A Vacuum-Operated Pore-Water Extractor for Estuarine and Freshwater Sediments

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Abstract. A vacuum-operated pore-water extractor for estuarine and freshwater sediments was developed and constructed from a fused-glass air stone attached with aquarium airline tubing to a 30 or 60 cc polypropylene syringe. Pore water is extracted by inserting the air stone into the sediment and creating a vacuum by retracting and bracing the syringe plunger. A hand-operated vacuum pump attached to a filtration flask was also evaluated as an alternative vacuum source. The volume and time to extract pore water varies with the number of devices and the sediment particle size. Extraction time is longer for fine sediments than for sandy sediments. Four liters of sediment generally yield between 500 and 1,500 mL of pore water. The sediment that surrounds and accumulates on the air stone acts as a filter, and, except for the first few milliliters, the collected pore water is clear. Because there is no exposure to air or avenue for escape, volatile compounds and *in situ* characteristics are retained in the extracted pore water.

A major thrust in current environmental toxicology is the evaluation of contaminants in sediments, particularly, in pore (interstitial) water. Most contaminants that enter aquatic systems are ultimately deposited in the bottom sediments (Salomons *et al.* 1987). The processes of sediment transport and deposition transfer pollutants from the water column into the bottom sediments, but the sediments also provide a source of contaminants to the surface waters (Davies-Colley *et al.* 1985; Salomons 1985). Consequently, the effects of contaminants in sediments on the environment must be identified for management and protection of aquatic resources.

To have a toxic effect on aquatic organisms, contaminants must be biologically available. In aquatic systems, the major route of exposure is solution. However, to elicit a toxic response, contaminants in sediments have to be sufficiently high for the equilibrium-partitioning concentration in pore water to exceed toxic levels (Adams *et al.* 1985; O'Donnel *et al.* 1985). Assessing the toxicity of pore water directly is a logical step in evaluating sediment and bioavailability of contaminants. In fact, equilibrium-partitioning of contaminants in pore water is a potential means of assessing sediment quality (Shea 1988). Unfortunately, techniques for isolating or collecting pore water for chemical analyses or toxicity testing require elaborate equipment and may alter the chemical characteristics of the sediment and pore water (Bender *et al.* 1987; Knezovich and Harrison 1988). In addition, most techniques do not produce enough pore water for standard toxicity testing.

A variety of methods have been used to extract pore water. Most early investigators were concerned with general chemistry of sandy intertidal and shoreline areas, and pore water was obtained by draining the sediment (Bruce 1928). Other methods of pore-water extraction have been developed and include various filtering devices (Borden 1931; Cole 1932; Pennak 1940; Ishida 1953; Jones 1955; Eriksen 1963; Brafield 1964; Saager *et al.* 1990), mechanical squeezers (Kruikov and Komarova 1954; Lusczynski 1961; Siever 1962; Hartman 1965; Manheim 1966; Reeburgh 1967; Presley *et al.* 1967; Bender *et al.* 1987; Jahnke 1988; Carr *et al.* 1989), and dialysis (Hesslein 1976; Carignan *et al.* 1985). Centrifugation and elutriate preparations are also commonly used for collecting water for the assessment of contaminants and toxicity in the liquid fraction of sediments (Shuba *et al.* 1977; Dutka and Kwon 1988; Geisy *et al.* 1988; Ankley *et al.* 1990; Saager *et al.* 1990). Many described methods for collecting pore water require extensive sample manipulations in addition to filtration or centrifugation prior to testing or analysis. However, vacuum-operated devices (Johnson 1967; Creaser 1971; Makemsom 1972; Sayles *et al.* 1976; Knezovich *et al.* 1987; Whitman 1989) have the potential to alleviate many of the problems associated with sediment sample manipulation and turbidity of extracted pore water. The vacuum-operated pore-water extractor described in this paper is inexpensive, easily constructed, and effective for sediments of all particle sizes. It provides sufficient quantities of pore water for toxicity testing and chemical analyses, and extracts do not require filtration or centrifugation to clear the pore water prior to toxicity testing or analyses.
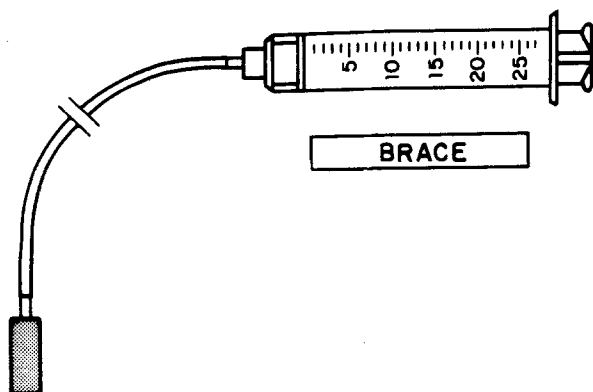
Fig. 1. Sediment pore-water extractor constructed from a syringe, brace, tubing, and an air stone
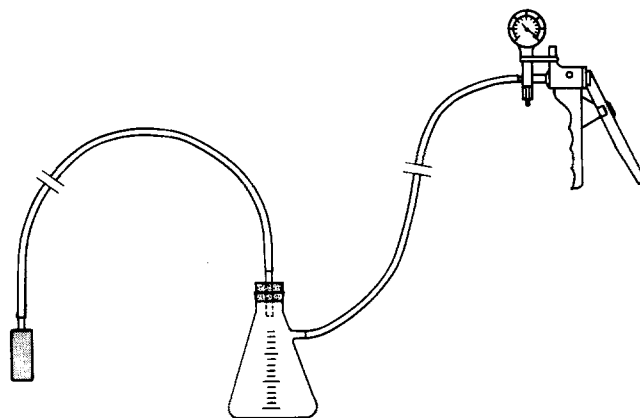

