DISCUSSION

It is not suggested that the new technique is better than the more standard methods. However, it is a technique which can serve quite well in an "emergency" or when the conventional materials are not available. The "Photo-mount" adhesive is readily available and is also relatively inexpensive. Additional advantages are ease of use and transport. There is no mixing of components, no messy containers, no difficult pouring or brushing, no critical setting period or exothermic reaction. Peels can also be produced in a relatively short period of time. The manufacturer's can does, however, carry hazard warnings, and their recommendations should be scrupulously observed. For instance, the technique would not be suitable for use on cave sediments because of the lack of ventilation.

Penetration of the adhesive into the sediment and particularly differential penetration into sedimentary features was not exceptional but appeared to be adequate on damp sediment (approximately 17% water content) under quite humid conditions. In addition to penetrating beyond smearing effects of the trowel, the adhesive successfully held shells within the sediment (Fig. 1). Other proprietary mounting media (e.g., Scotch "Spraymount" or Scotch "Displaymount") would perhaps give different degrees of adhesion and sediment penetration and could be experimented with. Pretreatment of the surface by spraying with a suitable solvent might also give improved penetration and adhesion. Similarly, other backing materials (e.g., composition board) of varying rigidity could be used, and the size and shape of the areas sampled could easily be varied. By combining peels with subsequent solvent dissolution of the adhesive and release of the sediment, the technique could be used for strip sampling for heavy mineral, micro-palaeontological or SEM studies. Coincidentally it was found that, by removing a thin layer of sediment, the technique produced fresh faces which facilitated field observation of the beds.

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A FIELD SAMPLING METHOD FOR OBTAINING REPRESENTATIVE SAMPLES OF COMPOSITE FLUVIAL SUSPENDED SEDIMENT PARTICLES FOR SEM ANALYSIS[1]

J.C. WOODWARD AND D.E. WALLING
Department of Geography
University of Exeter
Exeter, Devon EX4 4RJ, U.K.

THE CONTEXT

A significant proportion of the suspended load in many river systems is transported not as discrete particles but in the form of aggregates or flocs (composite particles) which may be an order of magnitude or more larger than their constituent particles (Walling and Moorehead 1989; Droppo and Ongley 1989). There is, therefore, a need to measure and compare the *in situ* or *effective* size distribution, which includes composite particles, with the *absolute* or *ultimate* distribution of the individual discrete particles obtained after disaggregation and chemical dispersion (cf. Ongley et al. 1981). While such measurements have highlighted the importance of aggregates and flocs in the transport of fine sediment, little is currently known about the genesis and structure of composite suspended particles, especially in the freshwater fluvial environment. Research in this area is hampered by the difficulties involved in isolating and recovering representative composite particles for further analysis. This paper describes a new sampling technique for collecting representative samples of composite suspended sediment particles for subsequent SEM investigation. This technique has been developed as part of a wider investigation of fluvial suspended sediment properties currently being undertaken in the Exe Basin in Devon, U.K.

SAMPLING CONSIDERATIONS

For effective SEM studies, the methods employed during sample collection and preparation must efficiently separate the particles of interest from the water sample without modifying their structure and deposit them upon a suitable substrate (cf. Trewin 1988). There is also a need to ensure that the number of particles deposited upon the chosen substrate is small enough to avoid particle overlap—thus allowing unequivocal identification of discrete composite particles.

Most published SEM studies of composite suspended sediment particles have focused on estuarine and continental shelf environments and have used filter membranes with pore sizes of 0.45 μm or smaller during sample preparation (Bigham 1974; Zabawa 1978; Pierce and Siegel 1979). Many workers have also used such small pore membranes in association with low vacuum filtration techniques to separate the sediment from the water sample. This procedure is not, however, to be recommended for the examination of flocculated/aggregated sediment, because, as Whalley (1979) has pointed out, even when using only a minimum of vacuum, the artificial compaction caused by filtering is likely to make individual grains adhere by Van der Waal's forces. Without the use of vacuum filtration, however, sample collection with conventional 0.45 μm filters is an extremely slow process and is impractical for environmental monitoring programs where rapid and repeated sampling is desirable (i.e., during storm discharge events). In addition, if conventional filtration methods are used as the sole means of separation, the high clay and fine silt content of many suspended sediment samples collected from rivers will produce artificial composite particles as the finer-grained particles in the sample settle onto and between larger grains during the filtration process. A further problem frequently encountered during the preparation of such samples for SEM work is a function of total sediment concentration. Even when filtering only relatively small volumes of turbid suspension (ca. 50 ml), a veneer of fine material is deposited across the membrane surface. This membrane "overcoating" creates an undesirable low-relief surface, analytically-produced compound structures, and a "dirty" background for SEM work on larger particles that preclude the unambiguous identification and observation of discrete composite particles.

