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Standardized Determination and Classification of Stability of Organic Matter in Compost

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Abstract

The stability of organic matter is a major determinant in assessing the effect produced by composts on the quality of soil or growth substrate. Thus, stability testing for composts is important in optimizing the effect of compost applications. Operators require a quick, cheap and reliable method with which to determine the stability of organic matter in compost. A standardized method for use in the determination of the intrinsic carbon mineralization rate of organic matter sources (composts, manures, etc.) was developed by means of an aerobic incubation in a suspension of the sample in a nutrient solution. The method avoids the drawbacks associated with solid state stability tests, in which the incubation conditions such as pH, aeration and moisture availability, limit the organic matter decomposition rate. The method thus reflects the intrinsic properties of the organic matter. The use of this method is not limited to advanced laboratories as the user-friendly equipment also enables application of the method by other groups such as compost producers (to monitor and optimize their composting process) and end-users (to assess compost quality). The new test method is currently being implemented by several commercial laboratories as standard test method. It has moreover recently been proposed for use in the EC-HORIZONTAL working group "stability" (CEN/TC 223 on soil improvers). Based on stability values for a range of different composts and various other organic matter sources, a classification with four stability classes for composts is proposed. The test method has also been standardized to determine the N-mineralization potential of organic matter sources as well as the biochemical methane potential of organic substrates applied as renewable energy sources.

1 Introduction

Organic matter (OM) sources such as crop residues, manures and composts are often added to soils and soil-less potting mixes to enhance soil or substrate quality by supplying plant nutrients, increasing natural suppressiveness of the soil

against plant pathogens and improving physical and chemical soil characteristics such as porosity, cation exchange capacity and water holding capacity (Veeken et al., 2005). However, potential risks are associated with the application of OM inputs. Possible negative effects include temporal oxygen depletion resulting in denitrification, stimulation of soil-borne plant pathogenic fungi (Termorshuizen et al., 2006) and nitrogen immobilization (Grigatti et al., 2007). To avoid the negative effects and maximize the positive effects of OM inputs on soil or substrate quality, application of a suitable quantity and quality of OM is of particular importance. The quality of OM is mainly determined by its stability. This stability or microbial degradability is an intrinsic property of OM and is determined by its composition, i.e. the relative amounts of the various compounds present such as cellulose, organic acids, proteins and lignin, and the spatial arrangement of the various compounds. To optimize the management of OM by farmers and land managers, quick and reliable tests are needed for use in evaluating quality of OM sources in order to be able to choose the most suitable source for a specific situation. These tests should develop adequate strategies taking into consideration not only mineral N levels present at the start of a cropping season but also the amount of N that can be expected to become available to plants through mineralization during the growing season, capable of minimizing nutrient losses and environmental damage due to NH_3 volatilization, NO_3^- leaching and N_2O emissions. With regard to the plant container industry these tests are of considerable value, leading to an increase in the use of composts to partially replace peat in potting mixes. For this industry a careful selection of high-quality composts is needed to avoid phytotoxicity and nitrogen immobilization and to optimize microbial activity in order to maximize disease suppression. For tests aimed at characterizing OM quality, both carbon and nitrogen dynamics should be considered due to the fact that (1) C and N are the main determinants of OM decomposition rate, (2) N is the most difficult to manage plant nutrient due to its many different forms (some of which are easily lost for crop production), and (3) C and N dynamics are strongly interrelated.

This paper describes the development of a standardized, reliable, quick and relatively cheap procedure that can be used for the routine testing of OM sources in terms of their C and N mineralization potential. Data are presented for the stability of a wide range of natural organic matter sources and composts and, based on these values, a classification scheme is proposed for the stability of compost.

