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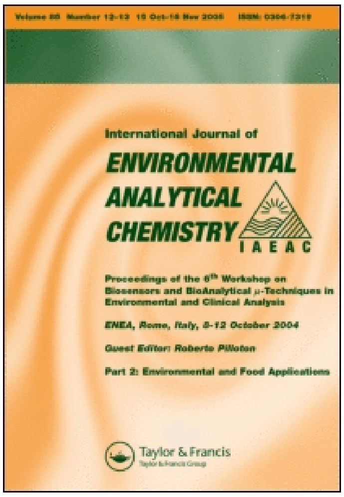

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Effect of Filtration and Centrifugation on the Determination of Aluminium in Water

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The effect of filtration and centrifugation on the determination of aluminium in surface and leachate water has been studied. For surface and leachate water samples only small effects were observed by filtration through 0.45 μm or 0.22 μm pore size membrane filters compared to unfiltered samples. For over 80% of the samples, which contained 0.01–15 mg Al L^{-1}, the differences between unfiltered and filtered samples were less than 0.10 mg Al L^{-1}, and only for about 5% of the samples were differences above 0.20 mg Al L^{-1} observed.

KEY WORDS: Aluminium, particulate matter, colloids, filtration, sampling, waters, spectrophotometry.

INTRODUCTION

The choice of sample pretreatment is a critical step in the development of procedures for determination of dissolved aluminium in water, as aluminium usually is present in various forms. The main difficulty is in distinguishing between dissolved aluminium species and particulate/colloidal aluminium hydroxides/silicates suspended in solution.[1] By such methods as atomic spectroscopy or neutron activation analysis, the sum of dissolved aluminium and aluminium in suspended particulates/colloids are determined. Conventional wet chemical methods such as spectrophotometry or fluorimetry are selective for monomeric species in solution, but it is common to acidify or digest the samples prior to determination of dissolved aluminium. In this way colloids and particulates may be partly or completely dissolved, leading to an overestimation of dissolved aluminium.

The problem of adequate sample pretreatment and filtration procedure has been dealt with in a number of papers.[2–10] In previous studies the amounts of aluminium retained by 0.45 μm or 0.10 μm pore size filters have been in the range of 0.01–0.10 mg Al L^{-1} for surface water.[2–8] Bergseth[10] found significant breakthrough of clay minerals using glassmicrofibre filters (Whatman GF/C, average pore size 1.2 μm). Significant differences (0.10–0.50 mg Al L^{-1}) were also found between filtrates from 0.45 μm compared to 0.10 or 0.05 μm pore size membrane filters. However, Bergseth used suspensions of soils (10 g to 100 ml water) to test the collection efficiency of the filters; his conclusions may therefore be misleading when applied to normal conditions for sampling of surface and leachate water containing relatively small amounts of particulate or colloidal matter.

In most of these previous studies only relatively few samples were examined, it is therefore difficult to draw general conclusions on the importance of an adequate filtration procedure. For this reason we examined the ffect of filtration on determination of aluminium in over 350 samples of surface and leachate water from podsolic forest soils and leachate water from soils with a higher concentration of clay minerals.

EXPERIMENTAL

Determination of aluminium

Aluminium in leachate and surface water was determined by a flow injection analysis spectrophotometric method using pyrocatechol violet as the chromogenic reagent.[11,12] In some samples with high concentrations of aluminium the determinations were performed by flame atomic absorption spectrometry under standard conditions.[13]

The group II samples were treated as follows: (a) not pretreated; (b) centrifuged (3000 rpm, 30 min.); (c) filtered through 0.45 μm or (d) 0.22 μm pore size cellulose acetate membrane filters (Millipore). The group I and III samples were only treated as (a) and (c). The samples were then preserved with 7 ml 37% w/v hydrochloric acid per litre and stored at 4°C. Before determination of aluminium by the flow injection analysis method, the water samples were digested by peroxydisulphate.[13]

Groups of samples

Three groups of samples were studied. Group I was runoff (collected weekly from May through October 1983) from three small watersheds in the southernmost part of Norway. The watersheds (plots 4, 5 and 6 in Seip *et al.*[14]) were all smaller than 100 m^2 and covered by shallow soil containing organic matter down to the lithic contact.