Ideally, the sample processing method should efficiently separate the larger composite particles from the remainder of the turbid suspension without either damaging the particles of interest or creating analytically-produced structures during the collection process. Preliminary measurements of the absolute and effective particle size distributions for fluvial suspended sediments in the Exe Basin in Devon indicate that most of the composite particles in the suspended load are typically greater than 20 μm in size. Thus, to isolate these larger composite particles from a suspended sediment sample, the use of filter membranes

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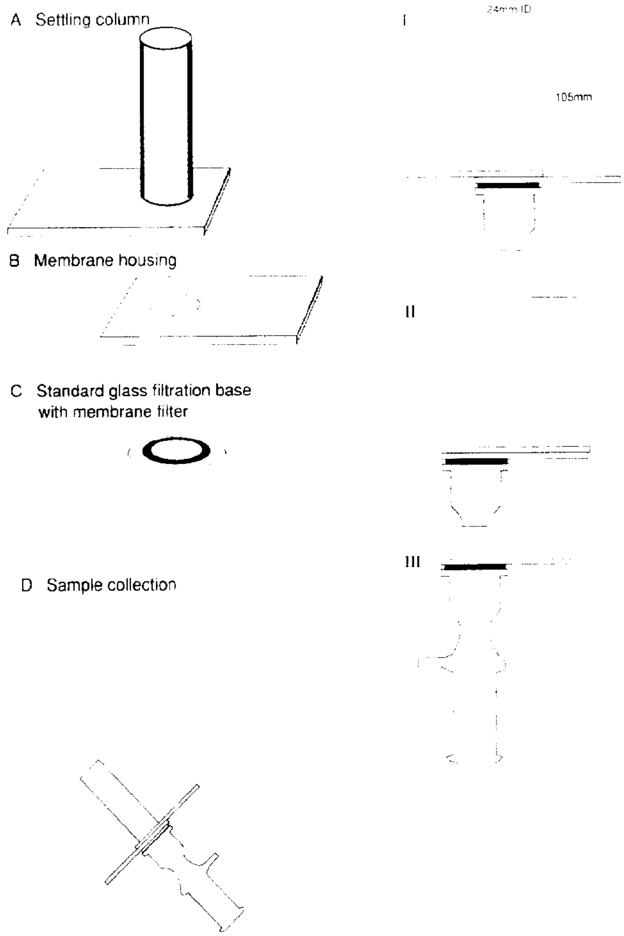

FIG. 1.—The sampling apparatus used in this study. Stages I to III illustrate the use of the settling tube and membrane housing as a selective separation device.

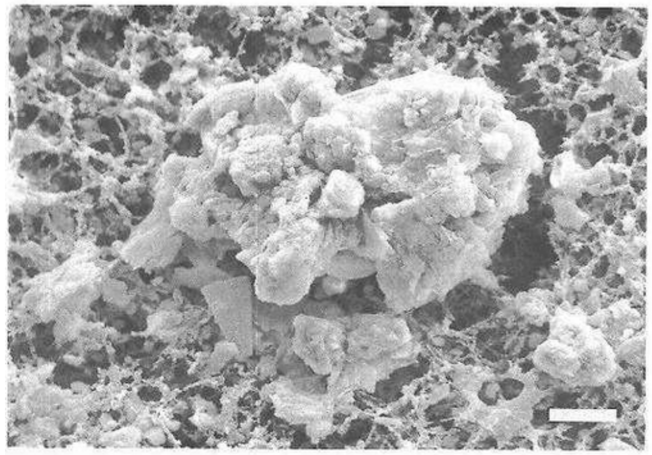

FIG. 2.—SEM photomicrograph showing a composite suspended sediment particle collected from the River Exe at Thorverton gauging station on 15 February 1991. Note the largely sediment-free membrane filter surface which allows identification and observation of individual composite particles (scale bar = 40 μm). This particle is a comparatively large example (ca. 200 × 100 μm) exhibiting a range of constituent particle grain sizes and shapes. A smaller (ca. 40 μm diameter) composite particle is also readily identifiable at the bottom right hand corner of this photomicrograph. We are grateful to John Merefield (Earth Resources Centre, University of Exeter) for providing access to SEM facilities and technical advice.

with very small pore sizes (i.e., < 1 μm) is both impractical and unnecessary.