2 Compost stability test methodology

2.1 Selection of test method

In scientific literature various definitions are in use for the evaluation of compost

$$C_cH_bO_cN_d + xO_2 + yNH_4^+ \rightarrow aCO_2 + zH_2O + \Delta H + X$$

stability. Here we define stability as the rate of microbial degradation of the organic matter under aerobic conditions. A higher stability corresponds to a lower degradation rate. The microbial degradation of organic matter ($C_aH_bO_cN_d$) can be represented by the following equation (ignoring stoichiometric coefficients):
where ΔH is the heat production and X represents produced microbial biomass. The stoichiometry of the reaction depends on the type of substrate. For a typical substrate as glucose or polysaccharides these coefficients are as follows (Haug, 1993):

- Oxygen consumption: 1.07 kg O_2/kg OM
- Carbon dioxide production: 1.47 kg CO_2/kg OM
- Water production: 0.6 kg H_2O/kg OM
- Heat production: 16 MJ/kg OM

The reaction equation shows that the degradation rate (or stability) of organic matter can be deduced from the oxygen uptake rate (OUR), the carbon dioxide production rate (CPR) or the heat production rate (HPR). By definition, readily degradable substrates are consumed first and more recalcitrant substrates are retained. This implies that the stability increases in time during composting and OUR, CPR and HPR decrease in time.

Various methods are commercially available for the determination of stability. These methods are based on determination of one or more of the parameters (O_2, CO_2 and ΔH) discussed above and are used with either a batch or continuous system. Most respirometric devices are expensive, complicated and require advanced laboratories and are, therefore, not suitable for non-advanced users. In this study we aim at the development of a simple test method using a batch system, namely the OxiTop system, a user-friendly device that will be described further in paragraph 2.3.

In several European countries the standard test for evaluation of compost stability is the Rottegrad test (self heating or Dewar test). With this method the temperature increase of a compost sample incubated in an insulated vessel is used as an indicator of stability. However, the measured temperature is not directly related to ΔH as temperature is a result of heat production and heat removal. Moreover, the Rottegrad test is performed in solid state, characterized by several disadvantages to be discussed in the next paragraph. We agree with Weppen that this method does not yield reproducible results and frequently leads to wrong results and interpretation (Weppen, 2002). Moreover, the method is not discriminative at high stability levels.

Besides the measurement of parameters that are directly related to the degradation process (O_2, CO_2 and ΔH), other parameters have also been suggested for the assessment of organic matter stability such as e.g. C/N ratio, NO_3^-/NH_4^+ ratio, CEC, humification coefficient and dissolved COD. However, these parameters are not directly related to the stability as defined previously and are highly dependent on the composition of the starting materials and conditions during the

composting process.

2.2 Standardization of the stability test

The stability (or degradation rate) of the solid substrate can be determined in the solid phase and the liquid phase. The advantages and disadvantages of both approaches will first be discussed (also see Lasaridi and Stentiford (1998)). In this section we restrict discussion to oxygen consumption (respirometry), although the same comments apply to measurement of stability according to carbon dioxide and heat production. The intrinsic stability of the solid substrate can only be assessed when optimal conditions are provided where the biodegradation of the solid substrate itself is the rate-controlling step of the respiratory activity. For this, the following conditions should be met:

- The complete surface of the solids should be accessible for aerobic degradation. This condition is only met when the solids are suspended in water.
- Availability of macronutrients and micronutrients should not be rate-limiting. This condition can be met by adding a standard macro and micro nutrient solution.
- Microbial growth should not be rate-limiting. This means that sufficient NH_4^+ should be present to meet the N-demand for microbial growth. This can be accomplished by adding NH_4Cl to the solution.
- Sub-optimal conditions such as an excessively low or high pH or presence of toxic compounds (e.g. VFA) should be prevented. To reach this the solution can be buffered.
- Initial (mesophilic) aerobic activity should be sufficient to start up the aerobic degradation. To ensure this a microbial inoculum can be added.
- Other oxygen consuming (microbial) processes should not occur. In this respect, the oxidation of ammonia (nitrification) is of particular importance, while other processes such as oxidation of sulfide and Fe(II) can be ignored. Nitrification can be suppressed by adding a nitrification inhibitor such as allylthiourea.
- Oxygen transfer from the gas phase to the liquid-solid phase should not be rate limiting.

Measurement of the stability in the solid phase is characterized by the following problems in relation to the above mentioned points:

- The surface of the solids is not fully accessible to degradation, as aggregates are present. The surface available for aerobic microbial activity depends on the moisture content and bulk density of the substrate.
- In most cases oxygen is the rate-limiting step. Oxygen is only directly available at the outer surface of the solid matrix but has to diffuse through pores to reach the inner core of the matrix. Therefore, the maximum OUR is never assessed and OUR will depend on the moisture content and density of the substrate.
- For a solid matrix, homogeneous pH buffering, addition of an inoculum and

addition of a nitrification inhibitor is problematic.