Group II samples were leachate from 20 lysimeters located at the Nordmoen field station 60 km north of Oslo. The lysimeters were 30 cm in diameter and 40 cm high and filled with four soil types (textural classes according to Sveistrup and Njøs[15]): medium sand (8 lysimeters), loam (4), coarse loamy sand (4) and medium loamy sand (4). In the spring 1984 some of the lysimeters of each soil type were treated with a herbicide, giving a strong nitrification and acidification. Leachate from all lysimeters was collected monthly or more frequently, depending on the amount of precipitation, in the frost-free period in 1984 and 1985.

Group III samples consisted of leachate from a pot experiment watered with nutrient solutions containing aluminium in the range of 0 to 160 mg L^{-1}. The pots were Buchner funnels 26 cm in diameter and 12 cm high with a nylon screen and a glassmicrofibre filter (No. 8, Schleicher and Schüll) at the bottom. Soil from the Bh- and the upper Bs1-horizons from a podsol profile was mixed and filled in the funnels (profile No. 2 in Stuanes and Sveistrup[16]). Leachates were sampled once a week in three months from a total of 24 funnels.

At the end of this experiment pore water was withdrawn by placing the soil in filter funnels connected to a vacuum pump. 100 ml of the pore water was then filtered (0.45 μm), the filter digested and the filterable acid soluble aluminium determined (Table II).

RESULTS AND DISCUSSION

Table I shows the results of a linear regression analysis for all the filtration data. Some of the results for the group II samples are also plotted in Figures 1–3. The samples containing aluminium above $2\,\mathrm{mg\,L^{-1}}$ (about 5% of the samples) were omitted in the figures to clarify the pattern in the range of 0–$2\,\mathrm{mg\,L^{-1}}$. Figures 1–3 show that most of the samples contain less than $1\,\mathrm{mg\,L^{-1}}$. The regression analysis of this data set was therefore repeated for only the samples in the range of 0–$1.0\,\mathrm{mg\,Al\,L^{-1}}$. Table I shows that there is little difference between filtered and unfiltered samples, as the slopes (b) are close to 1.00 and the intercepts (a) do not deviate much from

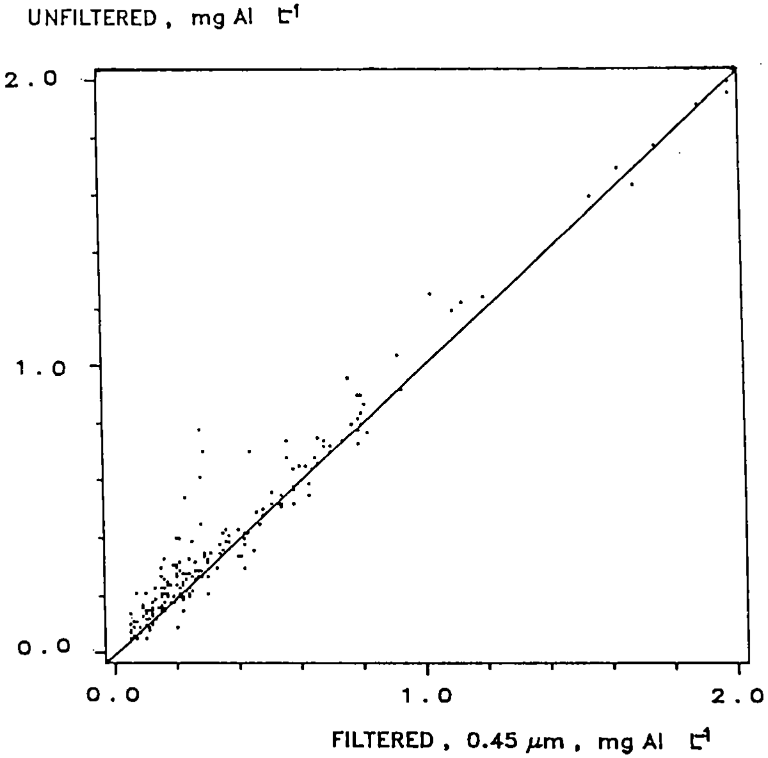

Figure 1 Comparison of aluminium found in leachate water (group II) before and after filtration (0.45 µm pore size). The line of unity are drawn up.

