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Application of a modified OxiTop® respirometer for laboratory composting studies

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Abstract: This study applied a modified OxiTop® system to determine the oxygen uptake rate during a 2-day respiration test of selected composting materials at different moisture contents, air-filled porosities and composition of composting mixtures. The modification of the OxiTop® respirometer included replacement and adjustment of a glass vessel (i.e. a 1.9-L glass vessel with wide mouth was used instead of a standard 1-L glass bottle, additionally the twist-off vessel lid was adjusted to attach the measuring head) and application of a closed steel mesh cylinder of 5 cm in diameter and 10 cm in height with the open surface area of the mesh of approximately 56.2%. This modification allowed obtaining different bulk densities (and thus air-porosities) of the investigated composting materials in laboratory composting studies. The test was performed for apple pomace and composting mixtures of apple pomace with wood chips at ratios of 1:0.5, 1:1, 1:1.5 (d.w), moisture contents of 60%, 65% and 75% and air-filled porosities ranging from 46% to 1%. Due to diverse biodegradability of the investigated apple pomace and composting mixtures this test allows for the determination of the effects of different air-porosities (due to compaction in a pile) on the oxygen uptake rate for mixtures with a fixed ratio of a bulking agent. The described method allows for laboratory determination of the effects of moisture content and compaction on biodegradation dynamics during composting.

Introduction

Composting is a process of natural aerobic decomposition where organic matter in biodegradable materials is oxidized by microorganisms at the atmospheric air and under controlled conditions. The process results in a product that is similar to humus, it contains humic and biogenic substances, and is stable and free from odors and pathogens (Haug 1993, Rynk 1992, Jędrczak 2007, Epstein 2011). Biodegradability of composting materials, dynamics of organic matter degradation during composting and stability of produced composts play an important role in composting. These parameters are directly related to the respiration rate which corresponds to metabolic activity of microorganisms that is higher in the presence of bioavailable organic matter (Haug 1993, Jędrczak 2007, Gómez et al. 2004). It is determined by such indicators as oxygen consumption (i.e. oxygen uptake rate) and carbon dioxide evolution (Haug 1993, Jędrczak 2007). The oxygen uptake rate can be expressed in mg O_2/g volatile solids (VS) per hour or day, whereas the production of carbon dioxide in mg CO_2-C/g C per hour or day (Jędrczak 2007). For composting studies it is recommended to measure the oxygen uptake rate as carbon dioxide can be produced both in aerobic and anaerobic conditions (Gómez et al. 2004, Jędrczak 2007, Villaseñor et al. 2011). The respiration rate based on oxygen consumption depends on several factors such as: mass of a material sample and its biodegradability and stability, temperature, moisture content, bulk density and air-filled porosity, time and conditions of preincubation and incubation (Haug 1993, Gómez et al. 2004, Richard et al. 2002, Cronjé et al. 2004, Malińska and Richard 2006, Ahn et al. 2008). Oxygen consumption and carbon dioxide production can be determined by respirometry including dynamic and static methods for both solid and liquid samples (Haug 1993, Jędrczak 2007). The dynamic method is based on the differences between concentrations of oxygen and carbon dioxide in the inlet and outlet from a composting reactor. In the case of static methods which use closed vessels, the oxygen uptake can be measured by e.g. manometric respirometers, whereas carbon dioxide can be captured by an alkaline trap (i.e. the titration method) (Haug 1993, Jędrczak 2007). Literature provides many examples of respiration rates based on the oxygen uptake rate employed for determination of biodegradability of selected substrates and composting mixtures (Ahn et al. 2008, Barrena et al. 2011), bench-scale and laboratory studies on composting (Gómez et al. 2004, Körner et al. 2003, Tremier et al. 2005,

Puyuelo et al. 2010, Grigatti et al. 2011) and determination of stability of produced composts (Sadaka et al. 2006, Kilian and Macedowska-Capiga 2011, Sánchez et al. 2012, Cáceres et al. 2015). Currently, the determination of respiration rates based on oxygen consumption can be performed with an advanced laboratory equipment, e.g. Micro-Oxymax (Gómez et al. 2004), electrolytic respirometers, e.g. Sapromat® (Villaseñor et al. 2011, Kilian and Macedowska-Capiga 2011, Sánchez et al. 2012, Binner et al. 2012), manometric respirometers, e.g. the OxiTop® system (Sadaka et al. 2006), diverse modified static and dynamic respirometers (Gómez et al. 2004, Malińska and Richard 2006, Körner et al. 2003, Tremier et al. 2005), and gas chromatography or oxygen electrodes (Jędrczak 2007).

