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Influence of Drying Temperature and Duration on the Quantification of Particulate Organic Matter

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ABSTRACT: Various drying conditions, temperatures (40 to 80°C) and durations (overnight to 72 hrs), for the particulate organic matter (POM) fraction after wet-sieving size fractionation have been applied for determination of POM contents in the weight loss-on-ignition method. In this study, we investigated the optimum drying condition for POM fraction in quantification of POM and/or mineral-associated organic matter (MOM; usually indirectly estimated). The influence of the drying conditions on quantifying POM was dependent upon soil properties, especially the amount of soil organic components. In relatively high organic soils (total carbon > 40 g/kg in this study), the POM values were significantly higher (overestimated) with drying at 55°C than those values at 105°C, which were, for example, 173.2 and 137.3 mg/kg, respectively, in a soil studied. However, drying at 55°C for longer than 48 hrs of periods produced consistent POM values even though the values were much higher than those at 105°C. Thus, indirect estimates of MOM (MOM = SOM−POM) also tended to be significantly impacted by the dry conditions. Therefore, we suggest POM fractions should be dried at 105°C for 24 hrs as determining POM and MOM contents. If the POM fraction is needed to be dried at a lower temperature (e.g. 55°C) with a specific reason, at least 48 hrs of drying period is necessary to obtain consistent POM values, and a moisture correction factor should be determined to adjust the values back to a 105°C weight basis.

Key Words: drying condition, soil organic matter, particulate organic matter, mineral-associated organic matter

INTRODUCTION

Soil organic matter (SOM) is an important component in regulating soil quality and a source of nutrients. Infiltration, water retention, soil structure, cation exchange capacity, and adsorption and/or deactivation of agricultural chemicals are affected by SOM[1].

Soil organic matter can be physically separated into different particle-size fractions by wet-sieving procedures[2-4]. Although the various fractionation methods have been designated in the literature, two fractions, particulate organic matter (POM) and mineral-associated organic matter (MOM), are most commonly used. The POM content in a soil is used as an indicator of soil quality because it changes rapidly with alterations in land use and soil management[5]. The POM fraction (POM + sand) is generally defined as the SOM fraction ranging in size from 0.05 to 2.0 mm, and the remaining finer SOM (< 0.05 mm) fraction is assumed to be MOM fraction (MOM + clay and silt)[3]. An accurate quantification of POM in soils is also important because it affects the indirect quantification of MOM (MOM = SOM - POM).

However, the methodologies commonly accepted to determine the content of POM in soils have not been standardized. Researchers have used similar procedures to isolate POM and/or MOM fractions, but several different drying temperatures and durations for the determination of POM and/or MOM contents using weight loss-on-ignition (WLOI) after the wet-sieving fractionation have been applied. For example,

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drying temperatures ranging from 40 to 80°C with different drying periods, from overnight to 72 hrs, have been used; moreover, some researchers only listed a drying temperature, but did not mention how long the POM fraction was dried for (Table 1)[3,6-16]. For this reason, the quantifications of POM and MOM have been estimated on a different weight basis.

The content of POM is usually calculated by the following equation after wet-sieving size fractionation[5]:

POM (mg/g) = [(POM fraction weight dried at 55°C for 24 hrs - POM fraction weight after 4 hrs at 450°C) / Soil weight dried at 55°C for 24 hrs] x 1000 [Eqn. 1]

Many researchers have adopted this equation to determine POM content using WLOI[17,18]. Most of researchers have ignored a moisture correction factor in the calculations of their results for the organic components obtained by WLOI with the wet-sieving size fractionation method; only a few researchers used a set of subsamples oven-dried at 105°C to calculate a moisture correction[19,20]. Thus, the standardization or optimization of drying conditions for quantifying POM and MOM is necessary.

The objectives of this study were to investigate the influence of different drying conditions on quantifying POM using a weight loss-on-ignition method after wet-sieving size fractionation and to find an optimum drying condition for POM fraction to quantify POM and MOM in soils.