Table 1 The parameters for linear regression ($Y=a+bX$), their standard errors (s_a, s_b) and correlation coefficient (r) for the correlation between pairs of treatments of not filtered, centrifuged and filtered samples

Sample type	Treatment pairs (Y, X)		N	a	b	s_a	s_b	r
Group II	Not filtered	Centrifuged	226	0.020	0.980	0.0099	0.0059	0.996
	Not filtered	Filtered 0.45 μm	226	0.041	0.997	0.0063	0.0038	0.998
(all data	Not filtered	Filtered 0.22 μm	226	0.051	1.021	0.0077	0.0048	0.997
0.01–20	Centrifuged	Filtered 0.45 μm	226	0.026	1.012	0.0090	0.0054	0.997
mg Al L^{-1})	Centrifuged	Filtered 0.22 μm	226	0.037	1.034	0.0123	0.0076	0.994
	Filtered 0.45 μm	Filtered 0.22 μm	226	0.010	1.023	0.0063	0.0039	0.998
Group II	Not filtered	Centrifuged	192	0.005	1.011	0.0087	0.0218	0.958
	Not filtered	Filtered 0.45 μm	192	0.044	0.977	0.0094	0.0252	0.943
(only data	Not filtered	Filtered 0.22 μm	192	0.056	0.983	0.0095	0.0262	0.937
0.01–1.0	Centrifuged	Filtered 0.45 μm	192	0.048	0.932	0.0084	0.0224	0.949
mg Al L^{-1})	Centrifuged	Filtered 0.22 μm	192	0.058	0.940	0.0084	0.0231	0.947
	Filtered 0.45 μm	Filtered 0.22 μm	192	0.017	0.989	0.0055	0.0151	0.978
Group I	Not filtered	Filtered 0.45 μm	48	0.039	0.919	0.0265	0.0585	0.917
Group III	Not filtered	Filtered 0.45 μm	48	0.30	1.065	0.27	0.015	0.997

zero. The correlation coefficients (r) decreases from about 0.99 to about 0.95 when the samples above 1.0 mg Al L^{-1} are excluded, but the slopes do not change much (< 10%). Figures 1–3 show that most of the samples are situated close the line of unity, but there is a small trend indicating that the unfiltered (Figure 1) (or centrifuged (Figure 2)) samples are the highest. For over 80% of the samples in Figures 1–2 the difference between unfiltered (or centrifuged) and filtered samples is less than 0.10 mg Al L^{-1}. Only 5% of the samples deviate more than 0.20 mg Al L^{-1}. Figure 3 shows that there is little difference between results of filtration through 0.45 μm compared 0.22 μm pore size filters. This indicates that colloids in the size range

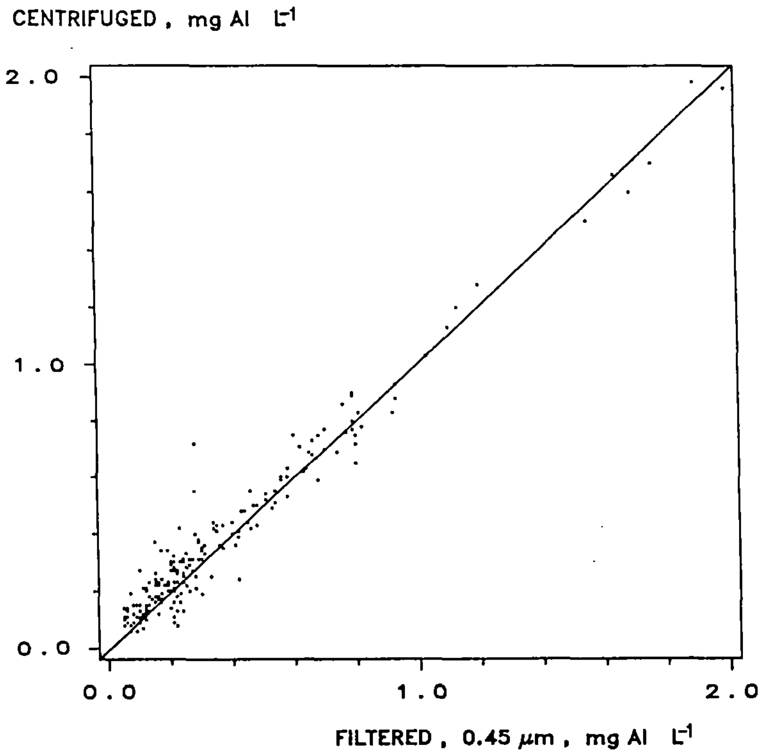

Figure 2 Comparison of aluminium found in leachate water (group II) in centrifuged and filtered samples (0.45 μm pore size). See Figure 1 for legend.

