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Precision determination for the specific oxygen uptake rate (SOUR) method used for biological stability evaluation of compost and biostabilized products

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Abstract

This work represents the first attempt to evaluate the precision of the specific oxygen uptake rate method expressed in terms of repeatability (r) and reproducibility limits (R). Three laboratories were involved in an inter-laboratory test for the validation of respiration analyses on six biomass samples (three composts and three biostabilized products) having different degrees of biological stability. Both the maximum specific oxygen uptake rate peak (SOUR) and the cumulative oxygen demand after 12 h (OD_{12}) and 20 h (OD_{20}) of respiration test were investigated. Precisions expressed as the relative standard deviation were in the range of 9–41%. Linear regressions found for r and R, versus OD_{12} and OD_{20}, enabled derivation of precision values (r and R) for all respirometric levels within the operating range. The OD_{12} and OD_{20} indices were found to be more adequate to indicate biological stability since they were less influenced by random errors than the SOUR index.

Keywords: Compost; Biostabilized products; Biological stability; Repeatability; Reproducibility; Precision; Oxygen uptake; SOUR

1. Introduction

Biological stability determines the extent to which readily biodegradable organic matter has decomposed (Lasaridi and Stentiford, 1998). A suitable method for determining biological stability should be capable of numerically representing the actual point reached in the process of decomposition through the use of a measurement on a recognized scale of values, with in turn enables the comparison of different decomposition processes (Lasaridi and Stentiford, 1998).

Several methods have been proposed in the past to measure biological stability (Chanyasak and Kubota, 1981; Iannotti et al., 1993; The US Composting Council, 1997). Amongst them, respiration methods that measure the oxygen uptake rate (OUR) are considered to be the most reliable (Adani et al., 2001). Dynamic respiration tests (DRI) (Adani et al., 2001) have recently received more attention and nowadays are also used for routine analysis (ASTM, 1996; Lombardia Region, 2003). This is because the dynamic conditions imparted through the continuous aeration during measurement allow oxygen to be dispersed, thus avoiding an oxygen diffusion limit in the bio-film mass (Palestki and Young, 1995) and permitting a more accurate measurement of the OUR. This does not occur with static methods, where an underestimation of the OUR is common (Scaglia et al., 2000). Nevertheless, the design of static methods could be improved in order to minimize this problem (Lasaridi and Stentiford, 1998).

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The specific oxygen uptake rate (SOUR) is a simple static test used to assess compost stability (Lasaridi and Stentiford, 1998). This method measures the OUR in a suspension of solid sample in an aqueous medium containing nutrients. Continuous stirring of the suspension and intermittent aeration allows oxygen to be efficiently dispersed, therefore giving a more correct estimation of the OUR (Lasaridi and Stentiford, 1998). Previous work indicated a good linear regression between results obtained by the SOUR test and the DRI, although a dependence of SOUR on the water-soluble organic fraction was identified (Adani et al., 2003). The SOUR test is currently under study in Italy to establish its validity as a possible tool for the determination of the biological stability of compost and biostabilized products, and some laboratories have already started using it on a routine basis. This means that validation of this test is indispensable to certify the quality of results obtained and to assure the scientific applicability of this method (Balls et al., 1990; Fentem et al., 1995).

Validation is defined as the process by which the reliability and relevance of a procedure are established for a specific purpose (Balls et al., 1990). A series of harmonised protocols have been introduced by international organizations to validate analytical methods (Holcombe, 1998; Horwitz, 1992, 1995, 1998; ISO, 1994; Thompson and Wood, 1995).

Several parameters such as (i) selectivity/specificity; (ii) limit of detection, (iii) limit of quantification, (iv) recovery, (v) working range and linearity, (vi) accuracy/trueness, (vii) precision (repeatability and reproducibility), (viii) ruggedness/robustness, should be calculated (FAO, 1998; Hill and Reynolds, 1999; ISO, 1994).

Although it is necessary for chemical methods to be "fully validated" the same cannot apply for biological methods (Wood, 1999). This is because appropriate guidelines for the validation of biological methods have not yet been agreed. In practice the protocols set up for chemical methods have been adapted to biological methods but this was not carried out in a systematic manner (Wood, 1999). The result of this reworking is that validation parameters have limited meaning in the context of biological methods (Schofield, 2000). For example, it is not possible for a biological method to determine the accuracy (accuracy, linearity and specificity values) because these assays should be carried out using reference materials that are not available for biological properties (Van Zoonen et al., 1999). For the same reason it is not possible to determine the limit of detection, the limit of quantification and robustness for such methods. These considerations lead to the fact that, for now, biological methods can only be validated in terms of their precision, for which well-defined protocols are applicable (ISO, 1994).

The precision of a method is represented by its repeatability and reproducibility. The repeatability (r) and reproducibility (R) limits (ISO, 1994; Horwitz, 1995) are the maximum admissible difference between two measurements made consecutively by the same laboratory (repeatability) and by two different laboratories (reproducibility) respectively. If the differences registered are higher than r or R, the results become suspect.

The aim of this work was to carry out the validation process for the SOUR method (Lasaridi and Stentiford, 1998) through an inter-laboratory study in Italy.

2. Methods

2.1. Sample preparation and work planning

Six biomass samples were used in this work. Samples A, B and C represented a compost from lignocellulosic and household wastes and samples D, E and F represented a biostabilized municipal solid waste (the organic fraction of mechanically separated MSW obtained through high rate composting). Each sample was collected at different time intervals during the respective process to represent different degrees of biological stability (samples A and D at the start of treatment, samples with low biological stability; B and E at the middle, with intermediate biological stability; and C and F at the end, with high biological stability) (Table 1). The use of biomasses having different degrees of biological stability (and so different OUR) (Table 1) reflected the necessity to perform precision measures using different levels (e.g., different OUR) of the parameters investigated (ISO, 1994). As a consequence, each sample studied assumed the means of level and in this work these terms will be used sometimes as synonymous. Each sample (3–4 kg)

Table 1
Origin and biological stability degree of samples studied

Samples	Origin	Process	Time of treatment (days)	SOUR (mg O_2 g^{-1} VS h^{-1})	Degree of biological stability[a]
A	Household + lignocellulosic waste	Composting	0	9	Low
B			21	4.94	Medium
C			90	2.7	High
D	Organic fraction of mechanically separated MSW	Biostabilization	0	8.58	Low
E			45	5.72	Medium
F			75	2.58	High

[a] Medium biological stability can be assumed for SOUR of 7 mg O_2 g^{-1} VS h^{-1}, according to Adani et al. (2003).