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A COMPARISON OF WET
AND DRY ASHING METHODS FOR
ELEMENTAL ANALYSIS OF PEAT AND MOSSES

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ELEMENTAL ANALYSIS OF PEAT AND MOSSES

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ABSTRACT

Peat and moss samples were subjected to wet and dry ashing and analyzed for the elements K, Ca, Mg, Cu, Zn, Mn, Fe, and Pb using atomic absorption spectrometry. Wet ashing was found to give somewhat higher analytical results than dry ashing, and lower coefficients of variation.

INTRODUCTION

The elemental analysis of peat and mosses is of interest for several reasons, including ecological studies of peatlands (Chapman, 1964; Malmer and Sjörs, 1955; Sonnesson, 1920; Stanek and Jeglum, 1977) and geochemical prospecting (Eriksson and Eriksson, 1976; Tanskanen, 1976; Usik, 1969). Mosses are of interest due to their possible use as indicators of atmospheric input of potential pollutants (Pakarinen and Tolonen, 1976). Such studies usually emphasize the total elemental content of peat compared with studies using selective extractants for specific nutrients (Boatman and Roberts, 1963; Maynard and Fletcher, 1973).

One problem is that researchers utilize different means of sample preparation and analytical methods. For example, dry ashing of samples (Chapman, 1964; Malmer and Sjörs, 1955), and wet ashing (Casagrande and Erchull, 1976; Ericksson and Ericksson, 1976; Maynard and Fletcher, 1973; Pakarinen and Tolonen, 1976; Stanek and Jeglum, 1977) followed by either flame photometric or atomic absorption analyses. Spectrographic procedures for peat analysis were used by Tanskanen (1976).

In order to evaluate the merits of dry and wet ashing of such peat and moss samples, the following study was carried out utilizing four peats and two moss species.

METHODS AND MATERIALS

The samples utilized in this study are listed in Table 1, along with a brief environmental description. The samples were collected in the peatlands surrounding Kinoje Lake, in the Hudson Bay Lowlands of northern Ontario (51°34'N, 108°00'W). Samples were collected in the field and stored in plastic bags at 4°C prior to analysis. Also in the field, pH and von Post (1922) humification readings were made. The samples were dried at 90°C for 48 hours, and ground to 80-mesh using a Wiley grinding mill.

For dry ashing, 1 g of sample was weighed into a high-form porcelain crucible, placed into a muffle furnace, and ashed at 500°C for two hours. The sample was allowed to cool. The ash was wetted with ten drops of double distilled H₂O, and 4 ml of HNO₃ (1+1) was added. Excess HNO₃ was evaporated on a hot plate set at 120°C. The crucible was placed in the furnace again for one hour at 500°C, removed, allowed to cool, 10 ml HCl (1+1) added, and the contents transferred to a 50 ml volumetric flask and made up to volume with 1N HCl. One gram of the samples to be wet-ashed was placed into a 150 ml Erlenmeyer flask. To this, 10 ml of HNO₃ was added and allowed to sit. Then 3 ml of 60% HClO₄ was added and the samples heated slowly on a hot plate until the HNO₃ was nearly evaporated. The sample was heated until the white fumes of HClO₄ were visible. The sample was then cooled, and 10 ml HCl (1+1) added, and the contents transferred to a 50 ml flask.

All analyses were carried out using a Varian Techtron AA-5 atomic absorption spectrophotometer. Samples were treated with 10 ml of 5% lanthanum solution to prevent interferences. Standard solutions used

were made up in a manner parallel to that of the field sample solution. To determine the difference between wet and dry ashing results, significance of difference between means was established by use of a T-test.

RESULTS

Our results, as presented in Table 2, indicate little difference between wet and dry ashing. When statistically significant analytical results were found, the wet ashing procedure usually yielded a somewhat higher value, i.e. K - sample 1, Ca - sample 7, Mg - 4 and 7, Zn - 2, Mn - 6, Fe - 1, 3 and 6, and pb - 5. The biggest discrepancies were found for Fe, Pb (sample 5) and K (sample 1 only), while the other wet ashing values were generally less than 10% greater. No consistent results were found with sample type as given in Table 1, except that the poorly humified peat sample (#1) and the NBS Orchard Leaf Standard (#7) had the most number of statistically significant discrepancies; three each, while sample 5, the *Sphagnum capillaceum* moss had no discrepancies between wet and dry ashing.

In terms of analytical accuracy, we can only cite the results of NBS Orchard Leaf Standard (sample 7). The NBS reported value was greater for the elements Ca, Mg (dry ash) and Mn, while our results were not different for Cu, Zn, Fe, and Pb. Our dry ashing value for K in the NBS standard was somewhat higher. As for the precision of other samples, Table 2 presents data on coefficient of variations and 95% confidence limits.