With these sampling and preparation considerations in mind, we designed and constructed a sampling apparatus which allows:

1) Efficient separation of the coarse, composite particles from the water sample and from the finer portion of the suspended load, and their collection upon a membrane filter without using vacuum filtration.
2) Fairly rapid sample processing (in minutes rather than hours).

APPARATUS AND FIELD SAMPLING

These objectives are achieved by combining a custom-built perspex settling tube and membrane filter housing with a standard glass filtration assembly (Fig. 1). Small settling tubes of similar design have previously been used in the collection of flocculated suspended sediment onto inverted microscope slides for particle size analysis (cf. Droppo and Ongley 1989). Together, the settling tube (A) and membrane housing (B) produce a total sample chamber volume of 50 ml. This apparatus is held together with two large bulldog clips, with the membrane filter held between the perspex housing (B) and the glass filtration base (C). We have used cellulose nitrate membrane filters (47 mm diameter) with 5 μm pores.

Prior to sample collection, a small volume of pre-filtered water is used to "wet" the membrane filter, under vacuum, using a hand held pump, because dry membrane filters transmit water only extremely slowly under "no-vacuum" conditions. Suspended sediment samples are collected by holding the apparatus at an angle of approximately 45 degrees to the water surface (Fig. 1D). The apparatus is then held in a vertical position as the largest particles in suspension settle to the bottom of the tube onto the membrane filter surface (Fig. 1I). After a few seconds, the bulk (ca. 95%) of the total sample is then separated from the membrane housing and discarded by sliding the settling tube horizontally, leaving the sediment particles with high fall velocities on the membrane surface in approximately 2.5 ml of water (Fig. 1II). The sample is then placed upon a filtration flask until this excess water has filtered away (without using vacuum filtration). The comparatively large (5 μm) pores in the membrane filter and the small volume of water retained (2.5 ml) combine to ensure that this final stage in the separation process takes only about 15–20 minutes. The membrane filter is then transferred to a plastic petri dish for transport to the laboratory. Following air- or freeze-drying, small portions of the membrane (ca. 6 × 6 mm) are mounted on standard aluminium SEM stubs with a carbon conducting cement and gold coated.

RESULTS

Figure 2 shows a composite suspended particle and highlights the effectiveness of the sampling technique in providing a "clean", sediment-free background which allows unambiguous observation of the particles of interest. It may be possible to obtain an approximate estimate of the particle size characteristics of individual composite particles from such micrographs using photo-enlargements and digitizing equipment. Such an approach, combined with Energy Dispersive X-ray analysis (EDS) for rapid checking of grain composition, may offer considerable potential for discriminating between composite particles of different origins, e.g., source-derived aggregates or the products of *in situ* flocculation processes.

CONCLUSIONS

The advantages of this method are as follows:

1) This selective separation apparatus is robust, portable and inexpensive. It allows efficient and rapid isolation of composite particles from a suspended sediment sample for study by SEM.
2) As the bulk (ca. 95%) of the finest particles in the sample are also removed via the settling tube—and are thus prevented from settling in the membrane housing—the dual problem of membrane "overcoating" and the creation of artificial composite structures described above is removed.
3) The use of membrane filters with 5 μm pores (an order of magnitude larger than most previous studies) reduces filtration time dramatically without the use of vacuum filtration. Furthermore, much of the fine material (< 5 μm) in suspension in the 2.5 ml of sample remaining in the membrane housing passes through the filter.

The basic method is equally applicable to other aquatic sedimentary environments (lacustrine, estuarine and marine) and is currently in use on a routine basis to collect samples for SEM analysis as part of a wider investigation of fluvial suspended sediment properties in the Exe Basin.

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