Prediction of mass loss in different organic materials under field mediterranean conditions: Influence of the initial chemical quality

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ABSTRACT

The influence of chemical quality on decomposition was studied under Mediterranean conditions using the litter bag methodology. Litter of twenty-seven organic materials of different origins was enclosed in litter bags, buried, and periodically sampled during a 14-month period. The mass loss of each material was calculated, different empirical models of one and two pools were adjusted, and the respective constants were estimated. Linear regressions were established between the labile (M_L) and recalcitrant (M_R) mass loss pools and initial material characteristics to determine the best predictor of decomposition. The M_L and M_R pools were related to the presence of soluble or recalcitrant compounds, with values varying between 66.7–685 and 316–934 g kg^{-1}, respectively. Materials with higher soluble compounds, such as PMM or LL, showed significantly higher M_L values than CSS or OWM materials, with higher M_R values where recalcitrant fractions were predominant. Various quality parameters were correlated with mass loss with best predictability of M_L and M_R presented by labile indicators as TAPPI soluble elements that explained 79% and 77% of variation results, respectively. For structural parameters, the best result was presented by total condensed tannins followed by lignin content that explained between 40% and 51% variation of M_L and M_R. Improved predictability was observed by integrating other labile indicators, like water soluble C and N and the respective ratio, or recalcitrant total condensed tannins and holocellulose, in M_L ($r^2_{adj} = 0.890***$) and M_R ($r^2_{adj} = 0.866***$) pool estimation, respectively.

Keywords: mass loss; decomposition, chemical quality, *in-situ* incubations.

INTRODUCTION

Decomposition/degradation of organic matter is one of the most important processes for nutrient recycling in agricultural ecosystems (Montagnini and Jordan, 2002). According to Swift et al. (1979), the decomposition of organic matter is a biological process that includes the physical breakdown and biochemical transformation of complex organic molecules into simpler organic molecules (structural decomposition) and inorganic molecules (mineralization). Nevertheless, even if the decomposition of organic matter is a biological process, it also depends on abiotic factors that influence the activity of the organisms involved. Climate, soil characteristics, and organic matter quality are the three major abiotic factors influencing the dynamics of the decomposition process (Lavelle et al., 1993).

Over the last few years, several methods have been proposed and developed to estimate decomposition processes. The success of these methods is supported by their sensitivity and ability to integrate the factors that influence biological activity. Litter bags are among the most frequently used methodologies, because of their simplicity,

practicability, low cost, and ease of obtaining results (Huang and Schoenau, 1997; Kumar and Goh, 2000; Heim and Frey, 2004; Pavão-Zuckermam and Coleman, 2005; Mungai et al., 2006; Alvarez et al., 2008). Furthermore, as an open reactor, litter bags show high sensitivity to temperature, moisture, soil aeration, and seasonal variations (Berg and McClaugherty, 2008).

In field or laboratory incubations, the quality of organic matter has been considered one of the most influential factors in the dynamics of the decomposition process (Jensen et al., 1995; Heal et al., 1997; Mafongoya et al., 1998). Swift et al. (1979) defined organic matter quality based on the lability of its components, namely C and N, and the presence of inhibitory secondary compounds, such as polyphenols or tannins (Palm and Rowland, 1997). Therefore, several indices have been proposed to evaluate the various metabolic and structural components of organic matter to estimate the decomposition process. Among the available indices, the initial N content and C:N ratio were the first and most used chemical parameters utilized in the decomposition process estimation (Heal et al., 1997; Kumar and Goh, 2000; Qian and Schoenau, 2002). However, the sole use of the initial C:N ratio of organic materials does not consider the lability of these nutrients for microbial growth or the presence of inhibitors. Many studies have revealed that the C:N ratio is a reliable predictor of decomposition process (McKenney et al., 1995; Gilmour et al., 1998). Other approaches have been tested by integrating different forms of C and N availability analyses. Earlier studies have suggested the use of C and N concentrations in the soluble material as a better indicator of the decomposition process (Henrikensen and Breland, 1999; Osono and Takeda, 2005). Fractionation by van Soest (1968), which differentiates the biological stability of C in hemicellulose, cellulose, and lignin fractions, has been successfully used in studies on the decomposition of various organic materials (Henrikensen and Breland, 1999; Magid et al., 2004; Jensen et al., 2005; Morvan et al., 2006). Other quality parameters of organic materials, such as polyphenols (Kraus et al. 2003; Mafongoya et al., 1998) and condensed tannins (Kraus et al., 2004; Olk et al., 2006) have also been reported to influence decomposition patterns. Furthermore, ratios such as lignin:N (Vigil and Kissel, 1991), polyphenols (Cobo et al., 2002) and polyphenols plus lignin to N (Constantinides and Fowes, 1994; Vanlauwe et al., 1997; Mafongoya et al., 1998) have also been used as indices for evaluating the decomposition of organic materials.

The existence of a wide number of parameters has resulted in several comparative studies to select the most important(s) indices in the assessment of biological process dynamics. Given their importance, this discussion has been updated accordingly. Moreover, in most cases, the potential prediction of these indices is evaluated based on the results obtained from biological laboratory incubations, with limitations associated with this methodology (Jarvis et al., 1996; Berg and Laskowski, 2006; Ros et al., 2011; Bai et al., 2013).