Fig. 2. Sediment pore-water extractor constructed from a hand-operated vacuum pump, tubing, filtration flask, and an air stone

Materials and Methods

The pore-water extractor that was developed consists of a fused-glass air stone attached by aquarium airline tubing to a 30 or 60 cc (other size syringes can be used) polypropylene syringe (Figure 1). The syringe provides the vacuum and also serves as the collecting chamber for the pore water. An alternate vacuum source is a commercial hand-operated vacuum pump. The hand-operated pump is attached with tubing to an Erlenmeyer or filtration flask which serves as the collecting chamber (Figure 2).

To extract pore water, the air stone was inserted 8 to 10 cm into the sediment sample and vacuum created by retracting the plunger of the syringe. The plunger is braced in the retracted position by inserting a piece of wood, cut to the proper length, between the end of the syringe and the plunger lip. When full, the airline tubing is removed from the syringe and the syringe is either capped for storage or emptied and reset for another extraction.

To test the device, pore water was extracted from the sediment samples from various freshwater and estuarine habitats in the Southeast (Table 1). Sediment samples were collected with either an Ekman or Ponar grab.

Sediment particle size was determined by wet sieving with U.S. Standard Sieves (500 μm, 250 μm, 125 μm, and 63 μm) and a 1.2 μm glass-fiber filter. Mean particle size was calculated by Inman's (1952) method; the resulting phi values were converted from $-\log_2$ back to metric units. An estimate of percent organic matter was determined by loss on ignition at 600°C for 4 h. Moisture content was determined as the percent difference between wet and oven-dried (105°C for 24 h) weight.

Results and Discussion

The vacuum-operated extractor collected pore water from bottom sediments at rates from 11 to >100,000 mL/h (Table 1). Extraction rates varied with sediment particle size and were lowest for sediments with the highest percentages of fines and smallest mean particle sizes. The amount of organic matter or percent moisture in the sediment did not influence extraction rates. Extraction of pore water from sandy sediments (mean particle size >140 μm) was very rapid—the syringes filled almost immediately when the vacuum was applied.

The required volume and time to extract pore water varies with the number of devices used and the sediment particle

size. About 150 mL of pore water/h can be extracted from most sediments with six 30-cc syringes or two-hand operated vacuum pumps. Four liters of sediment generally yield between 500 and 1,500 mL of pore water, but sediments with large particle sizes (sand) do not contain as much pore water as sediments with finer particles.

The first few milliliters of extracted pore water may be turbid. This water is discarded by first bending the airline tubing, which retains and maintains the vacuum at the air stone, and then releasing the vacuum at the syringe by pulling the tubing from the end of the syringe. After dispensing the turbid pore water, the airline tubing is re-affixed to the syringe. The bend in the tubing is then released and a fresh vacuum applied by retracting and bracing the syringe plunger.

The initial turbidity in the extracted pore water from some sediment samples apparently occurs before the finer sediment particles have accumulated on the surface of the air stone. The sediment that surrounds and accumulates on the air stone serves as a filter and screens the particulate matter. After discarding the first few milliliters of turbid sample, the rest of the collected pore water is generally clear. Mitchell *et al.* (1989) also found that pore water extracted with a vacuum-operated device became clear after the initial sample. An exception is sandy sediments (larger particle sizes)—larger volumes need to be extracted before the pore water becomes clear.