The OxiTop® system and its available options (WTW, Weiheim, Germany) has a wide range of applications in environmental engineering, including determination of a biological oxygen demand (BOD), oxygen uptake rate, soil respiration, biological decay, susceptibility of materials to biodegradation in aerobic and anaerobic conditions (Malińska and Richard 2006, Ahn et al. 2008, Sadaka et al. 2006, Zieliński et al. 2013, Lamy et al. 2013, Veeken et al. 2003). There are also many examples of the OxiTop® applications for composting studies, e.g. for determination of biodegradability of various types of waste materials in aerobic conditions, stability and maturity of produced composts (e.g. respiration activity AT_4) (Binner et al. 2012), and for investigation of composting dynamics parameters for selected mixtures (Malińska and Richard 2006, Myszograj et al. 2014). Veeken et al. (2003) used the OxiTop® system for determination of stability of various organic wastes and composts produced from green waste. Jeris and Regan (1973) studied the effects of moisture content and air-filled porosity on the biodegradation rate of the composting mixture of municipal waste with the paper content of 60–70%. The moisture content and air-filled porosity are important biodegradation parameters during composting processes, and they were studied by many researchers (after Mohee and Mudhoo 2005, Eftoda and McCartney 2004). Ahn et al. (2008) used the OxiTop® for investigation of susceptibility to biodegradation of materials applied as covers (i.e. envelope materials) for composting piles in mortality composting.

This paper presents the principle of respiration test with a pressure sensor and the application of the modified OxiTop® respirometer for composting studies under laboratory conditions to determine the effects of selected parameters, such as: moisture content, composition of composting mixtures and air-filled porosity on the oxygen uptake rate during a 2-day respiration test.

Materials and methods

Materials

In order to test the applicability of the modified OxiTop® respirometer for laboratory studies on composting the following substrates were used: apple pomace (AP), woodchips (WC) and composting mixtures: AP:WC (1:0.5), AP:WC (1:1), AP:WC (1:1.5) at target moisture contents of 65%, 70% and 75%. For the substrates and composting mixtures: real moisture content (MC), bulk density (ρ), mechanical strength (MS) and air-filled porosity (i.e. free air space – FAS throughout the article) were determined with the methods described in the literature (Richard et al. 2004, Malińska 2012, Malińska and Zabochnicka-Świątek 2013).

Principle of a respiration test with a pressure sensor

The respiration test with the OxiTop®-C system uses a pressure sensor that allows for the determination of a pressure drop in gaseous phase (ambient air) in a closed vessel at constant temperature. The standard OxiTop® system consists of a measuring unit – a respirometer. The measuring unit consists of a 1-L glass bottle with an attached measuring head with a pressure sensor and a data logger, an insert with NaOH pellets (OxiTop®-C WTW, Weiheim, Germany), and a hand held remote controller (OxiTop® OC 110, Weiheim, Germany) (WTW). The system allows for the determination of the oxygen respiration rate and carbon dioxide evolution based on a pressure drop measurement. The pressure drop is recorded in time increments during the respiration test. A typical pressure drop during a 2-day respiration test is presented in Figure 1.