It can be concluded that measurement in the liquid (suspension) state alone is capable of providing fully standardized conditions for an aerobic respiration test. Moreover, respiration measurement in the liquid state results in higher respiration rates and thus shorter measuring times.

2.3 Determination of respiration rate by the OxiTop system

A standardized respiration method was developed at the department of Environmental Technology of Wageningen University. We found several limitations of the existing commercial OxiTop® system (WTW, Wilhelm, Germany) that were handled as follows:

- The cup for CO_2 adsorption in lye was adjusted to improve CO_2 adsorption rate;
- The lye prescribed by the manufacturer (NaOH) was hygroscopic and clogged, significantly reducing the CO_2 adsorption capacity. Therefore, NaOH was replaced by a non-hygroscopic lye to prevent clogging;
- The solution was shaken (or stirred) to prevent limitations of O_2 gas-liquid transfer;
- The O_2 gas-liquid transfer rate became limiting at higher TS levels due to blocking of the liquid surface by the solids and increased viscosity, therefore, the sample amount was standardized to an amount of 2.0 g VS equivalent;
- Nitrification inhibitor ATU (allylthiourea) had to be added at much higher concentrations compared to wastewater applications; the amount was increased 10-fold;
- The test was performed at a constant temperature as microbial degradation rate is temperature-dependent, the temperature influencing water vapor pressure, thus contributing to the total pressure of the gas phase.

Following adaptation of the device and procedures the test method was performed according to the following standard experimental conditions. An amount of milled sample material equivalent to 2 g of VS was added to a bottle together with 180 ml of demineralized water and gently mixed. Subsequently, 10 ml of a macronutrient solution was added containing NH_4Cl (4.31 g l^{-1}), $CaCl_2 \cdot 6H_2O$ (5.39 g l^{-1}), $MgSO_4 \cdot 7H_2O$ (4.31 g l^{-1}) and $FeCl_3 \cdot 4H_2O$ (54.0 mg l^{-1}) and 0.2 ml of a micronutrient solution containing $CoCl_2 \cdot 6H_2O$ (2000 mg l^{-1}), $MnCl_2 \cdot 4H_2O$ (500 mg l^{-1}), $FeCl_3 \cdot 4H_2O$ (2000 mg l^{-1}), $CuCl_2 \cdot 2H_2O$ (30 mg l^{-1}), $ZnCl_2$ (50 mg l^{-1}), H_3BO_3 (50 mg l^{-1}), $(NH_4)6Mo_7O_{24} \cdot 4H_2O$ (90 mg l^{-1}), $Na_2SeO_3 \cdot 5H_2O$ (100 mg l^{-1}), $NiCl_2 \cdot 6H_2O$ (50 mg l^{-1}), EDTA (1000 mg l^{-1}) and 36% HCl (1 ml l^{-1}). To buffer the pH of the suspension at pH 7, 10 ml of a phosphate buffer (43.08 g l^{-1} KH_2PO_4 and 88.86 g l^{-1} $Na_2HPO_4 \cdot 2H_2O$) was added. To inhibit nitrification the inhibitor N-Allylthiourea (ATU) was added at a concentration of 50 mg l^{-1}. All reagents were of high-grade quality. Composition of the macronutrients, micronutrients, nitrification inhibitor

and pH buffer solutions were those used in standard respiration tests for wastewater, although concentrations were increased 10-fold to correct for the higher microbial activity of the suspensions. After filling of the bottles, the CO_2 trap was put in place and fixed on the top of the bottle and the OxiTop measuring head was screwed on. Finally, the heads were initialized and pressure measuring started. Prior to this study, extensive testing was performed to ascertain whether respiration was limited by mass transport of O_2 and CO_2 or by nutrient depletion for the applied concentrations of OM in the liquid suspension; it was found that this was not the case (Rudrum, 2005).