Table 1. Comparison of drying conditions used by various researchers for POM fractionation

Drying Temperature (°C)	Drying Time (hrs)	References (examples only)
40	24	6
50	overnight	3
50	48	7
55	72	8
60	48	9
60	overnight	10, 11
65	12	12
70	24	13
75	48	14
80	overnight	15
80	16	16

MATERIALS AND METHODS

Soils

The surface 15 cm (A and/or Ap horizons) of seven South Dakota soils were used in this research: Buse (Typic Calciudolls), Egan (Udic Haplustolls), Egeland (Calcic Hapludolls), Fordville (Pachic Hapludolls), Forman (Calcic Argiudolls), Lowry (Typic Haplustolls), and Parnell (Vertic Argiaquolls). Also a soil sample containing compost was collected from the base of a yard-waste composting operation at the Brookings City Landfill in Brookings, SD. The material consisted of a mixture of mature, composted yard-waste and soil materials from various sources. All eight-soil samples were air-dried and crushed to pass a 2-mm sieve. Selected physical and chemical properties of the soils studied are presented in Table 2.

Determination of SOM

Soil organic matter was determined using a weight loss-on-ignition (WLOI) modified from the method proposed by Cambardella et al.[5] and Schulte[21]. Thirty grams of the soil samples were weighed in 50 *ml* high-form crucibles. The samples were dried at 105°C for 24 hrs and their weight checked and recorded. Finally the samples were ignited at 450°C for 4 hrs and SOM calculated using equation 2:

SOM_{105} (g/kg) = [(Soil weight dried at 105°C - Soil weight after 4 hrs at 450°C) / Soil weight dried at 105°C] x 1000 [Eqn. 2]

Determination of POM

The procedure for separating the POM fraction was modified from the method described by Cambardella and Elliott[3]. Two sets of quadruplicated soil samples were used for two different drying cycles. Thirty grams of air-dried soil (< 2.0 mm) were placed into a 125 *ml* plastic bottle with 90 *ml* of 0.5% sodium-hexametaphosphate solution. The bottles were shaken at 120 rpm overnight. The resulting dispersed samples were wet sieved through a 0.053-mm sieve using several rinses with deionized water. The POM fraction retained on the sieve (POM and sand) was transferred into a 50-*ml* high-form crucible with deionized water. The wet-POM fraction in the crucible was dried using different temperatures and durations. POM contents

Table 2. Selected physical and chemical properties of soils

Soils	Taxonomic Class	Particle Size Distribution (%)			Dry Aggregate Stability (g/kg)	pH (soil:water ,1:2, w/v)	Soil Organic Matter (g/kg)	Total Nitrogen (g/kg)	Total Carbon (g/kg)	C/N Ratio
		Sand	Silt	Clay						
Lowry	Typic Haplustolls	15.9	69.3	14.8	731.7	7.94	30.2	1.24	14.0	11.4
Egeland	Calcic Hapludolls	59.6	28.9	15.5	911.3	5.98	31.6	1.14	12.7	11.2
Egan	Udic Haplustolls	5.5	63.7	30.8	983.6	6.36	66.6	2.37	29.0	12.2
Forman	Calcic Argiudolls	24.4	42.8	32.7	980.7	7.55	67.4	2.31	29.8	12.9
Buse	Typic Calciudolls	39.1	30.9	30.0	996.5	7.60	79.2	2.97	40.8	13.7
Fordville	Pachic Hapludolls	24.8	45.8	29.4	989.9	7.33	92.9	3.61	43.8	12.1
Parnell	Vertic Argiaquolls	13.8	49.3	36.9	991.8	7.85	269.4	12.42	163.8	13.2
Compost	-	47.7	34.3	18.0	941.6	7.77	155.6	9.08	106.1	11.7

Table 2. continued

Soils	County in South Dakota, USA	Parent Material	Latitude	Longitude	Land Use	Plant / Crop Rotation
Lowry	Hughes	Loess	44^{O} 30'N	100^{O} 31'W	Cultivated	Winter wheat/Fallow
Egeland	Brookings	Loamy glaciofluvial sediments	44^{O} 26'N	96^{O} 52'W	Cultivated	Corn/Soybean
Egan	Moody	Silty drift over loamy till	44^{O} 11'N	96^{O} 46'W	Cultivated	Soybean/Corn
Forman	Deuel	Loamy till	44^{O} 54'N	96^{O} 30'W	Cultivated	Corn/Soybean
Buse	Brookings	Loamy till	44^{O} 30'N	96^{O} 34'W	Pasture	Bluegrass/Bromegrass
Fordville	Brookings	Loamy alluvial sediments over glacial outwash	44^{O} 18'N	96^{O} 42'W	Cultivated	Soybean/Corn
Parnell	Brookings	Local clayey alluvium	44^{O} 31'N	96^{O} 06'W	Native wetland	Cattails/Bulrushes
Compost	Brookings	Landfill materials	44^{O} 21'N	96^{O} 45'W	Compost	None

due to the different drying conditions were determined using a WLOI method[5].