At the beginning of the test, the gaseous phase consists of ambient air. Variations in the pressure during the first 2–3 h can result from differences between the temperature of a sample and

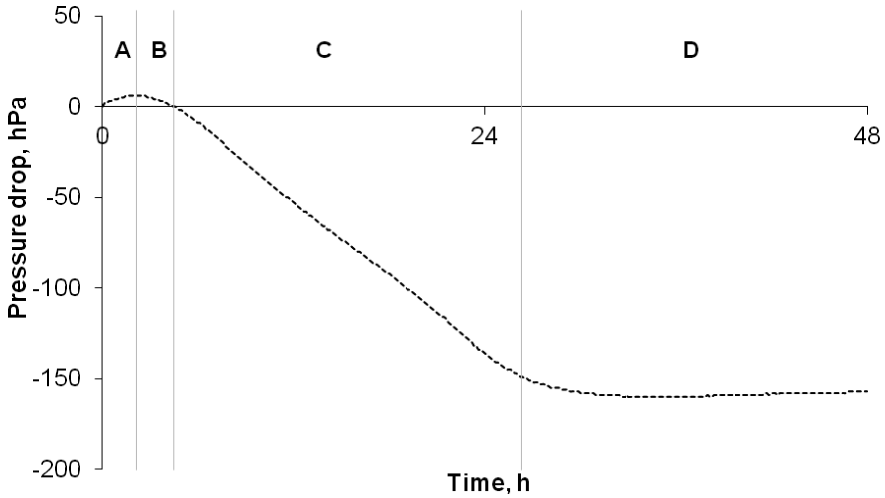

Fig. 1. A typical pressure drop during a 2-day respiration test

an incubator (phase A) and a lag period when microorganisms adapt to new conditions (phase B). After reaching the temperature equilibrium and completing a short adaptation period, the pressure drop reaches a quasi-steady state. During biodegradation of organic matter microorganisms use oxygen from the gaseous phase and produce carbon dioxide. Carbon dioxide is removed from the gaseous phase by absorption on NaOH pellets, and thus the pressure drop reflects the consumption of oxygen. The pressure drop is recorded with the data logger. The pressure drop in the vessel is directly related to the oxygen uptake rate (phase C) due to the fact that carbon dioxide produced during biodegradation is absorbed by NaOH pellets. The pressure drop is observed till depletion of oxygen in the gaseous phase leading to the limitation of aerobic microorganisms activity (phase D) (Veeken et al. 2003).

The oxygen uptake rate or carbon dioxide evolution can be determined by the relationship between the number of moles of a substance and a pressure drop from the ideal gas equation:

$$\Delta P = \frac{nR\Delta T}{V} \quad (1)$$

where:
ρP – pressure drop (kPa),
n – number of moles of the substance (kmole)
ΔP – change in the gas temperature (ºK)
R – general gas constant (8.134 kJ/mole· ºK)

$$\Delta n = \frac{\Delta PV}{RT} \quad (2)$$

where:
Δn – change in the number of moles of the substance (kmole)
T – gas temperature (ºK)

Equation 2 can include the number of moles based on the mass of the gas and the molecular weight:

$$\Delta m = \frac{\Delta PVM}{RT} \quad (3)$$

where:
Δn – change in the mass of substance (kg)
Δn – molecular weigh of the substance (32 kg/kmole for O_2, 44 kg/mole for CO_2)

From the slope of the linear phase C in the quasi--steady state region (Fig. 1) the oxygen uptake rate expressed in mg O_2/gVS^{-1}d^{-1} can be calculated from the following formula (Ahn et al. 2005, Ahn et al. 2008):

$$O_2 = \frac{\Delta P(\mathrm{hPa}) \times 100\left(\frac{\mathrm{Pa}}{\mathrm{hPa}}\right) \times 1\left(\frac{\mathrm{N}}{\frac{\mathrm{m}^2}{\mathrm{Pa}}}\right) \times V(\mathrm{m}^3) \times 32\left(\frac{\mathrm{g}}{\mathrm{mol}}\right) \times 1000\left(\frac{\mathrm{mg}}{\mathrm{g}}\right)}{8.314\left(\frac{\mathrm{J}}{\mathrm{mol} \times {}^{\circ}\mathrm{K}}\right) \times 1\left(\frac{\mathrm{N} \times \mathrm{m}}{\mathrm{J}}\right) \times \frac{\mathrm{t(h)}}{24\left(\frac{\mathrm{h}}{\mathrm{d}}\right)} \times W(\mathrm{g}) \times \left(\frac{100 - \mathrm{MC}}{100}\right) \times \frac{VS}{100}} \quad (4)$$

