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Some Comments on the Determination of Fluvial Sediment Concentrations in the Laboratory

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ABSTRAK

Pemisahan enapan ampaian dalam air semula jadi di makmal biasanya di tentukan oleh kaedah penapisan atau analisis gravimetri. Jenis turas yang digunakan berbeza dari seorang pengguna ke yang lain, kos dan kawasan penurasan efektif yang di perlukan. Dalam eksperimen ini, empat jenis turas yang biasa digunakan dan di cadangkan oleh berbagai agensi dan penyelidik diuji. Ia adalah Whatman 542, Whatman GFC, Whatman "cellulose nitrate" dan Millipore "Type HA". Eksperimen ini melibatkan pengeringan turas-turas dalam ketuhar dengan suhu 105°C selama 2 jam, 24 jam dan pengabuan di relau dengan suhu 550°C selama 2 jam, suatu prosedur biasa dalam penentuan berat enapan mineral. Penimbangan yang dilakukan setelah turas-turas tersebut di masukki relau menunjukkan perbedzaan berat yang tertinggi sekali, secara purata, untuk Whatman GFC, sebanyak 0.0712g atau pun 3.63% berat asalnya, diikuti oleh Whatman "cellulose nitrate" dan Whatman 542 (0.0014g), dan Millipore "Type HA" (0.0004g).

ABSTRACT

The separation of suspended sediment in natural waters in the laboratories is usually determined by using the filtration method or gravimetric analysis. The type of filter used, however, differs from one user to another, the type being largely determined by the quality of data needed, cost or the effective filtering area required. In this experiment, four commonly used and recommended filters by various researchers and agencies in the country were tested. They are the Whatman 542, Whatman GFC, Whatman cellulose nitrate and Millipore type HA. The experiment involved drying the filters in the oven at 105°C for over 24 h and ashing in the muffle furnace at 550°C for 2h, a normal procedure in determining the mineral sediment weight by eliminating any organic matter present. After ashing, the Whatman GFC registered the highest mean weight loss of 0.0712g which is 3.63% of the original weight, followed by Whatman cellulose nitrate membrane and the Whatman 542 (0.0014g) and the Millipore Type HA (0.0004g).

INTRODUCTION

In the laboratory determination of suspended sediment in natural waters, the most common method used is the filtration technique or gravimetric analysis. (Task Committee 1970; WMO, 1974). Different types of filters have been used by various workers and governmental agencies in Malaysia as well as those outside the country (Table 1). Glass fibre filters are the most commonly used among governmental research departments in the country.

Selection of Filters

Essentially, in fluvial sediment analysis, the selection of filters is largely determined by the type of work undertaken. In estuarine studies, for example, where sediments are usually fine, McCave (1979) suggested the use of Nuclepore papers for low concentrations, while cellulose ester membranes of Sartorious, Oxoid and Millipore papers were said to be ideal if sediment concentrations are greater than 10 mg/l. Or, if organic rocks are present in the study area, combustible filter

TABLE 1
Filters used and recommended in work on natural waters in Malaysia and other countries

Type	retention/pore size (um)	diameter (cm)	applications	source	used in fluvial sediment research
Whatman 542 quantitative filter paper	2.7	12.5	high retention of finest particulates	Cole-Parmer (1988)	Douglas (1971) - Eastern Australia Loughran (1976) - Chandler River, New South Wales, Australia (Whatman 41 first, then 542) Mykura (1989) - Kuala Lumpur, Malaysia Lam (1978) - Northern Tai Lam Chung New Territories, Hong Kong Lai (1992) - Selangor, Peninsular Malaysia
Whatman (GFC) glass microfiber filter paper	1.2	4.25	cell harvesting; liquid scintillating techniques	Cole-Parmer (1988)	Drainage and Irrigation Department Malaysia. Chemistry Department, Malaysia Lootens and Lumbu (1986) - Lubumbashi Zaire, Africa.
			made from borosilicate glass	Cole-Parmer (1988)	Lootens and Kishimbi (1986) - Kafubu River, Zaire, Africa. Bilby(1985) - JohnsonCreek, Washington, USA. Ogunkoya and Jeje (1987) - Basement Complex, Nigeria, Africa. Belperio (1979) - Burdekin River, Australia Biksham and Subramaniam(1988) - Godavari River, India
Millipore Type HA	0.45	4.7	for microbiological analyses - designed for complete retention and maximum recovery of total coliform and fecal coliform bacteria.	Millipore Corporation (1990)	Peh (1978) - Pasoh, Bukit Lagong, Bukit Mersawa, Malaysia. Peh (1981) - Sg. Tekam Experimental Basins, Pahang Malaysia. McCave (1979) - Recommended for estuarine studies. Eaton *et al.* 1969 - Hubbard Brook, USA
Whatman cellulose nitrate membranes	0.45	4.7			Greer *et al.* (1989) - Ulu Segama, Sabah, Malaysia. Finlayson and Wong (1982) - Victoria, Australia. Forest Research Institute Malaysia (previously GFC)

papers should not be used in the sediment separation process.