Pore-water extractors that drastically disturb the sediments (causing aeration and change in redox potential) and also require filtration or centrifugation may not be adequately sampling the dissolved fraction (or the actual constituents) of contaminants in the pore water (Knezovich *et al.* 1987; Puls and Barcelona 1989), although there is no satisfactory method for determining unambiguously the partitioning of contaminants in sediments (Tessier and Campbell 1987). Major problems of most pore-water extractors include contamination of pore water with overlying water and solid-solution reactions. Contaminants in elutriate or pore water rapidly partition back onto the underlying sediments following squeezing or centrifugation (Bender *et al.* 1987). Changing from anoxic to oxic conditions in the sediments could also alter the state (species) of contaminants in the pore

Table 1. Extraction rate (mL/h) for the vacuum-operated pore-water extractor from sediments characterized by percent moisture, percent organic matter, and sediment particle size

Station	Rate (mL/h)	Organic matter (%)	Moisture (%)	Percent composition by particle size (μm)				Mean size (μm)
				250	125	63	1	
Charleston Harbor	18	14.4	72	6.6	16.0	9.9	67.5	21
Savannah Harbor 1	27	15.7	78	1.1	5.3	5.5	88.1	10
Savannah Harbor 2	27	18.2	81	0.4	1.0	13.6	85.0	12
S. Thurmond Lake	25	6.5	41	13.6	38.9	16.4	31.1	5
St. Simons Est. 1	22	16.7	77	10.7	15.3	11.6	62.4	24
St. Simons Est. 2	15	12.8	68	6.1	41.6	9.4	42.9	31
St. Andrews Bay 1	35	15.3	69	7.9	21.3	24.0	46.8	28
St. Andrews Bay 2	49	19.5	75	8.5	27.2	15.2	49.1	28
St. Andrews Bay 3	35	9.2	61	22.5	35.9	13.3	28.3	51
Perdido River	108,000	0.2	24	96.8	2.8	0.1	0.3	380
Galveston Bay 1	11	6.4	53	4.2	30.0	18.4	47.4	27
Galveston Bay 2	12	2.6	34	1.8	44.8	26.0	27.4	48
Galveston Bay 3	6,750	3.0	27	2.5	67.4	20.6	20.6	140

water. Salomons *et al.* (1987) indicated that filtration of anoxic sediments (causing a shift to the oxic condition) could possibly cause iron oxidation and colloidal formation with subsequent sorption of previously dissolved contaminants. Consequently, not only could filtering cause aeration and loss of dissolved species, but the progressive clogging of the filter with the colloids results in a changing of the generally accepted level of separation between dissolved and particulate matter (Salomons *et al.* 1987).

Volatile compounds of sediments are retained in the pore-water extract or in the sampler described, because the system is enclosed and no avenue of escape exists. Knezovich and Harrison (1987, 1988) showed that a vacuum-operated device similar to the one described here was effective in collecting volatiles from pore water. The ability of a sampler to retain these fractions may be particularly important for aquatic sediments contaminated with volatile compounds. Although this was not tried, a carbon trap could be included in the device to collect volatile compounds as they are extracted with the pore water.

The extractor with the hand-operated vacuum pump was just as effective in obtaining pore water as the syringe system. This system was, however, more expensive and more cumbersome to use than the syringe. Attachment of a carbon trap for capture of volatile compounds would probably be easier to install on the hand-operated system than on the syringe extractor—a carbon trap could be inserted into the tubing that connects the filtration flask to the hand-operated vacuum pump.

The vacuum-operated extractor described here can be used to collect pore water *in situ* or from sediment samples in the field, thereby alleviating the need to return large quantities of sediment to the laboratory. We prefer extracting pore water from consolidated sediment samples collected with a Ponar grab. The extracted pore water is essentially the same as it is *in situ*, because exposure to oxidizing conditions is limited to the external surfaces of the sediment sample. Pore water was also extracted from sediment samples while they were still in an Ekman grab—lift the top flaps of the sampler, decant the surface water, and insert an air stone into the essentially undisturbed sediment sample.

Glass syringes and teflon tubing are more expensive options if there is concern about possible adsorption of organic contaminants to the walls of the extractor. However, the small amount of contaminants adsorbed to the extractor will probably not reduce or alter the toxicity of the pore water or be detectable analytically in light of the natural variability inherent in field samples.

The vacuum-operated pore-water extractor described in this paper provides an inexpensive, but highly versatile and easy device for collecting pore water for toxicity testing and chemical analyses. The major advantages of this method are: large volumes of pore water can be collected within a reasonable amount of time; it is effective for all types of aquatic sediment samples; it eliminates the need for extensive sample manipulation and processing; and pore water can be extracted from essentially undisturbed sediments, thereby minimizing activities that could alter the *in-situ* chemical characteristics of the pore water.

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