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Design, development and evaluation of novel equipment for compost stability tests

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Abstract. Compost stability is one of the essential criteria to evaluate the quality of organic fertilisers, and this parameter is often determined according to oxygen consumption (OC) test. In this study, novel devices were developed to assess compost stability based on OC measurement through pressure and temperature monitoring. The invented devices could operate well in the pressure range of -160 - 160 mbar and temperature range of -10 - 85 ^{o}C with a flexible data acquisition frequency from 1 minute/data to 1 day/data, and all data was saved on a microSD card. The devices were then applied to monitor the OC of two different types of compost samples (food waste and green compost) over 7 days. The results showed that the home-made devices operated stably throughout the test period, and the respiration activity (AT_4) calculated from the data obtained from the device was comparable to those obtained from the commercial device (Oxitop). Between the two types of samples, the compost based on food waste had higher AT_4 and infinite OC (OC_{max}) values but with lower kinetic constant (K) values. According to the EU regulation, both types of samples were considered stable compost.

Keywords: AT4, compost stability, organic fertiliser, oxygen consumption, respirometry.

Classification numbers: 3.1.1, 3.4.3, 4.1.1, 5.6.3.

1. INTRODUCTION

Compost is a product obtained from the biological decomposition of organic compounds from biomass, primarily under aerobic conditions [1]. The demand for compost is increasing with the expansion of organic farming, where chemical fertilisers are not utilised. Compost can be used as an organic fertiliser to supplement nutrients for the soil, especially since it is a good carbon source [2].

Evaluating compost quality is necessary before use, in which stability and maturity are essential characteristics [3]. While assessing maturity is complicated, involving a series of measurements related to promoting germination and growth of crops, stability is a more straightforward parameter and reflects the activity level of microorganisms in compost. A compost has high stability when the activity of microorganisms in it is low and vice versa. Stability is usually reported according to oxygen consumption (OC, mg O_2/g dry compost) or CO_2 production rate (mg CO_2/g dry compost). Therefore, respirometry is the most common method to assess the activity of microorganisms in compost and is the most suitable method to determine compost stability [4].

There is a biochemical similarity between the compost stability and biochemical oxygen demand (BOD) test, where microorganisms consume organic compounds and release CO_2 and water. While BOD_5 (total OC after 5 days) is used to estimate BOD characteristics of a water sample, AT_4 (total OC after 4 days) is utilised to evaluate compost stability [5]. According to the EU regulation, a compost sample is stable when its AT_4 value is below 10 mg/g [6, 7]. The difference between the operation of BOD and compost stability measurements lies in the sample state: compost stability tests are applied to solid samples, while liquid samples are used in BOD experiments. This distinction leads to differences in equipment design. While OC in BOD measurements can be monitored in both liquid (monitoring dissolved oxygen) and gas phases (observing pressure decrease), typically AT_4 tests only apply to oxygen in the gas phase and can be performed based on the measurement principle of pressure (e.g., Oxitop devices) or electrochemistry (e.g., Sapromat devices) [8-10]. The dissimilarities between the results obtained when applying these two methods (using Oxitop and Sapromat devices) for different samples were manageable. However, the values obtained from Oxitop devices were usually lower, as pointed out in the study by Binner *et al*. (2012) [8]. It is a fact that although measures of pressure depletion can be conducted more simply and at a lower cost, there are certain design limitations in Oxitop devices that can be improved to enhance accuracy.

In an Oxitop device, the compost sample is placed at the bottom of the bottle while the CO_2 absorption part (usually KOH solution or solid) is kept suspended in the bottle. This design leads to several disadvantages: i) The position of KOH makes CO_2 absorption more difficult due to the limitations of the contact area and the inability to stir; ii) For samples with significant OC, the amount of oxygen in the bioreactor may need to be increased, and in this case, manually opening the bottle cap to provide additional air will make the measurement process inconvenient; and iii) The biodegradation of organic materials (in unstable compost) may involve temperature changes, and placing a temperature sensor at the top of the bottle may not accurately reflect these temperature changes.