In this study, the mass loss process of twenty-seven organic materials with contrasting chemical characteristics in a Mediterranean climate under in non-cultivated soil conditions was investigated. The objectives were:
1. To provide information on the decomposition process under Mediterranean field incubation conditions for various organic materials with different chemical qualities, using the field litter bag method;
2. To find the best model that describes mass loss decomposition for each organic material by comparing two negative exponentials of one and two pool models; and
3. To estimate the importance of different initial (bio)chemical organic matter quality parameters on the dimension of pool(s) of the best-fit model selected.

MATERIALS AND METHODS

Study site

The present study was conducted in a non-cultivated field at the University of Trás-os-Montes e Alto Douro campus, located in Vila Real, Portugal (41°17' North and 7°44'West). In the study area, the prevailing Mediterranean climate conditions are characterized by an annual average rainfall of 970 mm, with more than 80% of the total concentrated between November and May (Agriculture Research Council, Institute for Climate, Soil and Water 1980–2004). The maximum and minimum temperatures occurred in August and January, matching the summer and winter seasons, with 28.9 and 2.5 °C values, respectively. The soil used in the experiment was classified as Eutric Regasol (IUSS Working Group, 2006).

The main physicochemical characteristics and procedures followed in the preparation before use are described in Sousa et al. (2015).

Organic materials selection and preparation

Twenty-seven organic materials were selected considering their different origin and chemistry quality:
1. Urban: municipal solid waste 1 (MSW$_1$), municipal solid waste 2 (MSW$_2$) and compost sewage sludge (CSS);
2. Agro-Industrial: coffee dregs (CD), olive mill waste (OMW), pig meat meal (PMM), vine grape stalk (VGS), and vine grape marc (VGM);
3. Commercial composts: compost 1 (CC$_1$), compost 2 (CC$_2$), compost 3 (CC$_3$), and compost 4 (CC$_4$);
4. Plant residues: chestnut tree leaves (CTL), grapevine leaves (GL), apple leaves (AL), wheat straw (WS), corn stubbles (CS), and lupinus luteus (LL); and
5. Husbandry residues: poultry manure (PM), horse manure (HM), goat manure (GM), sheep manure (SM), cow manure (CM), rabbit feces (RF), pig feces (PF), cow feces (CF), and solid fraction of dairy cattle manure (SFDCM).

The organic materials were air-dried at room temperature (20 ± 2 C) under shadow-ventilated conditions for a period sufficient to facilitate their manipulation and storage. All organic materials were individually ground and sieved, and the fraction between 1- and 2-mm diameter was selected for storage. The use of this fraction considers the mesh size of the decomposition bag selected in order to prevent the loss of materials during the process (Berg and McClaugherty, 2008).

Chemical and biochemical quality of the organic material

A representative air-dried sample from the selected fraction (± 200 g) was ground using a 1.0 mm sieve for initial (bio)chemical characterization, with results ranging between values presented in Table 1. The soil pH was determined in a material/solution suspension at a ratio of 1:2.5 (pH H$_2$O and pH KCl 1M). Total C was determined by dry combustion using an elemental analyzer (PRIMAC SC Skalar, Breda), whereas total N, P, and K were determined using the sulfuric acid wet digestion procedure (Nelson and Sommers, 1982), followed by spectroscopic determination using the Berthelot reaction (Houba et al., 1995), molybdate ascorbic acid method (Murphy and Riley, 1962) and atomic absorption. Water organic soluble C (WOSC) and N (WOSN) concentrations were determined following the methodology proposed by Zibilske and Materon (2005), in which an 5 g aliquot of each organic material was extracted in 50 mL of aqueous solution for 30 min at room temperature. After centrifugation and filtration, the contents of WOSC and WOSN in the extracts obtained were analyzed using a NIRD detector and chemiluminescent technique, respectively, using an elemental analyzer (FORMAC, Skalar, Breda). Other parameters related to the labile pool were measured using the TAPPI method (1978) and are described as water-soluble TAPPI elements. Total polyphenols (TPp) were determined following the methodology proposed by Anderson and Ingram (1993). Briefly, 0.25 g of organic material was extracted in 50% aqueous methanol solution at 60 °C for 1 h, followed by colorimetric quantification at 760 nm the Folin-Ciocalteu (1927) method. The results are expressed as milligrams of gallic acid equivalents (GA$_{eq}$) per kilogram of dry matter. Using the same original extracted matrix for TPp quantification, the total condensed tannin content was also analyzed by the butanol-HCl assay, with absorbance measured at 550 nm (Gessner and Steiner, 2005). The total condensed tannins (TCT) in the material extracts were expressed as mg of cyanine equivalent (Cy$_{eq}$) per kg of dry matter. For total polysaccharides (TPs) an aliquot 0.1 g of organic material was mixed with 100 ml of deionized water for 3 h at room temperature (Hirobe et al., 2004). The TPs in the obtained extracts were quantified at 490 nm (Dubois et al., 1956), the results were expressed as mg of glucose equivalent (Glu$_{eq}$) per kg of dry matter. Neutral detergent fiber (NDF), acid-detergent fiber (ADF), and acid-detergent lignin (ADL) were determined by stepwise chemical digestion using van Soest procedures adjusted for Fibertec (van Soest and Robertson, 1985). The hemicellulose (HEM) and cellulose (CEL) fractions were calculated as NDF-ADF and ADF-ADL differences, respectively. This last fraction can also include the products of microbial biomass decomposition. The ADL fraction is generally referred to as lignin (LIN) Although it includes recalcitrant products resulting from lignin decomposition, the ADL fraction is generally referred to as LIN. All parameter values were corrected and expressed in relation to free ash dry matter, using the methodology proposed by Rosario et al. (2000) for ash determination.