Also, the obtained pressure drop can be used to calculate the carbon dioxide evolution CO_2-C expressed in mg CO_2-C/gVS^{-1}d^{-1} from the following formula (Ahn et al. 2005, Sadaka et al. 2006, Ahn et al. 2008):

$$CO_2 - C = \frac{\Delta P(hPa) \times 100\left(\frac{Pa}{hPa}\right) \times 1\left(\frac{N}{\frac{m^2}{Pa}}\right) \times V(m^3) \times 44\left(\frac{g}{mol}\right) \times 1000\left(\frac{mg}{g}\right) \times \frac{12}{44}\left(\frac{gC}{gCO_2}\right)}{8.314\left(\frac{J}{mol \times {}^{\circ}K}\right) \times 1\left(\frac{N \times m}{J}\right) \times T({}^{\circ}K) \times W(g) \times \left(\frac{100 - MC}{100}\right) \times \frac{VS}{100}} \quad (5)$$

where:
ΔP – difference between the initial pressure and final pressure (hPa)
V – volume of the vessel (m^3)
T – incubation temperature (°K)
W – weight of the sample (g)
MC – moisture content (%)
VS – volatile solids (%)

Modification of the OxiTop® respirometer

Modification of the OxiTop® respirometer included replacement of a 1-L glass bottle for a 1.9-L wide mouth glass vessel equipped with a twist-off vessel lid with an attached measuring head. For the investigation of selected composting parameters, i.e. bulk density and air-filled porosity, on the respiration rate, the glass vessel was equipped with a steel mesh cylinder (diameter of 5 cm, height of 10 cm) with the open surface area of 56.2%. The cylinder was equipped with a lid and a rod that allow for compaction of the investigated material to a required bulk density. In addition, rubber rings were placed below and above the vessel lid to assure that the vessel is sealed to prevent from leaks.

Respiration test procedure

Preincubation of the samples

As substrates were stored at different temperatures (e.g. apple pomace was stored at -20°C), they were subjected to preincubation prior to the respiration test. In order to shorten the lag period (phase B) for microorganisms to adapt to new conditions the investigated substrates and composting mixtures were incubated in open plastic bags at 30°C for 48 h (Sadaka et al. 2006, Ahn et al. 2005). The samples were weighed daily and supplemental water was added in order to maintain the target moisture contents.

Incubation

The samples of the investigated materials (about 100–150 g) were transferred to the cylinders and compacted to the selected bulk densities that corresponded to a range of air-filled porosities. To remove carbon dioxide from the gas phase 6–7 NaOH pellets were used (the number of pellets was determined based on the preliminary tests). The samples were incubated in the respirometers for 48 h at 30°C (Sadaka et al. 2006, Ahn et al. 2005). Each respiration test was carried out in 3 replications.

Pressure drop measurement and data transfer

The frequency of pressure drop measurements depended on the test duration: for a 2-day test the change in the pressure drop was recorded every 8 minutes. The data were transferred from the measuring head through the infrared interface to the hand held remote controller (OxiTop® OC 110, Weiheim, Germany), and

then uploaded to the computer and read-out with the Achat OC software (version 2.03) included in the OxiTop® system. The Achat OC software allows transferring data to MS Excel for calculations of oxygen respiration rates. The built-in memory in the hand held remote controller allows for collection of up to 360 data sets depending on the test duration and presentation of data in the form of plots.

Results

Characteristics of substrates and composting mixtures

The initial properties, i.e. moisture content, bulk density, mechanical strength and air-filled porosity of substrates and composting mixtures are presented in Table 1.