The reality requires the development of a new device with similar features (determining OC by monitoring pressure depletion) that overcomes the design limitations of the Oxitop devices. However, research on developing equipment to evaluate compost stability is relatively limited [11], mainly focusing on the composting process and parameters related to maturity [12 - 14]. Therefore, this study was conducted to develop a new prototype measuring OC based on observing pressure variation. The specific features of this new device were determined as follows: 1) Using an alkali solution combined with magnetic stirring to absorb CO_2, and the sample was stored in a container placed in the middle of the bioreactor; 2) It could automatically supply more oxygen when the pressure in the bottle drops to a preset value; 3) Each device was capable of measuring two compost samples simultaneously; 4) In addition, the measurement frequency could be flexibly changed according to the user's purpose (from 1 minute to 1 day per data) and data was directly recorded onto a microSD card.

2. MATERIALS AND METHODS

2.1. Materials and chemicals

Electronic components used to develop the device included a microcontroller (ATMEGA2560-16AU, Microchip Technology Inc., USA), pressure sensor (HSCSAAN160MDAA5, Honeywell, USA), temperature sensor (Sensirion AG, Switzerland), 20x2 character LCD (Newhaven Display Intl., USA), 24 bit analog-digital converter (ADC) (ADS1256, Texas Instrument, USA), 16 GB microSD card (Samsung, Korea), and 12 VDC power supply (Tplink, China). A 1 L glass bottle (Duran, Germany) was used as the reaction vessel. The bottle cap was made from polyoxymethylene (POM) with a sample container of 304 stainless steel. Solid KOH (98 %, Samchun, Korea) was used to prepare the solution for absorbing CO_2 in the tests.

2.2. Design of the device

The design of a device to measure OC for evaluating the compost stability was made of three parts: the bioreactors, the electronic box, and the oxygen supply system, which allowed the measurement of two samples simultaneously. The bioreactors included two transparent wide-mouth glass bottles with a capacity of 1 L, each with a cap made of POM material. On each cap, there were positions to attach two gas fittings, a pressure sensor, and a sample container. The sample container was made of stainless steel to avoid corrosion in the highly alkaline environment and consisted of at least two concentric trays with a thickness of 3 mm, a diameter of 60 mm and a height of 10 mm. The difference between the two trays was that the upper tray had small holes with a diameter of 1 mm to increase the contact area between the sample and the gas phase. The lower tray was used to collect solid or liquid substances from the upper tray, avoiding contaminating the KOH solution below. In addition, the number of upper trays could be increased, and the lower tray could also be utilised to contain more samples if needed. Each tray could hold 10 to 15 g of sample with the designed size.

The electronic box was where the data from the sensors were recorded and processed and included the following main components: two pressure sensors, a microcontroller, an ADC, a real-time chip, a microSD card, an LCD screen, two solenoid valves, a power supply, and connection position for temperature sensors, pressure sensors and solenoid valves. The block diagram of the electronic components was illustrated in Figure 1. All these parts were neatly placed in a steel box with a length × width × height of 250 mm × 180 mm × 100 mm.

Finally, the oxygen supply system was responsible for supplementing oxygen to the bioreactors and consisted of two multi-layer gas bags with a metal coating to prevent gas leakage. Before each test, these two gas bags were filled with air or oxygen. During the measurement, if the pressure in the bioreactors drops below a predetermined value, -150 mbar corresponding to a 75 % reduction in oxygen in the bottle for example, the solenoid valves will open, and oxygen will flow from the gas bag into the bottles to compensate for the oxygen consumed.

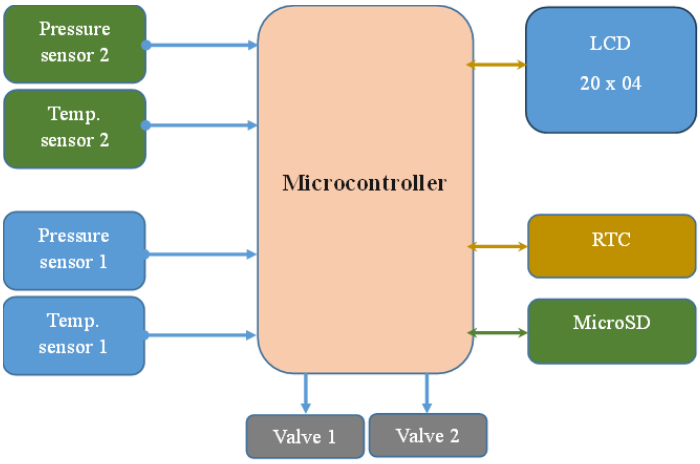


Figure 1. Block diagram of electronic components in the new device for measuring compost stability.