Table 1. Range values of some (bio)chemical characteristics related with initial quality of the various organic materials under study

Parameter	Range values
pH (H$_2$O)	4.1–8.8
Dry mass (g kg^{-1})	677.1–970.2
Ash (g kg^{-1})	23.8–570.5
C (g kg^{-1})	237.2–575.1
N (g kg^{-1})	3.9–76.1
P (g kg^{-1})	0.4–19.7
K (g kg^{-1})	0.8–56.0
Water organic soluble C (WOSC) (g kg^{-1})	0.9–50.5
Water organic soluble N (WOSN) (g kg^{-1})	0.1–12.9
Soluble Elements (TAPPI) (g kg^{-1})	38.0–511.0
Total Polysacharides (TPs) (g Glu$_{eq}$ kg^{-1})	5.0–63.1
Total Polyphenols (TPp) (g GA$_{eq}$ kg^{-1})	2.5–56.2
Total Condensed Tannins (TCT) (g Cy$_{eq}$ kg^{-1})	0.4–33.1
Hemicellulose (HEM) (g kg^{-1})	3.8–312.5
Cellulose (CEL) (g kg^{-1})	124.4–481.4
Lignin (LIN) (g kg^{-1})	32.8–505.1
C:N	3.4–86.4
Holocellulose# (HOL) (g kg^{-1})	142.0–744.0
WOSC:WOSN	0.8–370.0
LIN:N	1.4–20.7
TPp:N	0.1–5.8
TCT:N	0.1–3.6
(TPp + LIN):N	1.6–23.5
HOL:LIN	0.3–10.8
HOL:N	3.3–190.8
(HEM+CEL+LIN)	237.1–815.2
(HEM+CEL+LIN):N	10.4–208.5
LIN:CEL	0.2–0.9
HLQ^β	0.1–0.8
LCI^α	0.2–4.1

Note: # Holocellulose (Hol) = hemicellulose+cellulose (Hem+Cel); ^{β} HLQ – holocellulose to lignocellulose quocient; ^{α} LCI – lignocellulose index.

The initial quality information of the organic materials was complemented by other indices of organic matter, which are related to the ratio between some of the parameters determined and used by different authors to estimate the decomposition process (Fox et al., 1990; Mafongoya et al., 1998; Kumar and Goh, 2003) (Table 1).

Experimental and measurements procedures

The mass loss of the organic materials under study was determined using the litterbag methodology developed by Bocock and Gilbert (1957). An aliquot of 20 g the dried organic matter fraction

(Ø 1–2 mm) of each material was weighed and individually placed in nylon bags with 12 × 12 cm and 1 mm of screen screen-opening dimensions. This mesh size, beyond the prevention of material loss, allows the movement of the main soil fauna and microorganisms, facilitating accessibility to organic substrates and the respective decomposition process (Tian et al., 1992). Each litterbag was individually buried at 10 cm depth in an incubator field reactor developed for this purpose, and the main technical aspects are presented in Sousa et al. (2015). The litter bag improved the control of the quantification of the biological process, allowing a reduction in the number of replications required for each treatment by increasing the repeatability of the methodology. Thus, at the beginning of the incubation experiment (0 d), a total of 648 device reactors/incubators, with the respective litter bags, relative to four replications per treatment and six sampling dates (28, 56, 84, 122, 224, and 392 calendar days) were considered.

For each sampling date, four incubation device reactors, relative to four replications per organic material, in a total of 108 device reactors, were randomly collected and destroyed, and the respective litter bag was removed. Each litter bag was carefully cleaned to remove adhering soil particles or other foreign material, dried in a forced air camera at 35 for 72-h period, and the remaining mass was weighed and recorded. An aliquot was collected to correct the residual water content for the dry weight at 100 °C (Silva, 1967). This last fraction was used to determine the ash content, and the results were expressed in terms of the dry weight remaining ash-free (Rosário et al., 2000).

The evolution of the dry weight remaining (DWR) (g kg^{-1}) was determined by calculating the ratio between the material mass obtained at each sampling date (DWR$_{t=i}$) and the respective initial mass (DWR$_{t=0}$), using DWR=(DWR$_{t=i}$/DWR$_{t=0}$) × 1000 expression. To estimate the dimensions of the decomposable pool(s) and the respective kinetic constants associated with each organic material, two negative exponential models were considered. One is based on one decomposable pool (DMR$_t$=M$_0$*exp$^{(-k0*time)}$+(1000-M$_0$)) (Olsen, 1963), and the second is based on two decomposable pools (DMR$_t$=M$_L$*exp$^{(-kL*time)}$+M$_R$*exp$^{(-kR*time)}$ +(1000-M$_L$-M$_R$)) (Berg and Agren, 1984). *DMR$_t$* is the dry mass remaining after time *t*, M$_0$ is the potential decomposable pool, k_0 is the associated kinetic constant,

M_L is the dry mass relative to the labile pool, M_R is the dry mass relative to the resistant pool, and k_L and k_R are the associated constants. According Andren and Paustian (1987), these models are most frequently used to describe the nature of weight loss of decomposed organic materials in agricultural and forestall soils.