Comparing our NBS Orchard Leaf data with the results of a collaborative study of Isaac and Johnson (1975), our C.V. data were approximately the same or lower for most results. We found a higher C.V. for K by dry ashing (4.8 vs 2.1%), Mg by dry ashing (5.1 vs 3.4%), and Cu by wet ashing (16.1 vs 6.4%). Therefore, we feel our results to be acceptable for precision on the NBS standard. In terms of the peats and mosses, no definite trends were noticed for C.V.'s except dry ashing exhibited somewhat higher C.V.'s for K, Cu (except sample 6) and Zn, while Ca and Pb values were higher for wet ashing. Isaac and Johnson (1975) found the same trends except they did not analyze their samples for Pb. We also did not observe any definite effect of type of sample.

In general, dry ashing has been felt to be a satisfactory method of plant sample preparation. Authors such as Isaac and James (1972), Adrian (1973), and Isaac and Johnson (1975) show comparable results between dry ashing and other procedures. However, occasionally, dry ashing can lead to volatile element loss, i.e. Isaac and Jones (1972) report losses of K and possibly Cu, and Webber (1972) reported losses of Pb. Our data indicate wet ashing gave somewhat consistently higher results except the poor results with Cu in sample 2, where the wet ashing was 50% lower. Therefore, we recommend that wet ashing is a better procedure for peats and mosses both in terms of precision and accuracy.

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TABLE 1. Description of samples used in study.

| Sample No. | Sample description |
|------------|--|
| 1 | Peat collected to -20 cm depth from low shrub bog pH of sample 3.3 with von Post reading of 1-2. |
| 2 | Peat from 20-40 cm depth from same pit as sample #1. pH was 3.0 with van Post reading of 4. |
| 3 | Peat from black spruce island, 0-20 cm depth, pH = 3.1, van Post reading of 4. |
| 4 | Peat from 0-20 cm depth collected from graminoid fen. Sample pH was 5.8 with van Post reading of 3. |
| 5 | <u>Sphagnum capillaceum</u> moss. |
| 6 | <u>Sphagnum fuscum</u> moss. |
| 7 | NBS Orchard Leaf sample. |

TABLE 2. Analytical Results of Wet vs Dry Ashing of Samples. Means Based on 5 Replicates