2.3. Demonstration of device performance in the laboratory

Two types of compost samples were used in this research: green compost (sample type 1) and waste food compost (sample type 2). All samples were collected from IDELUX company (Habay, Belgium). At the time of collecting (February 2022), the sample type 1 was kept at the company for 7 months, and the sample type 2 was held for 3 months.

Each sample type was measured in duplicate, with the mass in each test being about 15 grams. KOH solution of 1.0 mol/L (250 mL per bottle) was used to remove CO_2 gas. The amount of KOH was always in excess in each test, and the KOH solution was put at the bottom of the bottle and well mixed by a magnetic stirrer system. The cross-checking test was done with a similar amount of sample type 1 as well as KOH in solid form and measured with an Oxitop device under the same conditions. All the bottles were put inside a plastic box filled with water to reduce temperature fluctuations. Besides, the data acquisition frequency of the developed devices was set at 5 min/data.

2.4. Software and data processing

The OC was calculated according to the following equation:

$$OC = \frac{\Delta m_{O_2}}{m_{\text{compost}}} = \frac{M_{O_2} \times \Delta n_{O_2}}{m_{\text{compost}}} = \frac{M_{O_2} \times V_g \times \Delta P}{R \times T \times m_{\text{compost}}} \qquad (1)$$

in which: OC: oxygen consumption (mg-O_2/g compost); m_{compost}: mass of dry compost (g); M_{O_2}: molar mass of oxygen = 32 g/mol; Δn_{O_2}: change in mole of oxygen (mmol); V_g: volume of gas phase in the bottle (mL); ΔP: change in pressure in the bottle (mbar); R: gas idea constant = 83.14 (mbar.mL/mmol/K); T: temperature (K).

According to equation (1), for each measured value of ΔP and T over time, the amount of oxygen consumed corresponding to each time point was calculated (other parameters were kept constant). The OC data were then smoothed by Savitzky – Golay method with seven points (SG-

7) [15]. The AT_4 was determined by the sliding method, as described by Binner *et al*. (2012) [8]. Besides, the infinite OC (OC_{max}) and the kinetic constant (K) values were calculated based on the first-order kinetic fitting model with the SG-7 data in 4 days (without lag phase). All calculations were done with Excel 365 (Microsoft, USA) and Origin Pro 2022 (OriginLab, USA).

3. RESULTS AND DISCUSSION

3.1. Prototype development

The design of the printed circuit board of the electronic components was illustrated in Figure 2A, while the front and back views of the electronics box after fabrication were shown in Figures 2B and 2C. An image of a complete device was presented in Figure 3. When the device was turned on, the microcontroller read the setup parameters from the memory card. Then, the microcontroller collected the values from the sensors in bottle 1 (temperature sensor 1, pressure sensor 1), bottle 2 (temperature sensor 2, pressure sensor 2), time from the RTC module and wrote the data to the memory card. If the pressure value in a bottle (1 or 2) was lower than the preset threshold, valve 1 or valve 2 was opened to allow oxygen gas from the gas bag to enter the bioreactor. At the same time, the values read from the sensors and date-time were also displayed on the LCD screen. After a preset time, the process was repeated until the experiment was completed and the user turned off the power. The preset parameters were stored in a text file (setup.txt) on the memory card, and the data were written to another text file (DATA.txt), as illustrated in Figure 4.