Main environmental factors were recorded during the experimental period. Rainfall was measured continuously at a meteorological station located at the University Campus, the soil temperature was recorded by the sensors connected to a DATALOGGER equipment, and volumetric soil moisture was periodically measured using a TDR sounder (Time Domain Reflectometry), model TRASE 6050XI (Topp, 1980). The calendar time was normalized at 15 °C temperature and soil moisture range, defined between the maximum and minimum values registered during the experimental period, using empirical models described by Recous et al. (1995) and Paul et al. (2002), where the procedures and results are presented in Sousa et al. (2015).

Statistical analysis

In the adjustment of the experimental results to empirical models, the *Nonlin* processed SYS-TAT 13® Windows Program (Systat Inc.) was employed based on the minimization of the squared difference, with the quality and selection model based on the evaluation of adjusted coefficients of determination (r^2_{adj}) and the root mean square error (RMSE) (Isacc et al., 2000). All data of the estimated models were subjected to analysis of variance (ANOVA), with significant differences between means separated using the least significant difference (LSD) test for a probability level of 0.05. The relationship between the estimated organic decomposable pool(s) materials and the respective initial chemical quality was determined using simple and multiple linear regression techniques, based on Pearson correlation (r) and determination adjusted (r^2_{adj}) coefficient calculations. In the construction of multiple regression models, the *forward stepwise* analysis was applied in order to increase the proportion of variance explained, following the criteria defined by De Neve et al. (2003), with an *F* probability for entry and exit of a variable of 0.05 and 0.1, respectively, eliminating the possible cases of *multicollinearity* among the variables (Doran and Parkin, 1994).

RESULTS AND DISCUSSION

Mass loss pool model

The quality of the mass loss values adjusted to the negative exponential model revealed high and significant results (p < 0.001). Practically, in all organic materials studied the negative exponential two pools model show best quality adjustment results comparative to one pool model (Table 2), confirmed the tendency observed in similar studies (Coûteaux et al., 1998; Camargo et al., 2002; Cattanio et al., 2008).

Despite the quality adjustment observed in the one-pool model for some organic materials, this model seems to overlook important aspects related to the organic matter quality differences observed among the organic materials studied, which influence the dynamics of the decomposition process during the incubation period. The one-pool model omits important phenomena related to the initial leaching of soluble compounds or the biological lignin resistance effect, which are responsible for an initial or final decay rate, respectively, that are higher or lower than the average (Berg and Laskowski, 2006). This limitation is more remarkable for the organic materials with high biochemical diversity, where the adjustment quality difference between the studied models is more pronounced, as in the cases of MSW_2, CSS, CC_4 and PMM, among others (Table 2). These differences, also observed by other authors (Deas et al., 1986; Matus and Rodrigues, 1994; Girisha et al., 2003), reflect the complexity of the decomposition process regulated by the chemical quality of organic matter, supporting the assumption that the studied materials are composed of different kinetic fractions. The two-pool model translated a decomposition process formed by a more active first stage, related to lixiviation and decomposition of soluble and low molecular weight compounds (Berg et al., 1982; Torres et al., 2005), followed by a less active second stage, dominated by resistant and recalcitrant compounds, regulated by cellulose and lignin (Berg and McClaugherty, 2008). Considering that the negative exponential two-pool function is a more realistic approach, this model was selected for the decomposition process.

The results indicated that the decomposition estimated model parameters, related to mass loss labile (A) and recalcitrant (B) pools, and the respective kinetic constants or decay rates, k_A and

Table 2. Values of potential decomposable organic labile (M_L) and resistant (M_R) pools and respective kinetic constants associated (k_L, k_R), estimated by two pool negative exponential model, for the organic materials under study