Drying procedures for POM fraction

In the drying cycles applied in this study, two different temperatures were adopted; 55°C as a low drying temperature[5,8] and 105°C as a high drying temperature generally used in the procedure of SOM determination.

Step 1. Initial drying (ID): dry in an oven set at 55 and 105°C for 24 hrs.

Step 2. Re-wetting (RW): Re-wet POM fraction by adding 20 to 30 ml of deionized water, based on the amount of POM fraction, for at least 12 hrs.

Step 3. Re-drying (RD): dry in an oven set at 55°C for 24, 48, 72, 96, and 120 hrs.

The step 2 and 3 were to examine possible physical impacts at higher drying temperature (105°C) on the POM content after finishing ID; for example, volatilization of certain compounds in the POM material at this high temperature.

Step 4. Final drying (FD): dry in an oven set at 105°C for 24 hrs.

After steps 1, 3, and 4 the weight of the dried POM fraction was measured. After completing the drying steps, the crucibles were placed in a muffle furnace and heated to 450°C for 4 hrs to determine the ash content of the POM fraction. After ashing the POM fraction, the crucibles were allowed to cool in a desiccator and then the remaining residue was weighed.

Calculations for POM

POM fractions were calculated as follows:

POM fraction and soil weight after drying at 55°C[5)]

POM_{55} (g/kg) = [(POM fraction weight dried at 55°C - POM fraction weight after 4 hrs at 450°C) / Soil weight dried at 55°C] x 1000 [Eqn. 3]

POM fraction and soil weight after drying at 105°C

POM_{105} (g/kg) = [(POM fraction weight dried at 105°C - POM fraction weight after 4 hrs at 450°C) / Soil weight dried at 105°C] x 1000 [Eqn. 4]

RESULTS AND DISCUSSION

The influence of different drying cycles on the estimated values of POM in relatively low organic soils (total carbon < 30 g/kg in this study) is presented in Table 3. The POM values here range from 3.9 to 9.3 g/kg and from 3.8 to 9.0 g/kg at 55°C and 105°C, initially dried (ID) weight bases, respectively. There is no significant difference between the two initial drying temperatures. The estimated amounts of POM in most of the low organic soils were not significantly different between the two different drying cycles (55-55-105°C and 105-55-105°C cycles). There were also no significant differences in POM contents between the samples initially dried (ID) at 105°C and finally dried (FD) at 105°C, which ranged from 3.78 to 8.98 g/kg with ID and from 3.76 to 8.90 g/kg with FD in the low organic soils tested. Nevertheless, significant differences in POM amounts were present in a few samples when the low organic soil samples were dried at 55°C during ID or re-dried (RD) after rewetting (RW) the samples as compared to drying at 105°C during ID or FD. For example, the POM contents in

Table 3. Influence of different soil drying cycles on particulate organic matter (POM) in relatively low organic soils

Soils	Dry Cycle‡	Contents of Particulate Organic Matter (g/kg)†						
		ID	RD					FD
		24 hrs	24 hrs	48 hrs	72 hrs	96 hrs	120 hrs	24 hrs
Lowry	55-55-105	7.65 b§	7.78 a	7.49 c	7.48 c	7.47 c	7.48 c	7.15 d
	105-55-105	7.17 b	7.50 a	7.41 ab	7.31 ab	7.23 b	7.20 b	7.14 b
	Difference¶	*	**	ns	*	*	*	ns
Egeland	55-55-105	3.89 a	4.04 a	3.93 a	3.89 a	3.90 a	3.84 a	3.76 a
	105-55-105	3.78 a	3.98 a	3.89 a	3.90 a	3.85 a	3.84 a	3.76 a
	Difference	ns	ns	ns	ns	ns	ns	ns
Egan	55-55-105	9.29 a	9.41 a	9.25 a	9.17 a	9.20 a	9.13 a	8.98 a
	105-55-105	8.98 b	9.25 a	9.18 ab	9.14 ab	9.08 ab	9.08 ab	8.90 b
	Difference	ns	ns	ns	ns	ns	ns	ns
Forman	55-55-105	7.18 a	7.34 a	7.12 a	7.10 a	7.12 a	7.05 ab	6.77 b
	105-55-105	6.80 cd	7.16 a	7.09 ab	7.00 bc	6.95 c	6.92 c	6.76 b
	Difference	ns	ns	ns	ns	ns	ns	ns

† ID = initial drying at 55 or 105°C; RD = re-drying at 55°C with increase in drying time after re-wetting the samples; FD = final drying at 105°C.