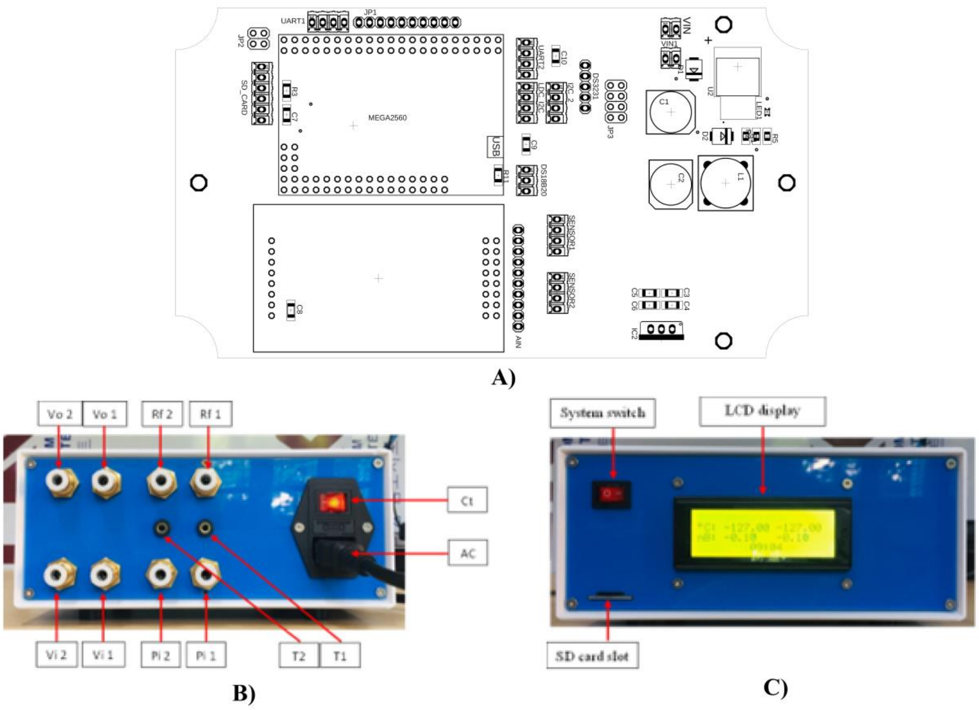
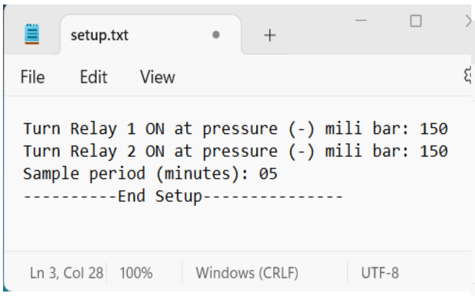


Figure 2. Image of A) the design of the printed circuit board, B) the back view and C) the front view of the electronic box.

In Oxitop devices or previously home-made devices, each bottle (measuring one sample) required its accompanying electronic system [16, 17]. Besides, it was mandatory for these devices to be opened manually to replenish oxygen in case the initial amount of oxygen in the bioreactor was not enough for the respiration process to take place. Meanwhile, in addition to the required components for each device (pressure sensor, temperature sensor, solenoid valve), other electronic components (microcontroller, ADC converter, real-time chip, memory card) could be used for multiple devices at the same time without affecting the operation and accuracy of the devices. Therefore, integrating multiple electronic hardware was an appropriate development to make the most of the processing capacity of components and save space, thereby increasing measurement performance and reducing production costs. Two solenoid valves and two oxygen gas bags were added to automatically supply additional oxygen when needed. The device was developed in this study based on this approach and could measure two independent samples simultaneously using only one electronic box.

Figure 3. Image of complete composting device.

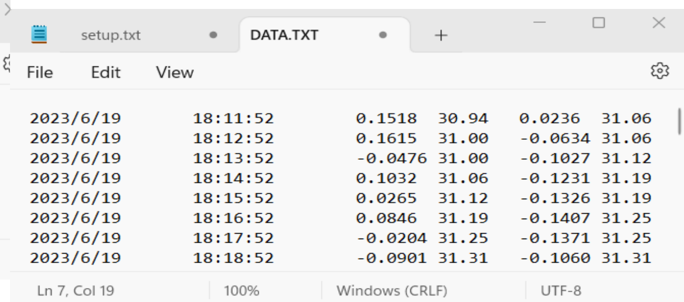


```
setup.txt
File  Edit  View

Turn Relay 1 ON at pressure (-) mili bar: 150
Turn Relay 2 ON at pressure (-) mili bar: 150
Sample period (minutes): 05
----------End Setup---------------

Ln 3, Col 28   100%   Windows (CRLF)   UTF-8
```

A)



```
setup.txt   DATA.TXT
File  Edit  View

2023/6/19   18:11:52   0.1518   30.94   0.0236   31.06
2023/6/19   18:12:52   0.1615   31.00   -0.0634  31.06
2023/6/19   18:13:52   -0.0476  31.00   -0.1027  31.12
2023/6/19   18:14:52   0.1032   31.06   -0.1231  31.19
2023/6/19   18:15:52   0.0265   31.12   -0.1326  31.19
2023/6/19   18:16:52   0.0846   31.19   -0.1407  31.25
2023/6/19   18:17:52   -0.0204  31.25   -0.1371  31.25
2023/6/19   18:18:52   -0.0901  31.31   -0.1060  31.31

Ln 7, Col 19   100%   Windows (CRLF)   UTF-8
```

B)

Figure 4. Illustration of A) setup.txt file and B) DATA.txt file.