The values of air-filled porosity and moisture content of the investigated materials selected for the respiration test are presented in Table 2. Apple pomace and prepared composting mixtures were subject to compaction to achieve 4 different air-filled porosities. For apple pomace with moisture content of 81.2% the investigated air-filled porosities corresponding to different depths in a composting pile were as follows (not included in Table 2): 27.2%, 17.1%, 8.0% and 2.3%. As woodchips showed the same air-filled porosity across a 2 m high pile, the respiration test was performed for the air-filled porosity of 76.9%.

Results of the OxiTop® respiration test

The OxiTop® respiration test is a static method which means that oxygen in the ambient air in the closed vessel is a limiting factor – after a period of time it is depleted. Due to this the pressure drop was determined after 15 h from the beginning of a 2-day test. For calculations of the oxygen uptake rate the pressure values were read-out at the 3rd h (after completion of the adaptation phase) and at the 15th h of the test.

Figure 2 shows a typical pressure drop during the 2-day respiration test for: (1) the mixture of AP:WC (1:0.5) at MC=77.7% and selected air-filled porosities (FAS), i.e. 21.3%,

Table 1. The initial physical properties of the investigated substrates and composting mixtures

Materials	MC (%)		ρ (kg/m^3)	MS (N/m²)	FAS (%)
	target	real			
Substrates					
Apple pomace (AP)	–	81.2±0.2	793	–	29.6
Woodchips (WC)	–	14.5±0.8	343	1057993	76.9
Composting mixtures					
AP:WC 1:0.5	65	–	–	–	–
	70	71.7±1.0	628	8469	43.9
	75	77.7±0.5	796	1694	27.1
AP:WC 1:1	65	64.2±1.6	598	32418	48.3
	70	68.6±1.0	680	16907	40.1
	75	74.6±1.0	842	6863	23.9
AP:WC 1:1.5	65	67.7±0.9	646	14527	43.3
	70	73.3±1.9	783	6225	29.6
	75	76.8±1.0	899	3710	18.1

Table 2. Selected air-filled porosities of composting mixtures for the respiration test

Materials	FAS (%)		
	MC=65%	MC=70%	MC=75%
Composting mixtures			
AP:WC 1:0.5	–	36.0	21.3
	–	31.6	15.8
	–	25.6	8.5
	–	19.6	1.1
AP:WC 1:1	46.9	36.8	22.3
	43.2	32.4	16.8
	38.2	26.6	9.6
	33.3	20.7	2.4
AP:WC 1:1.5	46.1	35.6	21.6
	42.3	31.1	16.1
	37.3	25.1	8.8
	32.3	19.1	1.5

15.8%, 8.5% and 1.1%, and (2) substrates, i.e. apple pomace at MC=81.2% and FAS=27.2% and woodchips at MC=14.5% and FAS=76.9%.

Figure 2 shows pressure drop plots for the composting mixture AP:WC (1:0.5) at moisture content of 77.7% for air-filled porosities of 21.3%, 15.8%, 8.5% and 1.1%. It was observed that at the highest air-filled porosity (21.3%) microorganisms used air faster (the oxygen partial pressure drop in time) showing increased activity. The decrease in air-filled porosity to 1.1% resulted in a significant reduction of oxygen consumption during the process. In all cases, in the phase C the pressure drop with time was linear till oxygen depletion in the vessel. In consequence, aerobic processes were inhibited. For other composting mixtures the pressure drop showed a similar trend as depicted in Figure 2. From the difference in pressure drops during the respiration test the oxygen uptake rate was calculated for apple pomace and composting mixtures at selected moisture content and air-filled porosities (Table 3). For the obtained values of the oxygen uptake rate the regression equations were provided.