Org. material	M_L (g kg^{-1})	k_L (day^{-1})	M_R (g kg^{-1})	k_R (day^{-1})	r^2_{adjust}
MSW$_1$	250.1	0.0453	752.0	0.0003	0.990 ***
MSW$_2$	379.1	0.1156	620.4	0.0024	0.999 ***
CSS	66.7	0.0575	933.7	0.0001	0.999 ***
CC$_1$	224.3	0.0501	776.2	0.0011	0.994 ***
CC$_2$	140.4	0.0815	859.2	0.0017	0.992 ***
CC$_3$	121.7	0.0824	878.1	0.0034	0.994 ***
CC$_4$	36.7	0.0490	968.7	0.0001	0.998 ***
CD	276.5	0.0232	720.8	0.0006	0.997 ***
OMW	115.7	0.1001	884.4	0.0005	0.995 ***
VGM	274.2	0.0508	726.3	0.0001	0.999 ***
VGS	158.6	0.0490	842.3	0.0014	0.997 ***
PMM	685.0	0.0785	315.5	0.0074	0.999 ***
CTL	114.8	0.0484	885.7	0.0030	0.997 ***
AL	287.3	0.0543	713.3	0.0034	0.995 ***
GL	252.5	0.0271	747.6	0.0027	0.995 ***
WS	261.3	0.0576	740.2	0.0038	0.992 ***
CS	174.7	0.0395	825.6	0.0038	0.995 ***
LL	475.9	0.1092	524.0	0.0031	0.996 ***
PM	391.3	0.0659	608.3	0.0044	0.997 ***
CM	241.4	0.0010	763.8	0.0001	0.939 ***
HM	158.6	0.0023	841.4	0.0001	0.973 ***
GM	108.5	0.0059	891.8	0.0001	0.981 ***
SM	201.5	0.0035	798.6	0.0001	0.996 ***
CF	247.5	0.0025	752.9	0.0001	0.986 ***
RF	171.2	0.0017	828.7	0.0001	0.989 ***
PF	280.8	0.0269	714.5	0.0019	0.979 ***
SFDCM	143.1	0.0010	857.1	0.0001	0.987 ***
Mean	231.1	0.0455	776.7	0.0016	
CV (%)	58.7	209.3	17.7	116.4	
LSD$_{0.05}$	82.3	0.0246	82.1	0.0016	

Note: *** – Significant at the 0.001 probability level, respectively; MSW$_1$: municipal solid waste 1; MSW$_2$: municipal solid waste 2; CSS: compost sewage sludge; CC$_1$: commercial compost 1; CC$_2$: commercial compost 2; CC$_3$: commercial compost 3; CC$_4$: commercial compost 4; CD: coffee dregs; OMW: olive mill waste; VGM: vine grape marc; VGS: vine grape stalk; PMM: pig meat meal; CTL: chestnut tree leaves; AL: apple leaves; GL: grapevine leaves; WS: wheat straw; CS: corn stubbles; LL: lupinus luteus; PM: poultry manure; CM: cow manure; HM: horse manure; GM: goat manure; SM: sheep manure; CF: cow faeces; RF: rabbit faeces; PF; pig faeces; SFDCM: solid fraction of dairy cattle manure.

k_B, varied significantly (p < 0.05) with the organic material studied (Table 2). The values ranged between 79.2 to 619.1 g kg^{-1} and 0.0010 to 0.1156 day^{-1} and 32.1 to 943.4 g kg^{-1} and 0.0001 to 0.0074 day^{-1} for labile and recalcitrant pools dimension and decay rates, respectively.

The B and k_B values were significantly higher in PMM, LL, PM, and MSW$_2$ (Table 2). The major soluble nutrients or organic compounds, which are related to the lowest C:N ratio values or WSOC and TAPPI soluble elements, were responsible for the significant mass loss observed in the initial phase of these organic materials (Figure 1). These findings are supported by other studies, where the authors reported a high initial dynamic decomposition phase associated with a significant decrease in soluble compounds resulting from their major biological accessibility and leaching effects (Parsons et al., 1990; Ajawa and Tabatabai, 1994; Huang and Schoenau, 1997; Girisha et al., 2003; Murovhi and Materechera, 2015).

Nevertheless, materials such as OMW, CC$_2$ and CC$_3$ presented elevated k_L values associated with lower M_L values (Table 2). Unlike the M_L dimension, which is more related to total nutrients and soluble compounds (Shepherd et al.,

2005), k_L is related to chemical quality diversity or organic compound type, with the respective value defined as the mean of all respective k_L compound types existing in the organic material (Rovira and Vallejo, 2002). These findings are supported by the highest CV values obtained for k_L compared with the other estimated parameters (Table 2), corroborating the chemical complexity and diversity of the labile pool reported by Berg and Laskowski (2006).

The highest values estimated for the M_R pool were observed for CC_4, CSS, PF, CTL, and OMW materials (Table 2). The results are associated with the major resistant or recalcitrant compost content (Table 1), namely cellulose and/or lignin, which are more resistant to microbial activity (Osono and Takeda, 2006; Zang et al., 2008), responsible for the lower mass loss values observed during the incubation (Figure 1). The low N content or high levels of inhibitory polyphenol compounds can also justify the low mass loss (Mafongoya et al., 1998; Kraus et al., 2004) observed in CTL or OMW materials, which increases the recalcitrant pool value dimension.

For the recalcitrant decay rate (k_R), the minor CV values obtained for the k_R materials reflect the higher chemical homogeneity of the remaining fraction when compared with the labile fraction (Table 2). The lowest k_R value of 0.0001 day^{-1} observed for CSS, CC_4 and other materials of animal origin reflect the major content of lignified compounds present in these materials and their respective major biological resistance (Figure 1). In husbandry materials, the low k_R values observed are related to the composition of the feed, with the fact that animals also absorb the more digestible (labile) fractions of the feed, and with the vegetal material used for the bed, constituted

by forest residues with high lignin content. In turn, the major significant k_R values observed in PMM (0.0074 day^{-1}), PM (0.0044 day^{-1}), and CC_3 (0.0033 days^{-1}), or materials of crop origin (0.0027 to 0.0038 day^{-1}) were associated with minor lignified compound content. Other structural compounds present in these materials, such as hemicellulose, cellulose, and other polysaccharides, are more labile than lignin (Jeffries, 1994; Cagnon et al., 2009), which explains the higher k_R values obtained.