3.2. Application of developed devices to evaluate compost stability

The changes in pressure and temperature during the tests are illustrated in Figure 5, and the OC of two duplicate samples is shown in Figure 6. It is clearly shown in Figure 5 that, for both types of samples, the rapid decrease in pressure in the bottle corresponded to a fast accumulation in the OC during the first 40 hours, and then there was a slight variation for the next 5 days. The maximum depletion of pressures observed with the replicates of sample type 1 and sample type

2 was about -32 and -52 mbar, respectively, both recorded on the second day of the experiment. This observation suggested that sample type 1 had a higher OC rate than sample type 2. The experiment's temperatures ranged from 18 to 21 °C with quite clear fluctuations between day and night, especially at the beginning and end of the test. These temperature changes led to fluctuations in pressure as pressure and temperature were proportional according to the ideal gas equation. In further studies, a thermostat will be required to keep temperature variations low, similar to the incubator for BOD measurements.

For each duplicated sample, the obtained OC values were similar, and the differences between AT_4 values of the two replicates were only about 15 % and 2 % for sample type 1 and sample type 2, respectively (Table 1), indicating that the device operated stably and accurately. The more considerable dissimilarity observed in sample type 1 might be due to the sample needing to be completely homogenised. There was no lag phase in the experiments. As shown in Figure 6, sample type 2 (food waste compost) had a higher OC with a maximum value of 4.6 mg/g compared to sample type 1 (green compost) with a maximum value of 2.8 mg/g. This was reasonable due to the compost age: sample type 1 was produced for 7 months while sample type 2 was only for 3 months. According to the EU regulation, both compost sample types are considered stable compost [7].