Discussion

The application of the modified OxiTop® respirometer for laboratory composting studies allowed for the determination of the effect of different moisture contents and air-filled porosities on the oxygen uptake rate. The values of oxygen uptake rate determined for apple pomace and composting mixtures were typical for materials during the first two weeks of composting (Veeken et al. 2003, Jędrczak and Haziak 2005) and were increasing with the increase of air-filled porosity. However, the highest oxygen uptake rates were observed for apple pomace at air-filled porosity in the range of about 17–27% and for composting mixture of AP:WC (1:0.5) at moisture contents of 71.7% and 77.7%, and air-filled porosities in the range of about 1–36%. For other mixtures the highest oxygen uptake rates were observed for AP:WC (1:1) and AP:WC (1:1.6) mixtures at the moisture content of about 65% (real values of 64.3% and 67.6%) corresponding to the range of air-filled porosity of 32–46%. The remaining mixtures showed a significant decrease in the oxygen uptake rate with the reduction of the air-filled porosity due to compaction and elevated moisture contents. Higher values of the oxygen uptake rate for apple pomace and composting mixtures with lower contribution of woodchips resulted from differences in susceptibility of these materials to biodegradation. Apple pomace due to high moisture and organic matter contents belongs to easily biodegradable materials whereas woodchips show very little susceptibility to biodegradation. The oxygen uptake rates for apple pomace (no compaction) were of 15.1±1.0 mg $O_2/gVS^{-1}d^{-1}$ and for woodchips of 0.7±0.1 mg $O_2/gVS^{-1}d^{-1}$. In consequence, higher contribution of woodchips in composting mixtures AP:WC (1:1 and 1:1.5) can result in lower oxygen uptake rates due to less susceptibility to biodegradation and limitation of active substrate surface for microorganisms. The obtained results confirmed the observations of other authors. Jeris and Regan (1973) and Moher and Mudhoo (2005) observed that the most beneficial air-filled porosity ranges from 30 to 60% and the moisture content should not exceed 65–68%. The recommended values of the air-filled porosity was obtained for composting mixtures AP:WC (1:1) and AP:WC (1:1.5) at the moisture content of about 65%. The lower oxygen uptake rate was observed for composting mixtures AP:WC (1:1 and 1:1.5) at the air-filled porosity below 30–35% and the moisture content exceeding 65–68%.

Conclusions

The modified OxiTop® respirometer can be applied for laboratory composting studies. The proposed modification allows for the determination of the effects of different air-filled porosities on the oxygen uptake rate during the respiration test. Due to diverse susceptibility to biodegradation of composting materials the effect of selected air-filled porosities can be determined for composting mixtures with the same ratio of substrates in the mixture. The time of the respiration test with the OxiTop® system depends on the available oxygen in the ambient air in the closed vessel. Therefore, the increase of the volume of the vessel can extend the time of the respiration test. Also, the application of the

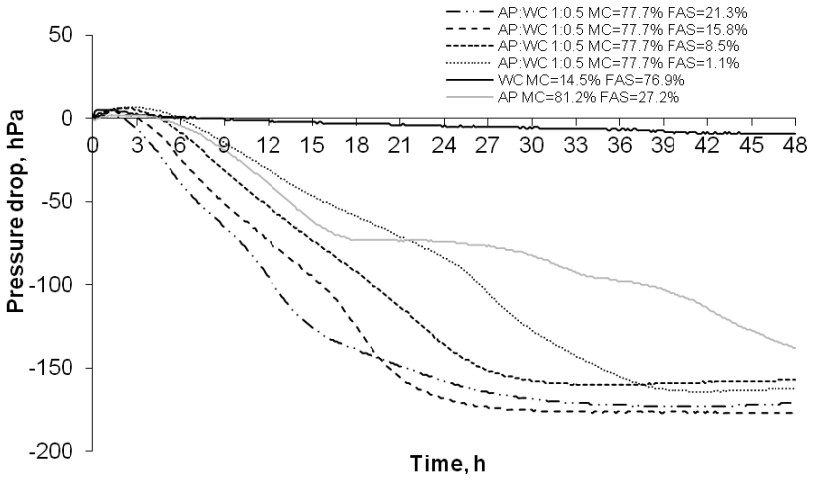

Fig. 2. An example of the pressure drop during the 2-day respiration test for the composting mixture of AP:WC (1:0.5) at MC=77.7% and FAS=21.3%, 15.8%, 8.5% and 1.1%, apple pomace at MC=81.2% and FAS=27.2% and wood chips at MC 14.5% and FAS=76.9%

Table 3. Oxygen uptake rate calculated for the investigated materials at selected moisture content and air-filled porosities

