



Does sample drying temperature affect the molecular characteristics of organic matter in soil and litter? A statistical proof using ATR infrared spectra



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ABSTRACT

At each step of sample handling and pretreatment it is important to consider potential effects on the result of the analysis. Sample drying is a basic procedure in sample preparation for many analyses. In practice, different sample drying procedures (e.g. freeze drying, air drying, or drying at different temperatures up to 105 °C) may be used prior to perform attenuated total reflection (ATR) spectroscopy, but the effect of sample drying temperature is rarely assessed. The latter technique was used to test the influence of sample drying treatment on the molecular characteristics of six contrasting substrates which are common in soil science (two mineral soils, two organic soils and two foliar litter samples). We compared the mid-infrared ATR spectra of the samples after freeze-drying, as well as oven-drying at 60 and 105 °C, respectively. Our results demonstrate a significant absence of any detectable influence of sample drying on the spectra of the six substrates under study, and hence on parameters which are reflected in the spectra. Accepting null hypothesis of no effect is linked to a risk of the second kind (β). Calculation of β needs a proper design of the experiment (DoE). By means of this DoE, the result is significant on a 5% level. In previous studies where drying temperature had influenced organic matter composition, either (1) labile compounds in fresh organic materials were under investigation, or (2) parameters were investigated which cannot be detected by using the ATR technique. Our results indicate that in the context of soil organic matter – where fresh materials containing labile compounds are of minor interest – the drying conditions do not affect the determination of parameters, which can be studied by means of the ATR technique.

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1. Introduction

In order to produce reliable data, laboratory analyses are ideally performed following standardized procedures (e.g., European standards (EN)) describing each step from sampling to data calculation. However, conducting experimental research generally implies the use of new techniques, for which no such standardized procedures exist. This can result in fluctuating experimental conditions between studies, the influence of which is not always accounted for.

For instance, the characterization of organic matter (OM) in soils and litter material is usually carried out on samples that are dried prior to analysis (Table 1). In practice, a number of procedures are employed to dry soil and litter samples. Apart

from the gentle freeze-drying procedure, air-drying as well as oven-drying at temperatures up to 105 °C are common. A number of examples are described in Table 1, involving mid-infrared spectroscopy. In some studies, the drying procedure and/or temperature were not mentioned (Table 1). These apparently assume that the different temperatures do not affect OM characteristics/composition, possibly because below 105 °C only water is assumed to be lost from the samples.

Oven-drying can be required to improve the analysis as exemplified by Miltz and Don for soil carbon (C) with near-infrared (NIR) spectroscopy [21]. However, the fact that only a rather small number of studies related to environmental sciences appear to have investigated the influence of the sample drying procedure on OM composition indicates that this potentially significant factor is rarely evaluated. Some studies have indeed shown that chemical properties of OM in diverse organic materials, soils and sediments were affected by the drying temperature. In

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