The pressure measured was used to calculate the cumulative oxygen uptake of the sample (COU in mol O_2 kg^{-1} VS), according to the formula for the ideal gas law (pV=nRT). COU was calculated as follows:

$$COU = \frac{\Delta P \cdot V_{gas}}{8.314 \cdot (273.13 + T)} \cdot \frac{1}{W \cdot TS \cdot VS}$$

where ΔP is the pressure drop of the gas phase (kPa), V_{gas} is the volume of the gas phase (l), T is the temperature of the measurement (°C), 273.13 was added to degrees C to arrive at K, W is the fresh weight of the sample (kg), TS is the total solid content of the sample (kg kg^{-1} wet weight) and VS is the volatile solid content of the sample (kg kg^{-1} TS). In the experiments, both the total volume and the incubation temperature were constant, CO_2 was removed from the system by the trap and nitrification was prevented, indicating that a drop in pressure was exclusively due to O_2 consumption by microbial carbon degradation. The ΔP was determined for the phase in which pressure decreased linearly, as only in this phase is respiration rate directly related to the degradation rate of the OM and can be used as indicator for OM stability. Further details of this procedure can be found in Grigatti et al. (2007). The pressure data from the data logger were processed using MS Excel®. First, COU was calculated on VS basis (mol O_2 kg^{-1} VS), which was then converted to the oxygen uptake rate (OUR; mmol O_2 kg^{-1} VS h^{-1}).

3 Classification of compost stability and basis of respiration rate

A classification of compost stability was derived from the following data:

- Respiration rates for a range of sources of natural organic matter, thus providing a foreseeable range of stability values;
- Respiration rates for a range of different substrates composted in laboratory reactors under well-controlled conditions, providing stability values resulting from biological treatment of organic substrates;
- Respiration rates for a range of fresh biowaste composts and green waste composts from commercial composting facilities in the Netherlands.

Table 1 shows respiration rates obtained from different organic matter sources,

clearly illustrating how fresh organic matter has high respiration rates that can be reduced significantly by several weeks of composting. When fresh compost is subsequently stored under aerobic conditions for 5 months the respiration rate drops further and becomes similar to that of soil organic matter. Peat, a very stable type of organic matter, features the lowest respiration rates. Application of the present method is capable of distinguishing even different types of peat on the basis of their stability level.

Tab. 1: Respiration rate for a selection of different types of organic matter under Oxi-Top standardized conditions at 30 °C

Type of organic matter	Respiration rate (mmol O_2 kg^{-1} OM h^{-1})
Fresh substrates (manure, biowaste, green waste)	50 – 300
Fresh biowaste compost (12 batches)	10 ± 3
Fresh green waste compost (13 batches)	17 ± 7
Fresh compost stored for 5 months (3 batches)	3 ± 1
Soil organic matter (12 batches)	2 ± 1
Light peat (15 batches)	1.7 ± 0.2
Dark peat (20 batches)	0.6 ± 0.1

Table 2 shows the course of respiration rate during composting of fresh biowaste under well-controlled laboratory conditions. These values can be used as a reference for composting facilities.

Tab. 2: Respiration rate before composting and after 1, 2 or 4 wk composting of biowaste under laboratory conditions (well-controlled composting experiments at 15 % O_2 and 50 °C at Dept. Environmental Technology, Wageningen University)

Type of organic matter	Respiration rate at 30 °C (mmol O_2 kg^{-1} OM h^{-1})
Fresh biowaste	50 – 300
Composted for 1 wk	15 - 25
Composted for 2 wk	7 – 15
Composted for 4 wk	7 ± 3

Figure 1A and 1B show the respiration rate of a range of fresh composts collected at Dutch composting facilities treating biowaste and green waste, respectively.