Several workers have examined in detail a common problem of the variability in weight of filter papers used. Eaton *et al.* (1969) for example, compared several membrane filters while working on the Hubbard Brook Experimental Forest in New Hampshire, United States of America. The Millipore type HA was tested against other makes, such as the Gelman type AN (an acrylic polymer membrane reinforced with nylon), Carl Schleicher and Schuell

Company type B-9 nitro-cellulose filters, Carl Schleicher and Schuell type C-5 nitrocellulose filters, Selas Flotronics silver filter and Millipore Pure Teflon filters. Unfortunately, they did not say which membrane filter is best suited for gravimetric analysis, but suggested several ways of handling the filters, because the variability in their weights was attributed to the environmental conditions, such as humidity, which can influence the uptake of moisture. Variable weights caused by static charge of the filter and leachable materials within the filter were the other reasons given.

The Whatman 542 filter paper was tested for its reliability by Loughran (1971) and Douglas (1971) in their work on Australian rivers. Loughran used the Whatman No. 41 to separate the coarser suspended sediment and subsequently the Whatman 542; he also recommended double filtering of the 542 when they were blocked. Against the Millipore GSWP, Douglas (1971) reported a smaller mean weight loss for ten Whatman 542 filters when compared to that of the former, although the variability in weight was higher when all were compared. Both recommended careful use of the filters with controls if necessary to achieve the desired accuracy and consistency.

To obtain a good record of sediment data usually requires adequate stream water sampling at various stages of flow. The frequency of sampling is sometimes constrained by the time taken for the water samples to be analysed in the laboratory. In studies where many streams are closely monitored, time spent in laboratory analysis becomes critical, especially during the rainy season when a large number of water samples collected from automatic samplers, for example, becomes more frequent. To handle the large number of samples during such times, the 12.5 cm diameter Whatman 542 may be used in gravimetric analysis because of its bigger effective filtering (Mykura 1989 and Lai 1992)

Fine organic materials in suspended form are expected to be present in streams, paricularly those draining forested watersheds. It is unlikely that organic rocks are present in significant proportions in study areas where igneous rocks predominate; however, the determination of sediment yield should denote mineral sediment discharge. Hence, the procedure of determining mineral suspended sediment usually entails ashing the samples in a muffle furnace at 550°C for 2h (Brown *et al.* 1970 - cited in Peh 1978).

Aims of the Study

In view of the various filters used by previous workers in the country and abroad, and the removal of organics in the suspended sample by ignition, it was necessary to determine the reliability of the filters used. This simple test aimed at:

i) assessing the reliability of the 12.5 cm diameter Whatman 542 through the entire laboratory procedure of sediment separation process as outlined in Table 2.
ii) comparing the reliability of the Whatman 542 with the Whatman GFC, Whatman cellulose nitrate and Millipore Type HA filters.

TABLE 2
Summary of treatments to determine filter weight variability

Treatment A	Filters were left in original box in laboratory environment for 24 h before weighing.
Treatment B	Filters were oven-dried for 2 h,then transferred to desiccator for 20-30 min before weighing.
Treatment C	Filters were oven-dried for 24 h, then transferred to desiccator for 20-30 min before weighing.
Treatment D	Filters were "ashed" in muffle furnace for 2 h at a temperature of 550°C, then transferred to desiccator for 30 min before weighing.

Note: Five empty porcelain dishes were also subject to the same test following treatments A, B, C and D to detect changes (see Table 3).

MATERIALS AND METHODS

A stainless steel forcep was used to handle the filters. The analytical balance, Sartorious Type 2842, which measures to the nearest 0.0001 g was calibrated before the experiment . A porcelain dish was used in each case for holding individual filters for drying the filter in the oven at 105°C, desiccating, weighing, ashing at 550°C, desiccating and final weighing.