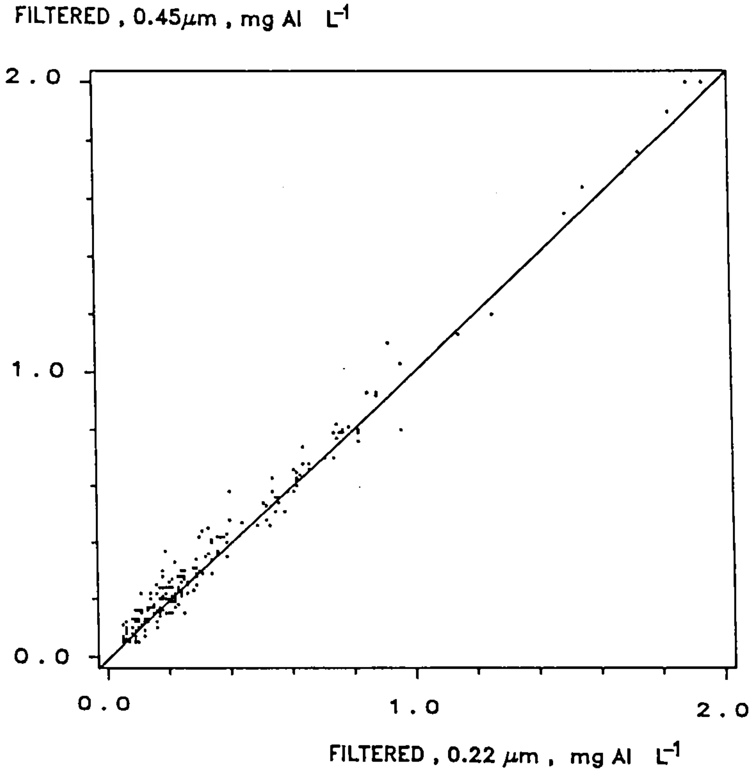

Figure 3 Comparison of aluminium found in leachate water (group II) in samples filtered through pore size 0.45 μm and 0.22 μm membrane. See Figure 1 for legend.

0.22–0.45 μm are not significant in the investigated samples, and that little of the scatter in Figures 1–2 can be explained by low reproducibility of the analytical method.

The results of the regression analysis of the group I and III samples (Table I) agreed well with the results discussed above for the group II samples. Patterns similar to those in Figures 1–2 were also found for the samples from group I and III.

The pore water withdrawn from the soil watered by aluminium containing nutrient solutions (Table II), contained filterable aluminium from 0.1–0.5 mg Al L ^{1}. These samples were withdrawn from a dis-

Table II Aluminium in pore water from a soil artificially watered by nutrient solutions containing aluminium[a]

Aluminium in nutrient solution	Total aluminium[b]		Filterable aluminium[b]	
	I	II	I	II
0	2.5	3.4	0.20	0.16
10	6.0	4.7	0.16	0.29
20	14.5	13.6	0.20	0.13
40	38.0	34.0	0.15	0.15
80	78.0	76.0	0.23	0.22
160	176	182	0.38	0.46

[a] pH of nutrient solution adjusted to 3.5. The pH of the pore water was 3.2–3.5.

[b] Concentrations in $\mathrm{mg\,Al\,L^{-1}}$. I and II are results from parallel treatments.

turbed soil profile (the wet soil was transferred to a filter funnel), and the pore water samples were also muddy by sight. The filterable aluminium presumably reflects suspended clay minerals (the filterable aluminium was not related to total amount of aluminium in the pore water).

CONCLUSIONS

Our leachate and surface water samples contained low amounts of particulate aluminium compounds which could be removed by $0.45\,\mu\mathrm{m}$ or $0.22\,\mu\mathrm{m}$ pore size filters. In most cases the filterable component contributed less than $0.10\,\mathrm{mg\,Al\,L^{-1}}$. These findings agree in most cases with earlier results, where concentrations of filterable aluminium compounds up to $0.10\,\mathrm{mg\,Al\,L^{-1}}$ have been found.[2–8] The pore water (from a disturbed soil profile) contained significantly higher amounts of filterable aluminium, although the figures in most cases were lower than the results obtained by Bergseth.[8] The values in Table II may represent a worse case situation were clay mineral particulates are suspended in solution.

It is, however difficult to draw general conclusions about the necessity of adequate filtration from this study. The need for filtration must be decided after careful evaluation of the samples. At total aluminium concentrations above $1\,\mathrm{mg\,Al\,L^{-1}}$ there is small risks of large errors due to inclusion of aluminium containing particulates. The need for filtration becomes important at lower concentrations of dissolved aluminium, where only small amounts of suspended particulate matter may lead to relatively large errors.

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