Simple method for determination of mass loss rates for soil-contaminated samples in decomposition studies

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Summary. Soil contamination frequently causes large errors in estimates of decomposition rates. Existing methods for correcting for this error are costly and/or time-consuming. A new method is presented for determining the composition by weight of two constituents of a mixture from the oven-dry weight of the mixture (W_m) and the gravimetric moisture contents of the mixture (g_m) and pure subsamples of the two constituents (g_1 and g_2), based on the following equation: $W_1 = W_m^*(g_m - g_2)/(g_1 - g_2)$, where W_1 is the oven-dry weight of component 1. The method was validated using pre-weighed mixtures of cattle dung and soil. There was a highly significant relationship between the moisture content of the air-dry mixture and the proportion of dung by weight ($p < 0.001$; $r^2 = 0.999$; $n = 16$). The moisture equilibration method for determination of weight loss rates for soil-contaminated samples offers the following advantages when compared with other methods (1) the only equipment needed is a balance and an oven; (2) very large samples can be utilized, minimizing or eliminating subsampling errors for the mixed samples; (3) no grinding is necessary. The method can also be applied to other types of samples, such as soil-contaminated roots.

Key words: Decomposition, methods, litterbags, soil, moisture retention, roots

Introduction

The rate of weight loss is one of the most commonly cited parameters in litter decomposition studies. In spite of recent advances in the direct measurement of respiration rates, litter bags, litter standing crop measurements, and decomposition standards continue to be recommended as the most reliable methods for determining absolute decomposition rates (Anderson & Ingram 1989). All three of these methods are frequently complicated by soil contamination. Three options are currently available to deal with this problem: (1) exclusion in the field with physical barriers, (2) mechanical separation in the laboratory, or (3) calculation based on the measurement of a constituent of the organic material, or of the soil.
Soil can be at least partially excluded in the field by reducing the mesh size of litter bags. However, reducing mesh size may exacerbate other widely-recognized limitations of litter bags by excluding macroinvertebrates (Holter 1979), or by affecting microorganism populations through microclimate modification (St. John 1980).
Soil can be mechanically removed in the laboratory by washing with water (Anderson & Ingram 1989), by flotation in water or organic solvents, or by dry-sieving and hand separation. These methods are time-consuming, and rarely yield a quantitative separation due to the cementation of the soil to the organic material. Dispersion agents can be used to separate the two components, but they also disperse the clay fraction of the soil, which

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must then be sieved our or reflocculated. Many of the organic solvents are both expensive and toxic, making them unsuitable for use on large samples.

Finally the contribution of soil to the total weight of the mixture can be calculated from the ash content of the mixture. Carbon and nitrogen, which are increasingly measured on a routine basis in decomposition studies, can be used in place of or in addition to total ash. However, in nearly all cases, a correction must be made to account for the ash in the litter sample, or the carbon or nitrogen in the soil. Most analytical techniques for these constituents require small, ground subsamples of the original. Subsampling and grinding both take time and subsampling increases error. Furthermore, the analytical techniques themselves are expensive, with the exception of ash content, and time-consuming. Finally, the initial moisture content of the mixed and pure samples must be determined to correct to dry weight.

A new method which requires only the measurement of the gravimetric moisture content of the mixed and pure samples is described here. The moisture retention characteristics of organic materials generally differ from those of soil in at least part of the range from saturation to oven-dry. For many organic materials and soils, the gravimetric moisture content for the range from air-dry to oven-dry can be used to quantify soil contamination. If desired, the difference between the two moisture contents can be increased by equilibrating the samples at different tensions using tension tables, pressure plates, or saturated salt solutions (Klute 1986). It is not necessary to know the tension at which the samples are equilibrated. Furthermore, it is not necessary that samples from the same experiment be equilibrated at the same tension. Pure subsamples should, however, be equilibrated with the mixtures which they represent if temperature or relative humidity varies between equilibration chambers. The samples should not be ground.