Table 2 summarises the four stages of treatments designed to test the extent of variabilities in filter weights. Five porcelain dishes were also subject to the same treatments to evaluate any weight change, because weighing was to be done with the individual filters placed in them after removal from the oven and muffle furnace, as described above. More importantly, this method was carried out in the actual suspended sediment separation procedure.

RESULTS AND DISCUSSION

In the assessment of weight change with time, readings were obtained for each minute and subsequently until the ninth minute for individual cases. It was found that most readings taken from the third minute onwards showed a change probably because of uptake of moisture. The general ruling adopted, therefore, was that sample readings were taken by the first minute the analytical balance was activated.

The weight differences of individual filters resulting from successive weighings are shown in Table 3. Interestingly, the mean weight of all filters, except for the Whatman GFC, suggests some degree of loss after oven drying and subsequent weighing for periods of 2 h and 24 h (Table 2). This loss in weight of individual filters, however, was more inconsistent when treated to oven drying of over 24 h for the Millipore. The Whatman 542, on the other hand, was more consistent - all ten individual filters weighing less than their original air-dried weight. The ten Whatman GFC filters on the other hand, weighed more, indicating perhaps the uptake of moisture was more significant compared to the other three, despite similar laboratory conditions. The mean weight gain of the five porcelain dishes was small at 0.0008 g with a standard deviation of 0.0006 g. The percentage weight gained compared to the first weighing was 0.0012%.

Following the treatment in the muffle furnace, the Whatman GFC registered the highest mean weight loss of 0.0027 g which is 3.63% of

TABLE 3
Summary of weight gain and loss from treatments at first minute weighing

(1) Description	(2) No.	(3) Absolute wt. before treatment (g) A	Weight change following treatment (4) B-A (g)	(5) C-A (g)	(6) D-A (g)*	(7) Wt. gain/loss of filter Col.(6)-(3)	(8) Percentage of wt. change Col.(6)/(3)x100	Comments
Porcelain	1	54.3110	0.0000	0.0003	0.0000		0.0013	relative humidity:
evaporating	2	58.8075	0.0011	0.0016	0.0013		0.0022	50.6% ± 2.2%
dish	3	57.8000	0.0013	0.0000	0.0018		0.0025	
	4	55.0391	-0.0009	0.0017	0.0004		0.0007	temperature:
	5	57.7999	-0.0009	-0.0011	-0.0003		-0.0005	25.4°C ± 1.4°C
Mean		56.7515	0.0001	0.0005	0.0006		0.0012	
S.d.		1.7501	0.0009	0.0010	0.0008		0.0011	
Whatman 542	1	1.1820	-0.0145	-0.0181	-1.1833	-0.0013	-0.1100	relative humidity:
	2	1.1888	-0.0085	-0.0265	-1.1889	-0.0001	-0.0084	51.8% ± 1.8%
	3	1.1885	-0.0155	-0.0171	-1.1907	-0.0022	-0.1851	
	4	1.1884	-0.0154	-0.0226	-1.1907	-0.0023	-0.1935	temperature:
	5	1.1641	-0.0135	-0.0316	-1.1653	-0.0012	-0.1030	25.5°C ± 1.3°C
	6	1.1827	-0.0236	-0.0117	-1.1837	-0.0010	-0.0840	
	7	1.1788	-0.0220	-0.0145	-1.1798	-0.0010	-0.0848	
	8	1.1777	-0.0191	-0.0168	-1.1801	-0.0032	-0.2038	
	9	1.2007	-0.0145	-0.0139	-1.2006	0.0001	0.0083	
	10	1.2056	-0.0072	-0.0266	-1.2069	-0.0013	-0.1078	
Mean		1.1857	-0.0154	-0.0199	-1.1870	-0.0014	-0.1072	
S.d.		0.0112	0.0049	0.0062	0.0110	0.0009	0.0686	

Table 3 (Continued)