Substrates	MC (%)	FAS (%)	OUR (mg O_2/gVS^{-1}d^{-1})	Regression equation
AP	81.2±0.2	27.2 17.1 8.0 2.3	15.7 ±1.0 13.7±2.0 7.1±0.7 4.9±1.3	y = -0.0107x²+0.7804x+2.6419 R²=0.9711
AP:WC 1:0.5	71.7±1.0	36.0 31.6 25.6 19.6	17.3±0.5 17.1±1.9 16.5±1.0 14.1±2.1	y = -0.0177x²+1.1684x-1.9931 R²=0.9860
	77.7±0.5	21.3 15.8 8.4 1.1	16.9±1.6 15.4±0.5 11.1±1.8 7.4±0.8	y = -0.0073x²+0.65x-6.555 R²=0.9923
AP:WC 1:1	64.2±1.6	46.9 43.2 38.2 33.3	14.6±0.9 14.2±1.5 13.6±1.7 14.3±0.8	y = 0.0138x²+1.0755x-34.675 R²=0.8740
	68.6±1.0	36.8 32.4 26.6 20.7	7.8±1.4 8.8±0.7 8.4±0.2 4.2±0.2	y = -0.0449x²+2.7898x-34.208 R²=0.9904
	74.6±1.0	22.3 16.8 9.6 2.4	9.3±0.8 7.5±1.1 3.7±2.0 4.4±0.6	y = 0.0194x²+0.2045x-4.5793 R²=0.9300
AP:WC 1:1.5	67.7±0.9	46.1 42.3 37.3 32.3	13.2±0.6 12.1±0.2 11.5±0.6 11.5±0.1	y = 0.0157x²+1.1123x-31.08 R²=0.9978
	73.3±1.9	35.6 31.1 25.1 19.1	8.8±0.3 8.9±0.2 8.0±0.6 5.9±0.9	y = -0.0169x²+1.0953x-8.7971 R²=1.0000
	76.8±1.0	21.6 16.1 8.8 1.5	7.6±0.1 6.7±0.6 5.3±0.4 4.7±0.5	y = -0.0042x²+0.0529x-4.603 R²=0.9900

modified OxiTop® respirometer for laboratory composting studies requires preincubation (for 2 days at 30°C) as well as adjustment of the moisture content in the investigated materials and the amount of NaOH pellets (depending on the initial mass of the investigated material).

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Wykorzystanie zmodyfikowanego systemu OxiTop® do laboratoryjnych badań procesu kompostowania

Streszczenie: W przedstawionych badaniach wykorzystano zmodyfikowany system OxiTop® do wyznaczenia szybkości oddychania w oparciu o zużycie tlenu podczas 2-dniowego testu respirometrycznego dla wybranych materiałów przeznaczonych do kompostowania o różnym składzie mieszanek, zawartości wody i porowatości. Modyfikacja systemu OxiTop® dotyczyła dostosowania szklanego naczynia oraz jego wyposażenie w cylinder w kształcie walca o średnicy 5 cm i wysokości 10 cm, wykonanego z siatki stalowej (oczka siatki stanowiły ok. 56,2% całkowitej powierzchni). Ta modyfikacja pozwoliła na uzyskiwanie różnych gęstości nasypowych (a tym samym porowatości) badanych materiałów w skali laboratoryjnej. Test respirometryczny został przeprowadzony dla wytłoków jabłkowych i mieszanek wytłoków jabłkowych i ścinek drzewnych w stosunku 1:0,5, 1:1, 1:1,5 (s.m) i zawartości wody 60%, 65% i 75% oraz porowatości w zakresie od ok. 46% do ok. 1%. Z uwagi na różną podatność badanych mieszanek na biodegradację, możliwe było określenie wpływu zmian porowatości powietrznej (spowodowanych kompakcją) na szybkość zużycia tlenu dla mieszanki o stałym udziale czynnika strukturotwórczego. Przedstawiona metoda pozwala na laboratoryjne badanie wypływu zawartości wody i kompakcji na biodegradację podczas kompostowania.