| Element | Sample No. | Wet Ashing | | | Dry Ashing | | | Significance of ashing procedures | NBS value |
|------------------------|------------|------------|----------------------|--------|------------|----------------------|--------|-----------------------------------|-----------|
| | | \bar{X} | 95% Confidence Limit | % C.V. | \bar{X} | 95% Confidence Limit | % C.V. | | |
| K(%) | 1 | .08 | .03 | 10.7 | .06 | .03 | 17.7 | s. | 1.47 |
| | 2 | .03 | 0 | 0 | .03 | .03 | 34.4 | n.s. | |
| | 3 | .06 | .01 | 9.8 | .06 | .05 | 27.1 | n.s. | |
| | 4 | .01 | .01 | 37.3 | .02 | .03 | 61.2 | n.s. | |
| | 5 | .21 | .05 | 11.2 | .19 | .08 | 13.8 | n.s. | |
| | 6 | .17 | .03 | 8.3 | .14 | .08 | 18.2 | n.s. | |
| | 7 | 1.46 | .10 | 3.1 | 1.60 | .21 | 4.8 | s. | |
| Ca(%) | 1 | .18 | .10 | 21.9 | .18 | .03 | 5.6 | n.s. | 2.09 |
| | 2 | .17 | .10 | 21.7 | .17 | .05 | 13.6 | n.s. | |
| | 3 | .76 | .21 | 10.3 | .74 | .21 | 10.8 | n.s. | |
| | 4 | 1.64 | .28 | 7.0 | 1.64 | .28 | 7.0 | n.s. | |
| | 5 | .15 | .08 | 18.3 | .13 | .05 | 18.8 | n.s. | |
| | 6 | .20 | .08 | 15.4 | .19 | .03 | 6.8 | n.s. | |
| | 7 | 1.93 | .26 | 5.1 | 1.79 | .13 | 2.7 | s. | |
| Mg(%) | 1 | .07 | .03 | 8.3 | .06 | .03 | 8.6 | n.s. | .62 |
| | 2 | .05 | .01 | 8.6 | .05 | 0 | 0 | n.s. | |
| | 3 | .10 | .02 | 8.2 | .10 | .01 | 5.7 | n.s. | |
| | 4 | .10 | .01 | 5.7 | .09 | .02 | 7.9 | s. | |
| | 5 | .09 | .03 | 6.4 | .08 | .03 | 10.2 | n.s. | |
| | 6 | .05 | .01 | 10.1 | .05 | .02 | 14.1 | n.s. | |
| | 7 | .60 | .05 | 3.6 | .56 | .08 | 5.1 | s. | |
| Cu ($\mu\text{g/g}$) | 1 | 2.68 | 1.47 | 21.3 | 2.06 | 1.65 | 31.0 | n.s. | 25 |
| | 2 | 1.58 | .49 | 12.2 | 3.16 | 3.45 | 42.3 | s. | |
| | 3 | 1.30 | .49 | 14.4 | 1.90 | 2.26 | 46.6 | n.s. | |
| | 4 | 2.70 | .75 | 10.8 | 4.72 | 6.51 | 83.5 | n.s. | |
| | 5 | 3.30 | 1.05 | 12.3 | 4.38 | 4.37 | 38.8 | n.s. | |
| | 6 | 4.80 | 11.34 | 92.0 | 4.16 | 5.22 | 48.7 | n.s. | |
| | 7 | 11.68 | 4.83 | 16.1 | 12.30 | 6.86 | 21.7 | n.s. | |
| Zn ($\mu\text{g/g}$) | 1 | 39.08 | 2.75 | 2.7 | 37.08 | 10.36 | 10.9 | n.s. | 91 |
| | 2 | 27.08 | 2.03 | 2.9 | 24.76 | 2.16 | 3.4 | s. | |
| | 3 | 14.38 | .93 | 2.5 | 13.38 | 5.35 | 15.5 | n.s. | |
| | 4 | 29.06 | 3.65 | 4.9 | 29.30 | 9.72 | 12.9 | n.s. | |
| | 5 | 23.90 | 3.34 | 5.4 | 23.88 | 4.96 | 8.1 | n.s. | |
| | 6 | 26.91 | 2.67 | 3.8 | 27.98 | 10.67 | 14.8 | n.s. | |
| | 7 | 25.96 | 7.07 | 10.6 | 28.52 | 11.93 | 16.3 | n.s. | |
| Mn ($\mu\text{g/g}$) | 1 | 157.4 | 20.62 | 5.1 | 152.0 | 20.95 | 5.4 | n.s. | 300 |
| | 2 | 18.0 | 1.83 | 3.9 | 17.2 | 3.34 | 7.6 | n.s. | |
| | 3 | 50.2 | 3.81 | 3.0 | 48.4 | 4.29 | 3.5 | n.s. | |
| | 4 | 108.4 | 5.91 | 2.1 | 106.2 | 9.77 | 3.5 | n.s. | |
| | 5 | 373.6 | 37.54 | 3.9 | 370.3 | 30.59 | 3.2 | n.s. | |
| | 6 | 400.4 | 71.22 | 6.9 | 383.3 | 36.77 | 3.7 | s. | |
| | 7 | 87.2 | 4.94 | 2.2 | 81.4 | 7.84 | 3.7 | n.s. | |
| Fe ($\mu\text{g/g}$) | 1 | 644 | 133 | 8.0 | 527 | 169 | 12.5 | s. | 300 |
| | 2 | 547 | 150 | 10.7 | 530 | 333 | 24.4 | n.s. | |
| | 3 | 1632 | 180 | 4.3 | 1522 | 138 | 3.5 | s. | |
| | 4 | 15262 | 2639 | 6.7 | 14858 | 1210 | 3.2 | n.s. | |
| | 5 | 219 | 191 | 33.9 | 188 | 139 | 28.7 | n.s. | |
| | 6 | 284 | 44 | 6.0 | 211 | 140 | 25.9 | s. | |
| | 7 | 283 | 39 | 5.4 | 280 | 41 | 5.7 | n.s. | |
| Pb ($\mu\text{g/g}$) | 1 | 22.4 | 4.5 | 7.8 | 18.4 | 5.7 | 12.0 | s. | 45 |
| | 2 | 8.2 | 7.8 | 37.2 | 5.9 | 6.0 | 39.5 | n.s. | |
| | 3 | 3.0 | 3.3 | 42.2 | 3.8 | 3.1 | 32.5 | n.s. | |
| | 4 | 18.5 | 13.6 | 28.7 | 19.6 | 17.9 | 35.5 | n.s. | |
| | 5 | 16.1 | 13.3 | 32.2 | 14.1 | 6.0 | 16.5 | n.s. | |
| | 6 | 17.5 | 11.6 | 25.8 | 14.5 | 5.6 | 15.0 | n.s. | |
| | 7 | 42.9 | 17.9 | 16.2 | 45.2 | 20.2 | 17.4 | n.s. | |

* n.s. = means not significantly different (at $P < .05$ confidence limit).
s. = means significantly different.

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