Mass loss pool and chemical quality organic material relation

The Pearson coefficient values obtained between the chemical characteristics of organic materials as well as decomposition of labile and recalcitrant pool correlation analysis are presented in Table 3. Significant relationships were observed between the correlated parameters, with the results depending on the studied chemical indices and the pool quality. Positive significant correlation was found between mass loss labile pool (M_L) and N, WOSC, WOSN, TAPPI soluble elements and TPp parameters (Table 3).

An increase in the nutrient or soluble compound content promotes a higher labile pool and mass loss of organic materials (Figure 1). The strong correlation between M_L and soluble nutrient or compound indicators such as TAPPI, OWSC, or OWSN demonstrated that the availability of labile forms determined the mass loss during the early stages of decomposition. These results are consistent with the conclusions of Huang and Schoenau (1997) and Girisha et al. (2003), where the mass loss of leaf litter was related to WOSC and N content. Similarly, Benbi et al.
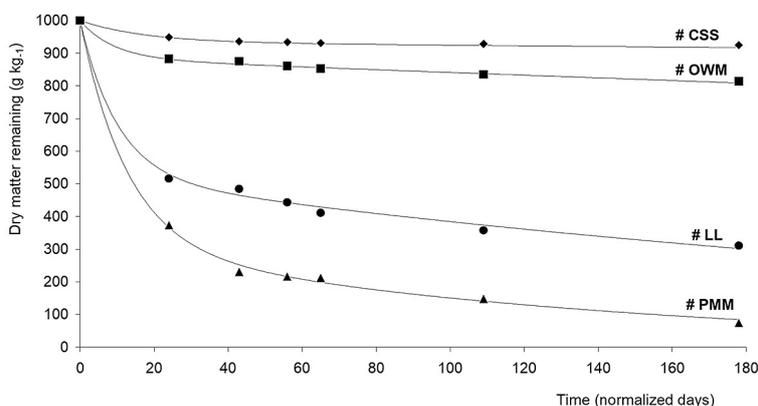

Figure 1. Dry mass values observed during the time incubation

Table 3. Pearson coefficients (*r*) values obtained between (bio)chemical characteristics of organic materials and labile (M_L) and recalcitrant (M_R) pools correlation

Parameter	M_L	M_R
C	- 0.384 *	0.398 *
N	0.744 ***	-0.729 ***
WOSC	0.709 ***	-0.649 ***
WOSN	0.789 ***	- 0.737 ***
TAPPI	0.888 ***	- 0.875 ***
TPs	0.167 ns	- 0.114 ns
TPp	0.447 *	- 0.407 *
TCT	- 0.718 ***	0.726 ***
HEM	- 0.391 *	0.257 ns
CEL	- 0.529 **	0.554 **
LIN	- 0.629 ***	0.660 ***
C:N	- 0.397 *	0.381 *
WOSC:WOSN	0.778 ***	- 0.777 ***
LIN:N	- 0.539 **	0.409 *
TPp:N	- 0.275 ns	0.232 ns
TCT:N	- 0.384 *	0.436 *
(TPp+LIN):N	- 0.436 *	0.487 *
HOL	- 0.457 *	0.471 *
HOL:N	- 0.291 ns	0.248 ns
HOL:LIN	- 0.153 ns	0.169 ns
HEM+CEL+LIN	- 0.636 ***	0.658 ***
(HEM+CEL+LIN):N	- 0.473 *	0.287 ns
LIN:CEL	- 0.179 ns	0.138 ns
HLQ	- 0.449 *	0.487 *
LCI	- 0.425 *	0.497 *

Note: Quality parameters abbreviations are as show in Table 1. *, **, *** – probabilities associated to Pearson correlation coefficients at $p < 0.05$, $p < 0.01$ and $p < 0.001$, respectively.

(1998) found that the WOSN content was strongly correlated with mass loss during the early phase of decomposition of the leaves, roots, and straw of a range of agricultural residues. For TPs, the positive correlations obtained can be explained by the chemical complexity and reactivity diversity of these compounds (Palm and Rowland, 1997). Soluble phenolic compounds, namely hydrolysated tannins, can serve as a C source for microorganisms or lixiviated (Fierer et al., 2001; Kraus et al., 2004), contributing to mass loss in the initial stage of the decomposition process (Bending and Reading 1997; Girisha et al., 2003).

Significant positive correlations were observed between the M_R pool and other chemical indicators related to the structural fraction of the materials (Table 3).

Unlike the M_L pool, structural fraction indicators related to HEM, CEL, LIN, and TCT content were positively correlated with M_R pool materials, suggesting that these compounds are resistant to biological activity, contributing to an increase in the respective dimensions. The higher results obtained for LIN revealed that the M_R pool dimension is related to the major biological resistance of cellulose or hemicellulose. This recalcitrant effect promotes an increase in lignin levels over time (Girish et al., 2003), with the inhibitor effect proving to have a more significant influence on mass loss and process dynamics after the disappearance of labile fractions (Rutigliano et al., 1996; Isaac et al. 2000).

