mol\(^{-1}\)),
- \( T \) – measurement temperature (K),
- \( m_{Bi} \) – dry mass of substrates in a sample (kg of dry mass),
- \( \Delta p \) – pressure drop during measurement (hPa).

4. RESULTS AND DISCUSSION

The mixture of municipal waste biofractions and fermented sewage sludge that made up the heaps turned for the composting process was characterized by the following initial parameters (start of process):
- Moisture - 61%
- Organic substance content - 58% of d.m.
- AT4 – 64.5 gO\(_2\)/kg of d.m.

Monitoring of temperature changes in the heaps has confirmed that the correct course of the composting process comprises of three phases:

I phase – Initial composting, also called the mesophilic or raising temperature phase, lasting 3 days, with an average temperature of 39°C;

II phase – Intense composting, also called the thermophilic or high temperature phase; during this phase easily decomposable organic compounds are subject to degradation, with the end products of such degradation being water, ammonia, and carbon.
dioxide. The duration of intense composting is approx. 4-5 weeks, with a temperature increase to approx. 61-67°C.

III phase – Proper composting, also called the conversion phase. The characteristic features of this phase are a decrease in temperature and, most of all, conversion of hard and decomposable compounds by fungi and mesophilic bacteria. This phase begins around the 5th week of composting, with a temperature approx 55-35°C.

The IV phase of compost maturing, which lasts up to several months and exceeds beyond the period of the study, was not observed.

Changes to the content of organic substances and the AT4 parameter during the 10-week composting process are presented in Fig. 4. The organic matter content changed by decreasing gradually within a range from 46% after the first week of composting to 27% in the stabilized compost.

After the first week of composting the AT4 parameter value decreased from the initial value in the raw substrate of 64.5 gO₂/kg of d.m. to 26.7 gO₂/kg of d.m. and maintained that level until the 3rd week of the process. Further aerobic stabilization of the organic substance during 4 subsequent weeks caused a decrease of that parameter’s value to approximately 12 gO₂/kg of d.m. By the end of the composting process, AT4 changed only to a slight degree, from 6.8 gO₂/kg of d.m. to the end value in the stabilized compost at the level of 4.9 gO₂/kg of d.m.

The primary aim end effect of the composting process is the decrease of the organic substance content in the processed substrates. The effectiveness of organic substance mineralization in the conducted composting process is presented in Fig. 5.

After 10 weeks of the process, a reduction of the organic substance content by 54% was achieved compared to initial value in start process.

After the first week the effect measured by this parameter was 21%, in the second and third week it maintained a level of approximately 29%. In weeks 4 to 9 the effectiveness of organic substance mineralization altered between 40 to 47% (average effectiveness of 44%). The achieved effectiveness and course of the sewage sludge and municipal waste biofraction composting process were comparable with the research literature and operational data [13,14].

In Poland, the process of biological mixed municipal waste recycling should be conducted in a manner that makes the resulting stabilized compost meet the following requirements, corresponding to the 11th of September 2012 Resolution by the Minister of Environment “regarding the mechanical and biological recycling of mixed municipal waste” [14], namely:

- Ignition losses of the stabilized compost are below 35% of dry matter, while the organic carbon content is below 20% of dry matter, or
- The loss of organic matter in the stabilized compost compared to the organic matter in waste measured by the ignition loss or organic carbon content is above 40%, or
- The AT4 value is below 10 g O₂/kg of dry matter.

In the conducted studies, the stabilized compost reached the above limits at the following times:

- Ignition loss - in the 4th week of composting;
- Loss of organic matter in the stabilized compost compared to the organic matter in waste measured by the ignition loss – in the 4th week of composting;
- AT4 value – in the 8th week of composting.

As can be seen, meeting the condition of the required AT4 value was not tantamount to achieving the limit for organic matter content in the stabilized compost and loss of organic matter. It was also confirmed in the analysis of correlations between those parameters. Changes to the AT4 parameter depending on the organic matter content in stabilized waste are presented in Fig. 6. The required level of stabilized compost ignition losses of below 35% of dry matter corresponds to the AT4 parameter value of approx. 15 g O₂/kg of d.m. (Journal of Polish Laws of 2015, pos. 257).

The study results have shown that changes to the organic matter content in waste and stabilized compost subjected to the composting process are directly proportional to changes of the AT4 parameter value, which characterizes the respiration activity of substrates. This correlation may be accurately described by a linear equation (R²=0.93).
Fig. 4. Changes to the organic matter content and AT4 parameter during the 10-week composting process

Fig. 5. Percent reduction of organic matter compared to initial value during composting process

Fig. 6. The relation between organic matter and AT4 values
5. CONCLUSION

Organic matter, after being converted to compost, may be used as a fertilizing, structure-forming or re-cultivating material. It is a valuable organic fertilizer able to replace manure and other organic fertilizers in garden production, especially in suburban regions, where there is a deficit of the latter. Compost also shows utility in the establishment and maintenance of urban greenery. The high temperature of the composting process ensures the sanitary safety of the compost, which must also meet requirements in terms of heavy metal content. Organic substances contained in compost influence the physical and chemical properties of soil, improve water-air ratios and the soil’s richness in nutrients. But before utilizing the compost created from sewage sludge or other bio-waste, it is necessary to check the degree of its stabilization by evaluating loss of organic matter or the biological activity.

The conducted test of composting of mixing sewage sludge and municipal waste biofractions demonstrated that it is possible to obtain a stabilized product. Accordance with the requirements has been achieved the limits: ignition loss and loss of organic matter in the stabilized compost compared to the organic matter in waste in the 4th week of composting but AT4 value in the 8th week of composting. After typical process time for composting - 10 weeks - a reduction of organic substances by 54% was achieved, while the value of the AT4 parameter in the stabilized compost was 4.9 gO2/kg of dry matter. These results confirm the high effectiveness of the process.

The study results have shown that changes to the organic matter content in waste and stabilized compost process are directly proportional to changes of the AT4 parameter value. This correlation may be accurately described by a linear equation (R²=0.93).

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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