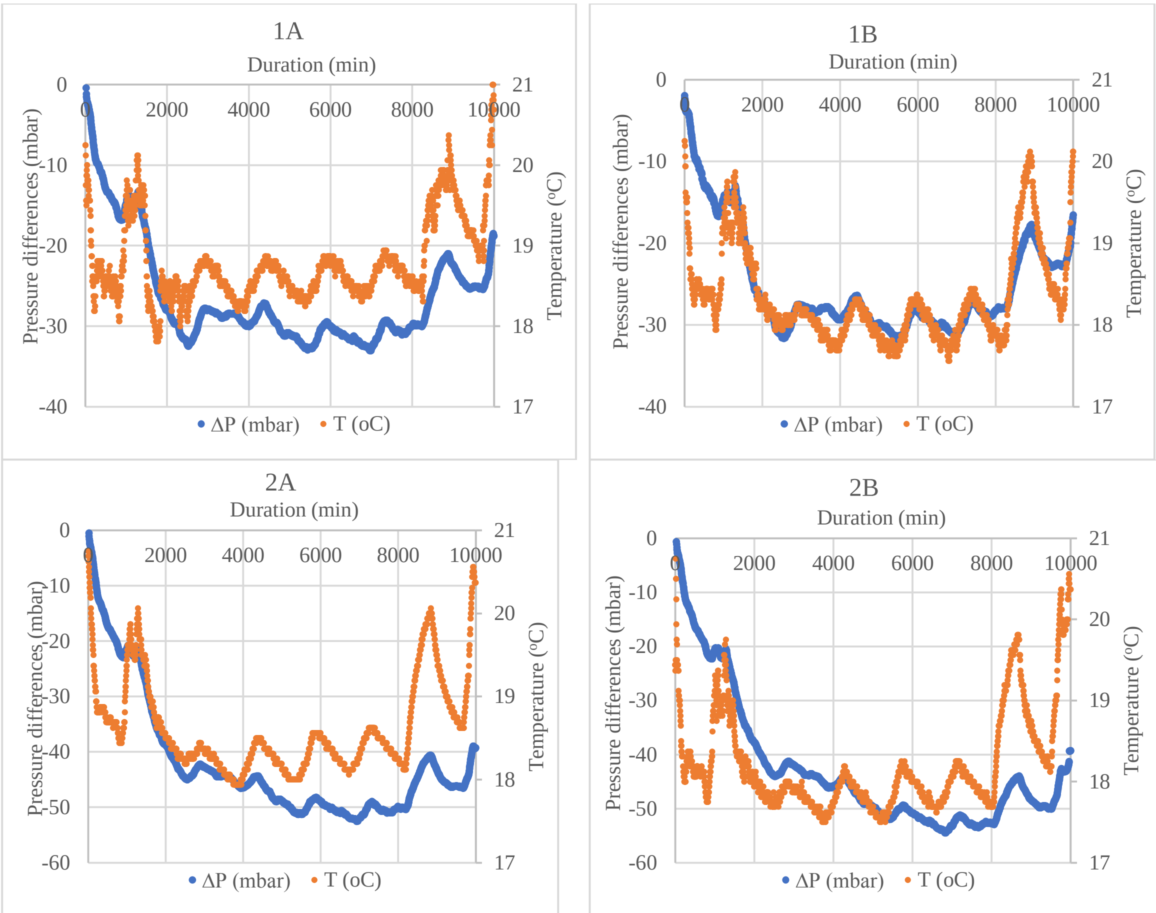


Figure 5. The variation of pressure and temperature during the tests of two duplicate samples: sample type 1 (1A and 1B) and sample type 2 (2A and 2B). The blue dots represent pressure values, and the orange dots represent temperature data.

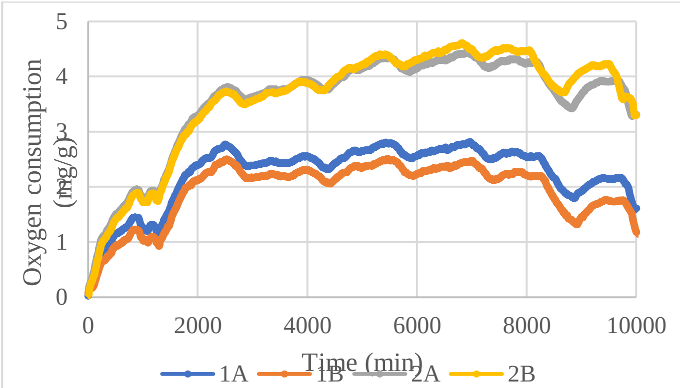


Figure 6. Comparison of oxygen consumption of four samples.

Table 1. AT_4, OC_{max} and K values of four samples.

Parameter	Sample			
	1A	1B	2A	2B
AT_4 (mg/g)	2.55	2.21	4.10	4.19
OC_{max} (mg/g)	2.65	2.41	4.26	4.34
K (hr^{-1})	0.051	0.047	0.040	0.036