Whatman GFC	1	0.0720	0.0010	0.0010	-0.0010	0.0710	-1.3889	relative humidity:
2		0.0735	0.0018	0.0013	-0.0036	0.0699	-4.8980	51.0% ± 0.9%
3		0.0756	0.0000	0.0010	-0.0032	0.0724	-4.2328	
4		0.0740	0.0008	0.0026	-0.0025	0.0715	-3.3784	temperature:
5		0.0737	0.0000	0.0002	-0.0021	0.0716	-2.8494	25.8°C ± 1.4°C
6		0.0733	0.0013	0.0007	-0.0027	0.0706	-3.6835	
7		0.0722	0.0012	0.0009	-0.0034	0.0688	-4.7091	
8		0.0760	0.0006	0.0023	-0.0032	0.0728	-4.2105	
9		0.0736	0.0000	-0.0003	-0.0019	0.0717	-2.5815	
10		0.0732	0.0003	-0.0002	-0.0032	0.0700	-4.3716	
mean		0.0737	0.0007	0.0010	-0.0027	0.0710	-3.6304	
s.d.	0.0012	0.0006	0.0009	0.0008	0.0012	1.0402		
Whatman	1	0.0867	-0.0006	-0.0002	-0.0881	-0.0014	-1.6148	relative humidity:
cellulose	2	0.0878	-0.0026	-0.0003	-0.0884	0.0006	0.9112	49.5% ± 0.8%
nitrate	3	0.0878	-0.0021	0.0004	-0.0891	-0.0013	-1.4806	
4		0.0877	-0.0034	-0.0014	-0.0901	-0.0024	-2.7366	temperature:
5		0.0862	-0.0027	-0.0020	-0.0889	-0.0027	-3.1323	25.8°C ± 0.2°C
mean		0.0872	-0.0023	-0.0007	-0.0889	-0.0014	-1.6106	
s.d.		0.0007	0.0009	0.0009	0.0007	0.0012	1.4114	
Millipore	1	0.0935	-0.0018	-0.0015	-0.0932	0.0003	0.3209	Relative humidity:
Type HA	2	0.0942	-0.0003	0.0006	-0.0939	0.0003	0.3185	49.3% + 1.0%
3		0.0930	0.0000	-0.0003	-0.0928	0.0002	0.2151	
4		0.0927	-0.0001	0.0002	-0.0938	-0.0011	-1.1866	Temperature:
5		0.0930	-0.0011	0.0015	-0.0946	-0.0016	-1.7204	25.9°C + 0.2°C
Mean		0.0933	-0.0007	0.0001	-0.0937	-0.0004	-0.4105	
S.d.		0.0005	0.0007	0.0010	0.0006	0.0008	0.8690	

* Values in this column indicate total loss on ignition at 550°C

the original weight. The main reason is that this filter loses its form and does not burn at 550°C. The temperature was further raised to 600°C, 650°C and 700°C, but analyses showed no marked changes. Because the filter is made from borosilicate glass, it does not burn, unlike the Whatman 542 and the two membrane filters which are combustible at 500°C.

Of the remaining three filters, the Whatman cellulose nitrate membrane appears to have a higher mean weight loss of 0.0014 g or -1.61% of its original weight after the final treatment. Although the Whatman 542 lost 0.0014 g, the proportion of loss to the original weight was only 0.11%. The Millipore Type HA, on the other hand, lost only 0.0004 g, but this constitutes 0.41% of the original weight. The standard deviation ranged from 0.0009 g for Whatman 542 and 0.0008 g for Millipore Type HA while that for the GFC and Whatman cellulose nitrate was slightly higher at 0.0012 g.

It is not certain why the average successive and final weighing showed negative values from the original weight as no washing of the filters was carried out despite careful handling during each treatment. Washing of filters has been attributed to loss of glycerol in solution in the Millipore membranes (e.g. Douglas 1971). The relative humidity and temperature of the laboratory were kept relatively stable during the experiment to keep variability in moisture uptake by the filtering media to a minimum (Table 3). The results obtained may, however, be attributed to the influence of static charge of leachable materials from individual filters which were not determined. An alpha emitting source to remove the effects of static charge as advocated by Eaton *et al.* (1969) was not used in this case.

CONCLUSION

The glass fibre filters may not be suitable to determine the inorganic sediment concentrations. The filter is not combustible at 550°C but loses its form even at higher temperatures. The Whatman 542, Whatman cellulose nitrate and Millipore type HA are combustible at 550°C, the temperature at which organic matter was removed from suspended sediment samples in this study.

In this experiment, the performance of the 12.5 cm diameter Whatman 542 filter, which has a greater filtering area compared to the other three appeared satisfactory after treatment in the muffle furnace for 2 h at 550°C for removal of organic matter and the filtering medium by ignition. The weight loss, if measured in proportion to its weight loss from its original, is the lowest among those tested. In any case when high suspended sediment concentrations are expected in the water samples, especially those from stormflow samples of disturbed catchments, the difference in filter weight becomes relatively insignificant. This variability can therefore be regarded as the minimal acceptable difference when the sediment data are subsequently further analysed. Closer attention, however, must be paid to the consistency of method and frequency of sampling to obtain a good range of sediment record.