The results also showed that TCT was a material constituent that affected the M_R pool dimensions and the respective mass loss process (Table 3). Condensed tannins decompose slowly by binding to the cell wall and proteins, making them physically or chemically inaccessible to decomposers in a similar manner to lignin (Mafongoya et al., 1998). The high inhibitory microbial activity and recalcitrant characteristics, resulting from their phenolic nature, induce an increase in M_R pool materials with high TCT content, reducing the respective mass loss (Figure 1). This finding is supported by several studies showing that the organic materials with high tannins are associated with slower decomposition mass loss (Constantinides and Fownes, 1994; Handayanto et al., 1997; Driebe and Whitam, 2000).

Considering both pool results, a simple linear regression analysis indicated that best prediction results were given by TAPPI soluble elements, which explained 78.8 % and 76.6 % of the variation in M_L and M_R results, respectively (Figure 2). With proximate results, other soluble compound indicators also reveal highly predictable results for M_L and M_R pools, such as the WOSC:WOSN ratio with r^2_{adj} values of 0.605 (***) and 0.604 (***), respectively. These results indicate that soluble compounds are the main constituent materials that affect the mass loss process for the organic materials studied, influencing both the dimension values of the M_L and M_R pools. The LIN index revealed modest predictability results, explaining between 40% and 43% of variation pools estimated results. Nevertheless, the results agreed with the conclusions presented by Murovich and Materechera (2015) and other authors (Vaieretti et al., 2005; Mungai and Motavalli, 2006), where lignin content had a more significant influence on the mass loss of the materials, especially in the second stage of the decomposition process
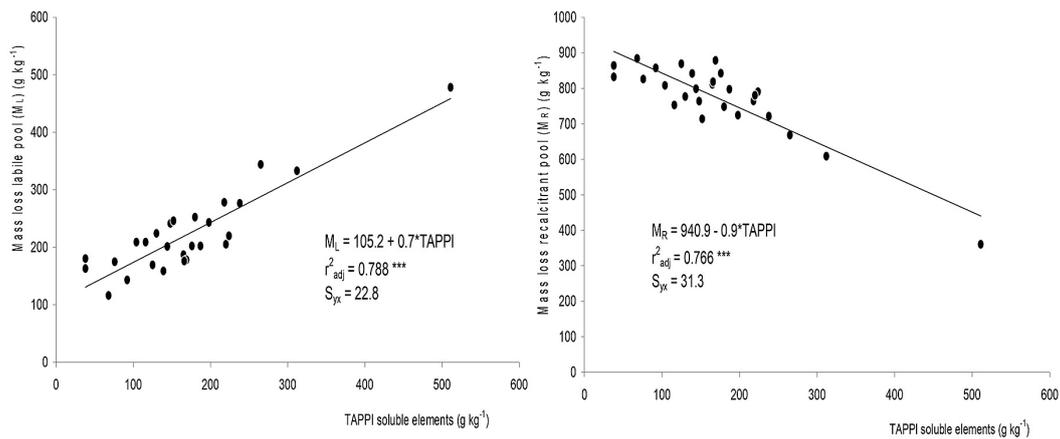
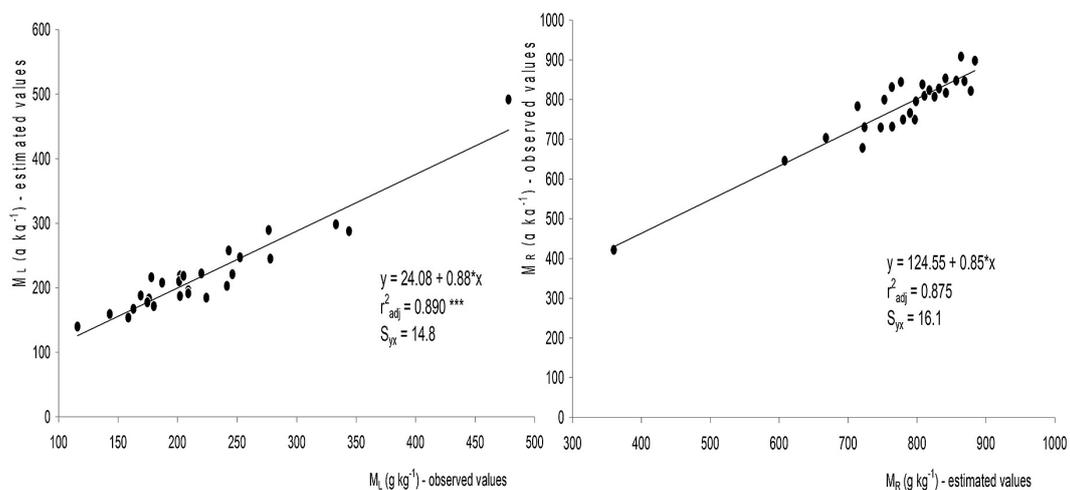**Figure 2.** Linear regression indicated by TAPPI soluble elements

Figure 3. Linear regression in predictability of M_L and M_R pools

(Girish et al., 2003; Rahman et al., 2013). The factors related to a longer incubation time, with a better-defined second stage, where lignin has a more pronounced effect on mass loss, possibly explaining the highest correlations observed by these authors compared to the presented results. This fraction includes the compounds resulting from microbial activity (Berg and Laskowski, 2006). This modified lignin fraction can be more easily degraded by lignolytic enzyme systems than true lignin (Steffen, 2002), affecting the correlation with M_R pool values, especially compared to TCT, which explains 53% of M_R value variation observed. The C:N ratio, one the most studied and analyzed parameters in decomposition and mineralization predictability (Whitmore et al., 1997; Seneviratne, 2000; Kumar and Goh, 2003), revealed lowest regression results that do not explain more than 15% of labile and recalcitrant pool variation results. The inability to

differentiate the quality of C forms limits their utility in predicting the decomposition process, especially in relation to the OWSC:WOSN ratio, as Roberts et al. (2009) observed.