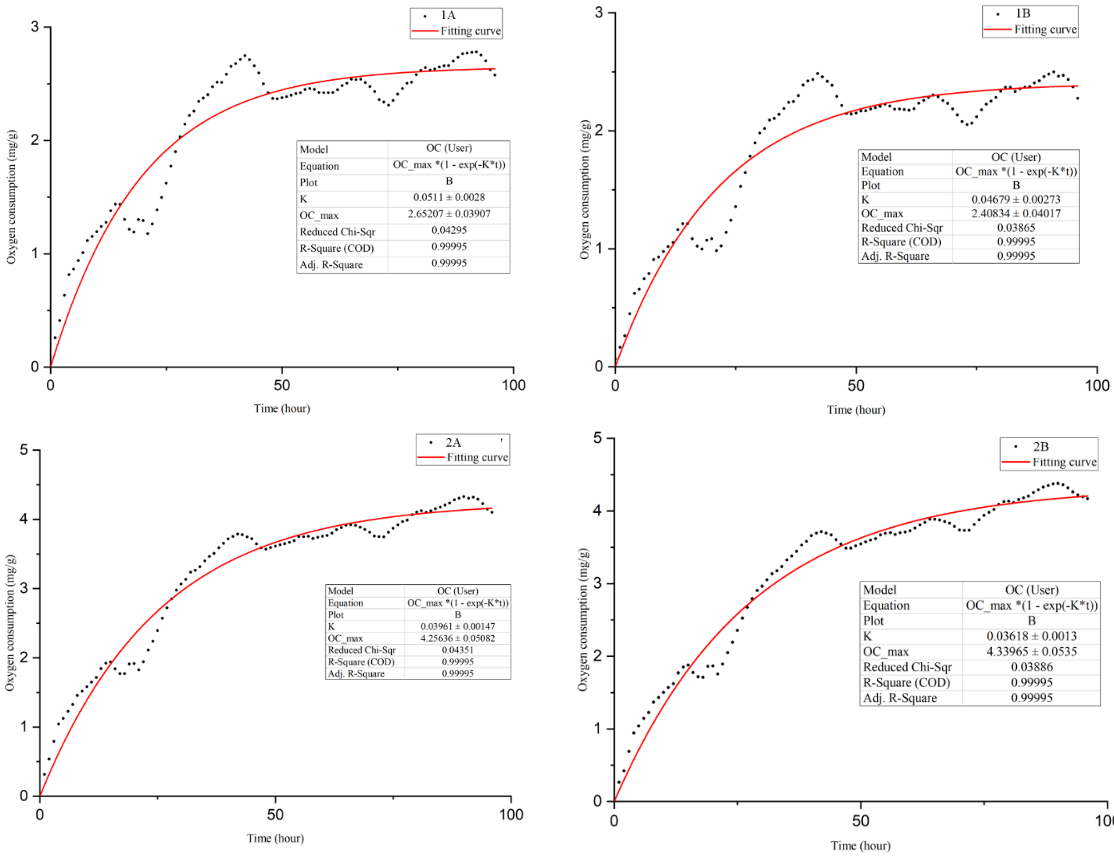


Figure 7. Fitting first-order kinetic model to the OC data of four samples.

Compared with the results obtained from the commercial Oxitop device for the first type of sample ($AT_4 = OC_{day\ 4} - OC_{day\ 0}$ = 2.35 mg/g), the AT_4 value calculated by the sliding method and the data obtained from home-made devices (average: 2.38 mg/g) were almost equivalent. It was noted that the compost sample tested was stable, therefore possible effects from design limitations of the Oxitop device were not demonstrated. However, the Oxitop device only allowed storing data per day (1 data/day) for 7 days, so the convenience was less than that of the developed devices.

The kinetic parameters were calculated based on the fitting of the first-order model, and the results obtained are shown in Table 1 and Figure 7. As can be seen in Figure 7, the OC data obtained on both types of samples fitted well according to the first-order kinetic equation, with R^2 coefficients greater than 0.999, indicating that the respiration taking place in the bioreactor followed the first-order decomposition law. The OC_{max} values were in the range of 2.65 - 4.34 mg/g, only about 3.5 - 9.1 % higher than the corresponding AT_4 values, showing that the respiration process was quite complete after 4 days of the experiment. Lag phases were not observed during the experiment, indicating that the sample preparation and experimental conditions were suitable for the aerobic respiration of microorganisms in the tested compost samples. Besides, the differences between K values of duplicate samples were smaller than 10 %. In contrast with the trend of OC_{max}, the K values of sample type 1 (green compost) were greater than those of sample type 2 (food waste compost).

4. CONCLUSIONS

In this study, novel devices with an improved design were successfully built to assess the compost stability based on monitoring the pressure depletion in the bioreactor. Each developed device was capable of measuring two compost samples simultaneously and could automatically provide more oxygen to the bioreactor. The devices were then applied to monitor the OC of two types of compost samples over 7 days. The results showed that the devices operated stably throughout the test period, and the difference in AT_4 values obtained from the home-made device and the Oxitop device was less than 2 %.

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CRediT authorship contribution statement. Nguyen Thanh Dam: Methodology, Investigation, Formal Analysis, Writing – original draft, Writing – review and editing. Nguyen Canh Viet: Methodology, Investigation, Software, Writing – original draft, Writing – review and editing. Phung Thi Vi: Conceptualization, Funding acquisition, Project administration, Writing – original draft, Writing – review and editing. Nguyen Manh Huy: Formal analysis. Ta Thi Thao: Supervision. Duong Hong Anh: Supervision. Jean-Luc Vasel: Methodology, Supervision. Pham Hung Viet: Methodology, Supervision, Writing – original draft, Writing – review and editing.

Declaration of competing interest. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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