The figure reveals that the mean stability level of composted biowaste is higher (lower OUR) than that of composted green waste. This difference can partly be explained by the differences in composting conditions used for these two types of materials. Biowaste is composted in closed facilities with regular mixing and control of temperature and often also oxygen. Green waste composting is carried

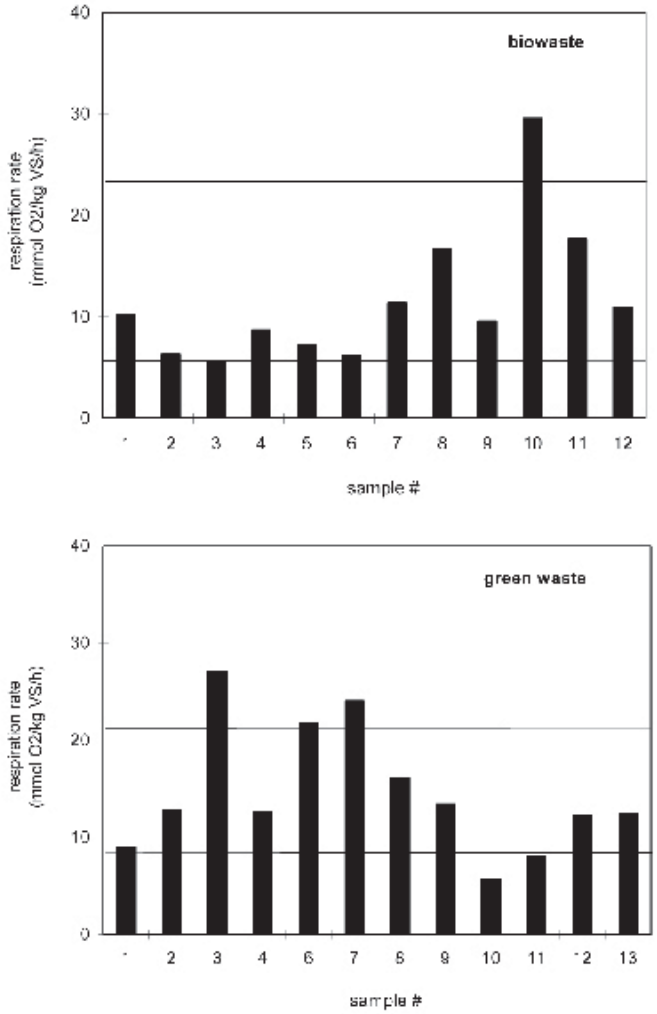


Fig. 1: Respiration rate of fresh biowaste compost (top) and green waste compost (bottom) from commercial composting facilities in the Netherlands

out outdoor using passively or actively aerated windrows where controls are less stringent or even lacking. The figure further shows that stability levels of commercial composts differ largely, meaning that the stability of composts should be determined for those applications in which stability is of critical importance.
Based on the respiration rates presented for various materials we propose a classification with four stability classes for composted materials (Table 3).

Tab. 3: Classification of stability of composted materials with four stability classes, based on OUR values as determined by the method proposed.

Stability class	Respiration rate (mmol O_2 kg^{-1} OM h^{-1})	Respiration rate (mg O_2 g^{-1} OM d^{-1})
Very stable	<5	<4
Stable	5-15	4-11
Unstable	15-30	11-23
Very unstable	>30	>23

4 Discussion and conclusion

The OxiTop stability test can be used to characterize the stability of composted or otherwise stabilized organic materials in a reproducible, fast and reliable way. Knowledge of the stability of composts is important in choosing the right compost for a specific application. For example, if one wants to use compost to enhance disease suppressiveness of a soil or potting mix, it is important to avoid composts with an exceedingly low stability, potentially capable of stimulating soil-borne fungal plant pathogens (Termorshuizen et al., 2006). However, extremely stable composts will no longer stimulate microbial activity and will thus not result in enhanced disease suppression. Compost may also be applied to stimulate the growth of established trees in cities: composts are applied around the tree roots, taking care to avoid use of unstable mixtures capable of producing oxygen depletion resulting in root damage.

The present paper has proposed introduction of a classification comprising four stability classes. We realize that the practical value of such a classification is rather limited, not yet being supported by information concerning areas of application for materials falling into the stability classes proposed. Therefore, further research should be performed to ascertain the stability class best suited to the various applications of compost.

The test described can also be used to determine the N-mineralization potential of organic matter sources. For this application, flasks are used with a sample port and a needle through which samples can be extracted from the suspension without disturbing the pressure measurements. Details of such an extended test can be found in Grigatti et al. (2007). In addition, an anaerobic version of the test has been developed to determine the biochemical methane potential of organic substrates to be used as renewable energy sources.

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