‡ 55-55-105 = drying cycle of ID at 55°C, RD at 55°C, and FD at 105°C; 105-55-105 = drying cycle of ID at 105°C, RD at 55°C, and FD at 105°C.

§ Mean values (n = 4) within same drying cycle (a row) not followed by the same letter differ significantly (0.05 level) from each other as determined by Student's *t* test.

¶ Denotes significance between the values in each step of two different drying cycles for each soil at the 0.05 (*) and 0.01 (**) probability levels, respectively; ns = non-significant.

Lowry and Forman soils with different drying temperatures were significantly different, which was between 7.65 and 7.15 g/kg and between 7.18 and 6.77 g/kg, respectively.

However, the differences of POM contents determined due to the different drying conditions were much more obvious in relatively high organic soils (total carbon > 40 g/kg in this study). Influence of the different drying cycles on calculated POM contents in the high organic soils are given in Table 4. The estimated POM contents ranged from 24.1 to 173.2 and from 23.0 to 137.3 g/kg at 55°C and 105°C ID weight basis, respectively. All of the POM values were significantly lower with drying at 105°C than with drying at 55°C during ID, and the difference in POM value increased with increasing POM content in the soil. After finishing the ID step, the POM contents obtained from RD and FD were not different between two different drying cycles, 55-55-105°C and 105-55-105°C. There was no difference in the POM contents during the RD no matter how long the duration was, even though the POM estimates decreased with increasing drying period at 55°C during the RD stage of the experiment. Also, there were no significant differences between POM values estimated at 105°C drying condition before (ID) or after (FD) the rewetting period. These results indicated that the differences in POM values estimated due to 105°C versus 55°C drying conditions, regardless of the duration, were dependent on excess moisture in the samples. Also, there was no physical impact, such as volatilization of certain compounds in POM materials, at 105°C on the POM content.

The POM estimates of the high organic soils, such as Parnell soil were markedly influenced by drying temperature and duration than those of the low organic soils, such as the Egeland soil. The POM contents obtained by the WLOI method were highly correlated with the amounts of particulate organic carbon (POC) and particulate carbon (PC, particulate organic and inorganic carbon), R^2 = 0.943 and 0.782, respectively, obtained by a dry combustion method[22]. In contrast, the contents of POM were reported to be weakly correlated (R^2 = 0.215) with POC contents[5]; these results might be caused from as follows: 1) their POM and POC data shown were not feasible at some points

Table 4. Influence of different soil drying cycles on particulate organic matter (POM) in relatively high organic soils

Soil	Dry Cycle‡	Contents of Particulate Organic Matter (g/kg)†						
		ID	RD					FD
		24 hrs	24 hrs	48 hrs	72 hrs	96 hrs	120 hrs	24 hrs
Buse	55-55-105	24.13 a§	24.24 a	24.02 a	23.89 a	23.92 a	23.86 a	23.01 b
	105-55-105	23.02 c	24.15 a	24.03 ab	23.95 ab	23.86 b	23.85 b	22.92 c
	Difference¶	*	ns	ns	ns	ns	ns	ns
Fordville	55-55-105	25.15 a	25.22 a	24.76 a	24.75 a	24.74 a	24.71 a	23.43 b
	105-55-105	23.42 b	25.08 a	25.04 a	24.98 a	24.93 a	24.88 a	23.44 b
	Difference	**	ns	ns	ns	ns	ns	ns
Compost	55-55-105	85.41 a	85.42 a	84.66 ab	84.57 ab	83.99 b	83.97 b	80.38 c
	105-55-105	80.39 b	85.22 a	84.88 a	84.49 a	84.36 a	84.34 a	80.82 b
	Difference	**	ns	ns	ns	ns	ns	ns
Parnell	55-55-105	173.18 a	166.20 b	149.64 c	146.74 c	145.56 c	143.66 c	136.96 d
	105-55-105	137.32 d	162.45 a	150.00 b	146.48 bc	145.55 c	143.77 c	137.25 d
	Difference	**	ns	ns	ns	ns	ns	ns

† ID = initial drying at 55 or 105°C; RD = re-drying at 55°C with increase in drying time after re-wetting the samples; FD = final drying at 105°C.