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REFERENCES

BELPERIO, A.P. 1979. The combined use of wash load and bed material load rating curves for the calculation of total load: an example from the Burdekin River, Australia. *Catena* **6**: 317-329.

BIKSHAM, G. and V. SUBRAMANIAM. 1988. Sediment transport of the Godavari River Basin and its controlling factors. *J. Hydrol.* **101**: 275-290.

BILBY, R,E. 1985. Contributions of road surface sediment to a Western Washington stream. *For. Sci.* **31**: 827-838.

BROWN, E., M.W. SKOUGSTAD and M.J. FISHMAN. 1970. Methods for collection and analysis of water samples for dissolved minerals and gases. *Techniques of Water Resources Investigation of the United State Geological Survey* **Book 5** Washington: Government Printing Office Chapter A1.

COLE-PARMER Instrument Company. 1988. Cole-Parmer 1989-1990: *Instruments for Research, Industry and Education.* Chicago. U.S.A: 1072p.

DOUGLAS, I. 1971. Comments on the determination of fluvial sediment discharge. *Aust. Geogr. Studies* **9**: 172-176.

EATON, J.S., G.E. LIKENS, and F.H. BORMANN. 1969. Use of membrane filters in gravimetric analyses of particulate matter in natural waters. *Water Resour. Res.* **5**: 1151-1156.

FINLAYSON, B.L. and N.R. WONG. 1982. Storm runoff and water quality in an undisturbed forested catchment in Victoria. *Austr. For. Res.* **12**: 303-315.

GREER, T., W. SINUN, I. DOUGLAS, T. SPENCER and W.M. WONG. 1989. Impact of rainforest logging on storm period runoff, suspended sediment concentration and conductivity of small streams in Ulu Segama, Sabah, Malaysia. Paper presented at *The Regional Seminar on Tropical Forest Hydrology* held in Kuala Lumpur, Malaysia, Sept. 4-9, 1989.

LAI, F.S. 1992. Sediment and solute yields from logged, steep upland catchments in Peninsular Malaysia. Ph.D Thesis. University of Manchester. 590p.

LAM, K.C. 1978. Soil erosion, suspended sediment and solute production in three Hong Kong catchments. *J. Trop. Geog.* **47**: 51-62.

LOOTENS, M. and Y. KISHIMBI. 1986. Some aspects of water and sediment discharge in the upstream section of the Kafubu River (Shaba-Zaire). *Geogr. Ann.* **68A**: 383-392.

LOOTENS, M. and S. LUMBU. 1986. Suspended sediment production in a suburban tropical basin (Lubumbashi, Zaire). *Hydrol. Sci J.* **31**: 39-49.

LOUGHRAN, R.J. 1971. Some observations on the determination of fluvial sediment discharge. *Aust. Geogr. Studies* **9**: 54-60.

LOUGHRAN, R.J. 1976. The calculation of suspended sediment transport from concentration v. discharge curves: Chandler River, N.S.W. *Catena* **3**: 45-61.

MILLIPORE CORPORATION. 1990. *Millipore The Guide.* Bedford. U.S.A: 392p.

MYKURA, H. 1989. Erosion of humid tropical construction sites, Kuala Lumpur, Malaysia. Ph.D Thesis. University of Manchester. 355p.

McCave, I.N. 1979. Suspended Sediment. In *Estuarine hydrography and sedimentation*. ed. K. R. Dyer, p 13-185. London: Cambridge University Press.

Ogunkoya, O.O. and L.K. Jeje. 1987. Sediment yield from third order basins in the Basement Complex rocks in southwestern Nigeria. *Catena* **14**: 383-397.

Peh, C.H. 1978. Rates of sediment transport by surface wash in three forested areas in Peninsular Malaysia. *Occasional Paper* **No. 3**. University of Malaya. Kuala Lumpur. 130p.

Peh, C.H. 1981. The suspended and dissolved sediment load of three small forested drainage basins in Peninsular Malaysia. *Malay. For.* **44**: 438-452.

Task Committee. 1970. Sediment sources and sediment yields. *J. Hydraul. Div. ASCE.* **96**: 1283-1329.

World Meteorological Organisation. 1974. *Guide to Hydrological Practices.* WMO No. 168. Geneva, Switzerland. 3rd Edition: Sections 2 and 3.

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