The following protocol is applied to a mixed sample. The procedure used to test this protocol is decribed below in "Materials and Methods". For the purpose of this explanation, a soil-contaminated litter sample is used. The samples are air-dried. Sub-samples of pure litter and pure soil are then separated and placed in pre-tared moisture tins without caps. The requirements for the pure samples are identical to those used for ash or a particular element: they must be representative of the material in the mixed samples. All of the samples are then placed in an equilibration chamber, such as a sealed cooler, in which constant temperature and vapor pressure can be maintained. More rapid equilibration will occur under vacuum, but this is not necessary, particularly if the samples have been air-dried in the same environment in which they are placed in the cooler. After the samples have come to equilibrium, they are capped, removed, weighed, dried to constant weight, and weighed again. Oven and equilibration chamber conditions must remain constant for each set of contaminated and pure samples; they do not, however, need to be consistent for different sets, provided that pure samples are equilibrated with each set. The weight of the litter in the mixture is then calculated from the dry weight of the mixture, and the gravimetric moisture contents of the pure soil, pure litter and the mixture itself (Eq. 1).

$$W_1 = W_m^* (g_m - g_2)/(g_1 - g_2) \tag{1}$$

where W_1 is the oven-dry weight of the litter in the mixture, W_m is the weight of the mixture, and g_m, g_1 and g_2 are the equilibrated gravimetric moisture contents of the mixture, the litter, and the soil, respectively.

Materials and Methods

The method was tested with pre-weighed air-dry mixtures of soil and dung. Three ratios were used, in addition to the pure samples: 20, 50 and 80% dung. The total mass of each sample was 10 g. Three replications of each mixture were equilibrated in a 48-L picnic cooler placed on cardboard slats on a desk for 1 week. Gravimetric moisture content at 70 °C and 105 °C (± 2 °C) was then determined by drying for 48 h in a forced draft oven. The samples dried to constant weight within 24 h and dung content was calculated using Equation 1.

Results

There was a highly significant linear relationship between the proportion of dung in the sample and the gravimetric moisture content of the mixture (Fig. 1). This resulted in an equally significant relationship between the actual and predicted weight of the dung ($p < 0.001$; $r^2 = 0.999$; $n = 9$ for both oven temperatures). Statistics for the pure samples are presented in Table 1.

Table 1. Average percent gravimetric moisture content of equilibrated pure dung and soil determined at two oven temperatures. Means are based on three replications and are followed by standard deviations in parentheses

	Dried at 70 °C	Dried at 105 °C
Dung	5.898 (0.001)	7.225 (0.020)
Soil	2.839 (0.008)	3.560 (0.014)
Difference	3.060	3.665

Discussion

The results demonstrate that the method can be extremely precise. The precision of the method can be increased by (1) increasing sample size, and (2) selecting an equilibration moisture tension and an oven temperature which maximize the difference between the gravimetric moisture contents of the pure samples. For example, the differences for the materials used in this trial were 3.06% and 3.66% at 70 and 105 °C, respectively.

Potential pitfalls

Most errors due to variable equilibration and drying conditions can be eliminated by blocking pure samples into equilibration containers and ovens with mixed samples. However,
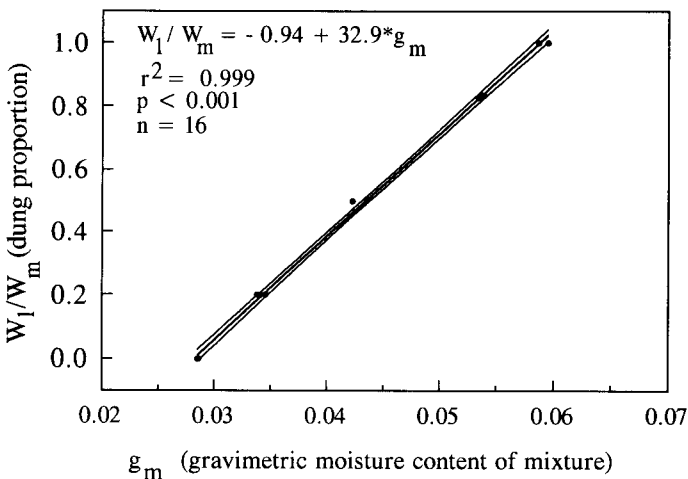
Fig. 1. Proportion of dung by weight in a mixed soil and dung sample as a function of the gravimetric moisture content of the mixture (70 °C) based on Eq. 1. Gravimetric moisture content is presented as a ratio. Outer lines describe the 99% confidence interval for the inner regression line

variability in environmental conditions within the oven or sharp changes in oven vapor pressure within several hours of oven-dry weight determinations can invalidate the method. Sample temperature in laboratory ovens can vary by over 30 °C. Temperature variability can be controlled by using forced-draft ovens and keeping samples away from the walls and floor. Sharp changes in oven atmospheric vapor pressure immediately prior to weighing can also bias the results because the diffusion paths will vary among samples.

Additional applications

The method is applicable to any system in which the individual weights are needed for a mixture of two materials which have distinct moisture retention characteristics. Correction for soil contamination of root samples is one potential applications.

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