‡ 55-55-105 = drying cycle of ID at 55°C, RD at 55°C, and FD at 105°C; 105-55-105 = drying cycle of ID at 105°C, RD at 55°C, and FD at 105°C.

§ Mean values (n = 4) within same drying cycle (a row) not followed by the same letter differ significantly (0.05 level) from each other as determined by Student's *t* test.

¶ Denotes significance between the values in each step of two different drying cycles for each soil at the 0.05 (*) and 0.01 (**) probability levels, respectively; ns = non-significant.

because POC values for some soils (Valentine and Brookings soil series) were as high as or higher than POM values, and 2) they used a low temperature (55°C) and short duration (24 hrs) for drying the POM fraction, so that these drying conditions may have caused some erratic results in POM and POC determinations.

The contents of MOM in soil are usually determined indirectly using two parameters, POM and SOM contents. Therefore, it is imperative to know the effects of the drying conditions used for quantifying POM because the other parameter, SOM content, is commonly estimated with 105°C dry weight basis. The influence of drying temperature used for quantifying POM on the indirect estimation of MOM content is shown in Table 5. The range of SOM values for the soils used was between 30 and 270 g/kg (105°C dry-weight basis). The MOM values using SOM_{105} and POM_{55} were not significantly different when compared with those calculated using SOM_{105} and POM_{105} in the relatively low organic soils. However, the MOM values were always significantly higher in the relatively high organic soils. The difference as affected by the drying conditions increased with increasing soil organic components, such as SOM. The MOM values ranged from 22.5 to 96.3 g/kg that estimated by SOM_{105} and POM_{55}, and from 23.0 to 132.1 g/kg that calculated by SOM_{105} and POM_{105} in the different soils. The results of this study demonstrate that POM and MOM values in soils containing high organic matter are significantly affected by the drying conditions used in the POM determination procedure. The organic matter has been demonstrated to increase the water holding capacity of a mineral soil[23]. Plant available water holding capacity in a soil was reported to be more than doubled when the organic matter content increased from 0.5 to 3.0%[24]. This held true for all textural groups. Therefore, thorough drying of soils and separates during the processes of POM determination procedure is an important factor to consider, especially for soils containing high organic matter.

CONCLUSIONS

The moisture condition of soil and POM fraction needs to be known in order to determine POM and MOM contents in soils using WLOI. Different drying conditions for the soil and POM fraction with incomplete dryness cause over- or underestimated values of the target organic material content. The difference in POM values as affected by the drying conditions increased with increasing soil organic matter content. Erratic result of POM content will cause the incorrect estimation of MOM content in soil because MOM content is generally determined by indirect estimation (SOM - POM = MOM). Based on the results of this study, the optimal drying temperature and duration to be acceptable for determining POM and MOM in soils is 105°C for 24 hrs. If there is a specific reason to use a low temperature (50 to 60°C), especially for soil samples containing high organic matter, at least 48 hrs of drying periods is necessary to obtain consistent POM and MOM values even though the values will be overestimated or underestimated. Also, the dry-weight basis of POM fraction should be adjusted with a moisture correction factor estimated from drying the sub-sample at 105°C.

Table 5. Estimates of MOM using POM and SOM values from different drying temperatures

Organic Matter†	Dry Temperature Basis	Soils							
		Lowry	Egeland	Egan	Forman	Buse	Fordville	Compost	Parnell
SOM (g/kg)	105°C	30.19	31.60	66.57	67.44	79.17	92.90	158.55	269.44
POM (g/kg)	105°C	7.17	3.78	8.98	6.80	23.02	23.42	80.39	137.32
	55°C	7.65	3.89	9.29	7.18	24.13	25.15	85.41	173.18
MOM (g/kg)	SOM_{105} - POM_{55}	22.54 a‡	27.70 a	57.28 a	60.25 a	55.04 a	67.75 a	73.15 a	96.26 a
	SOM_{105} - POM_{105}	23.02 b	27.82 a	57.59 a	60.64 a	56.15 b	69.48 b	78.16 b	132.13 b

† SOM = soil organic matter; POM = particulate organic matter; MOM = mineral-associated organic matter.
‡ Mean values (n = 4) within a MOM-column in each soil not followed by the same letter differ significantly (0.05 level) from each other as determined by Student's *t* test.

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