Stepwise multiple linear regression showed an increase in predictability of M_L and M_R pools by including more chemical parameters related to initial material characteristics (Table 4). For mass labile pool, the best prediction model included TAPPI soluble elements, OWSC:OWSN ratio, TCT, HOL, and WOSN, which together explained approximately 90% of variation results of M_L (Figure 3). A higher number of parameters is needed to improve the fraction labile estimation, confirming their high chemical complexity (Filep et al., 2022). The results also showed that soluble nutrients or organic compounds were the main contributors to the mass loss observed during the initial decomposition process, as revealed by the higher standardized regression coefficients (β) of

Table 4. Multiple linear regression equations obtained between labile (M_L) and recalcitrant (M_R) mass loss pool and initial characteristics chemical parameters of organic materials by stepwise analysis method

Model No.	Regression equation	r^2_{adj}	S_{yx}	β_{TAPPI}	$\beta_{OWSC:OWSN}$	β_{TCT}	β_{HOL}	β_{OWSN}
	Mass labile pool (M_L)							
1	$M_L = 105.2+0.7*TAPPI$	0.788***	22.8	0.982	-	-	-	-
2	$M_L = 97.9+0.6*TAPPI+$ $+2.5*OWSC:OWSN$	0.816***	20.8	0.726	0.248	-	-	-
3	$M_L = 160.6+0.4*TAPPI+$ $+3.0*OWSC:OWSN-12.5*TCT$	0.849***	17.7	0.550	0.299	-0.241	-	-
4	$M_L = 218.5+0.3*TAPPI+$ $+3.6*OWSC:OWSN-15.9*TCT-$ $0.1*HOL$	0.871***	16.6	0.452	0.351	-0.307	-0.196	-
5	$M_L = 231.4+0.1*TAPPI+$ $+4.1*OWSC:OWSN-14.2*TCT-$ $0.1*HOL+6.4*OWSN$	0.890***	14.8	0.423	0.403	-0.263	-0.255	0.259
	Mass recalcitrant pool (M_R)							
1	$M_R = 940.9-0.9*TAPPI$	0.766***	29.2	-0.880	-	-	-	-
2	$M_R = 955.7-0.7*TAPPI-$ $5.1*OWSC:OWSN$	0.825***	19.4	-0.652	-0.339	-	-	-
3	$M_R = 862.9-0.5*TAPPI-$ $-5.8*OWSC:OWSN+18.5*TCT$	0.875***	16.1	-0.477	-0.391	0.289	-	-

Note: β_{TAPPI}; $\beta_{OWSC:OWSN}$; β_{TCT}; β_{HOL}; β_{OWSN} – standardized regression coefficients for TAPPI, OWSC:WOSN; TCT; HOL; OWSN, respectively, *** – significantly for a probability level of 0.1%.

the respective parameters (Table 4). Nevertheless, for better predictability, it is also necessary to include the information related to the TCT and HOL content of the materials.

In relation to the M_R pool, the stepwise multiple regression obtained continued to reveal the necessity of including more chemical quality parameters to improve the predictability of mass loss results. The best model obtained using TAPPI, WOSC:WOSN, and TCT as independent variables explained 88% of mass loss recalcitrant pool value variation observed (Figure 3).

The best model was selected continuously to reveal the significant importance of labile fractions in recalcitrant pool estimation, with TAPPI presenting the highest importance, followed by the WOSC:WOSN ratio and TCT (Table 4).

CONCLUSIONS

Considering the results obtained, it is possible to conclude that: (i) mass loss of organic materials generally follows an exponential two-phase trend; (ii) the labile (M_L) and recalcitrant (M_R) mass loss pool dimensions were significantly affected by chemical quality material characteristics, with high levels of soluble or recalcitrant organic compounds increasing the M_L or M_R pools in the organic materials, respectively; (iii) k_L was significantly higher than k_R, confirming the major

solubility and accessibility of C forms of labile compared to the resistant recalcitrant pool; (iv) the TAPPI soluble elements are the chemical indicator quality that best predicts the M_L and M_R pool dimension values; (iv) an increase in the predictable capacity of both pools is observed with inclusion of more chemical parameters, especially those related to soluble nutrients or compounds.

The results of the present study support the contention that material chemical quality is one of the most important factors influencing decomposition under field conditions. On the basis of the initial chemical material characteristics, particularly those related to soluble fractions, it is possible to satisfactorily predict the mass loss of different pools. The study confirmed the necessity of using a group of indicators to better predict the different stages of the decomposition process, including other simultaneous parameters in the minimum dataset characterizing the material quality related to tannins (TCT) and holocellulose (HOL).

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