

COMPARATIVE STUDIES ON EXTRACTION OF SEDIMENT INTERSTITIAL WATERS: DISCUSSION AND COMMENT ON THE CURRENT STATE OF INTERSTITIAL WATER STUDIES*

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Abstract—The implication by Murthy and Ferrell (1972) that interstitial water studies are in a confused state is criticized on the basis that the authors have not drawn on a considerable body of data, especially Soviet studies since the 1950's, and results of the Deep Sea Drilling Project. Pressure filtration systems for extracting interstitial waters are currently the methods of choice for marine studies and have achieved substantial reliability and reproducibility. Although gaps and problems remain, many aspects of interstitial composition of marine sediments have been clarified; these include the substantial constancy of composition of interstitial waters in deep sea pelagic deposits, depletion of interstitial cations owing to authigenic mineral formation in more rapidly accumulated (especially terrigenous) sediments, and special phenomena in sediments overlying salt deposits.

Murthy and Ferrell (1972) obtained differences in the composition of interstitial solutions from Gulf of Mexico sediments by dilution and centrifuging and by pressure filtration techniques. They interpreted their results in terms of failings of the pressure squeezing technique of Reeburgh (1967). I suggest that the problem may be the other way around. I also wish to offer clarification of the impression left by Murthy and Ferrell (1972) that knowledge of interstitial waters of marine sediments is in a rather anarchic state, especially with regard to methodological validity. This appears to be necessary because the above authors, as well as some previous workers who have contributed to the subject, appear to be unacquainted with, or have not availed themselves of a great deal of information that has been accumulated in the field of interstitial water studies. The extensive studies of Russian workers since the 1950's and the interstitial water program of the Deep Sea Drilling Project are particular cases in point. The present considerations also apply to a subsequent paper by the above authors (Murthy and Ferrell, 1973).

LEACHING OR DILUTION METHODS FOR RECOVERING INTERSTITIAL SOLUTES

For most geochemical and hydrological purposes, one wishes to analyze the composition of the mobile, non-adsorbed fluid component of the sediment. In some cases (e.g. brittle, consolidated rocks) grinding and leaching (dilution) may be the only recourse, at least for weakly reactive ions such as chloride. However, it has long been known that the cationic composition of leachates of sediments is influenced, often in a complex manner, by the type of sediment, dilution, temperature, and length of leaching, as well as dissolution (or precipitation) of solid phases. One need not dwell on this area, for Murthy and Ferrell's own leaching results show more than 100 per cent variability in their calculated units, depending on dilution. Moreover, although one can in theory approach a 'true' interstitial composition by extrapolation from successively smaller dilutions, in practice this is a difficult matter because of lack of linearity in the complex changes in sediment-water chemical systems, difficulty in achieving equilibration of leaching solutions (Walters, 1967) and separating leachate at low dilutions, and the problematical influence of adsorbed water on calculations. I confess that I myself earlier abandoned attempts to use leaching techniques because of inability to achieve adequate reproducibility and reliability

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in sea water systems, in addition to the tedium and slowness of the method.

In the case of Murthy and Ferrell's work, there is additional difficulty in interpreting or applying their data, because results are reported only as m-equiv per 100 g sediment, without description of how the numbers were obtained, without information on total of interstitial water content, without data on chloride, desirable as a control element because of its relative freedom from ion exchange reactions with sediment, and without reproducibility data.

PRESSURE FILTRATION TECHNIQUES

Existing experience with pressure squeezers or other comparative extraction studies is dealt with in a grossly inadequate way. For example, Manheim (1966) is cited in support of the contention that pressure squeezing yields dubious results. In fact, this paper showed that chloride concentration in interstitial waters from typical marine sediments varied less than 1 per cent when extracted by hydraulic squeezer under pressures varying from 580 to 12,000 psi (39–820 kg/cm²). The only significant variations cited referred to

literature data (including cations) on sediments under extreme pressures, at which adsorbed water having lower (not higher) electrolyte content began to be expelled from clays, or more dilute solutions, where the pressure threshold to reach adsorbed waters layers is lower. Similar results were obtained by Sayles (1970) in a reevaluation of the hydraulic squeezers that are standard equipment on board *Glomar Challenger* (Deep Sea Drilling Project). Working with pure Na bentonite clays at relatively low pressures, Bolt (1961) demonstrated that significant influences of pressure on filtrates did not occur with solutions at 0.1 N NaCl concentration, whereas some effects were found at or below 0.015 N NaCl concentration.

Extensive studies of pressure filtration systems have been performed by Soviet authors (see discussion and references in Kriukov, 1971). Perhaps the most careful examinations of pressure filtration systems for marine geochemical work are those of Shishkina (1968 and references cited); examples of her data are given in Table 1. It is evident that for the clayey samples, influence of variable pressure in filtration on extracts largely lies below the reproducibility of even highly precise analytical technique*.

Good reproducibility (shown to an unusual degree in Table 1) is an essential requirement for effective work in marine systems. It is also suggestive, though not conclusive, with regard to the validity of fluid obtained by extraction systems. The absolute composition of extracts may be checked by comparative tests on interstitial solutions from sediments approaching the sediment–water interface in sea water. Owing to diffusion such solutions ought to have a major element

* At very high pressures and in carbonate rich sediments Shishkina obtains an increase in Ca and alkalinity, presumably due to the effect of pressure in solubilizing carbonate. This is not a significant effect under ordinary conditions in squeezings of relatively short duration. Precautions also need to be taken in high-alkalinity sediments so that carbonate does not precipitate in the stored interstitial waters, giving rise to spuriously reduced calcium and bicarbonate alkalinity (Bernier *et al.*, 1970).

Table 1. Effect of pressure on concentration of ions in interstitial water squeezed from clayey marine sediments (from Shishkina, 1968). Chlorinity is close to bottom water values for area in question

Pressure kg/cm ²	g/kg			m-equiv/kg Alk.*	g/kg				Sum
	Cl	Br	SO ₄		Na	Ca	Mg	K	
Bering Sea clayey mud									
0–270	18.38	—	0.86	1.648	—	0.349	1.18	—	32.9†
270–405	18.39	—	—	1.650	10.47	0.351	1.17	—	32.7†
405–675	18.39	—	0.84	1.651	10.45	0.350	1.16	—	32.9†
675–1080	18.39	—	0.84	1.682	10.45	0.361	1.18	—	32.8†
1080–1630	18.39	—	0.88	—	10.33	0.346	1.15	—	—
1630–2150	18.38	—	—	—	—	0.348	1.18	—	—
2150–3100	18.35	—	0.88	1.625	10.43	—	—	—	32.8†
Okhotsk Sea diatomaceous clay									
0–270	19.15	0.063	2.71	0.295	10.72	0.421	1.27	0.556	35.18
270–405	—	—	2.70	0.301	10.74	0.425	1.26	0.561	—
405–675	19.15	0.064	2.70	0.297	10.76	0.427	1.26	0.563	35.22
675–1080	19.15	0.063	2.68	0.294	10.74	0.423	1.27	0.570	35.19

* Alkalinity calculated as bicarbonate ion, in m-equiv/kg.

† Dried residue.

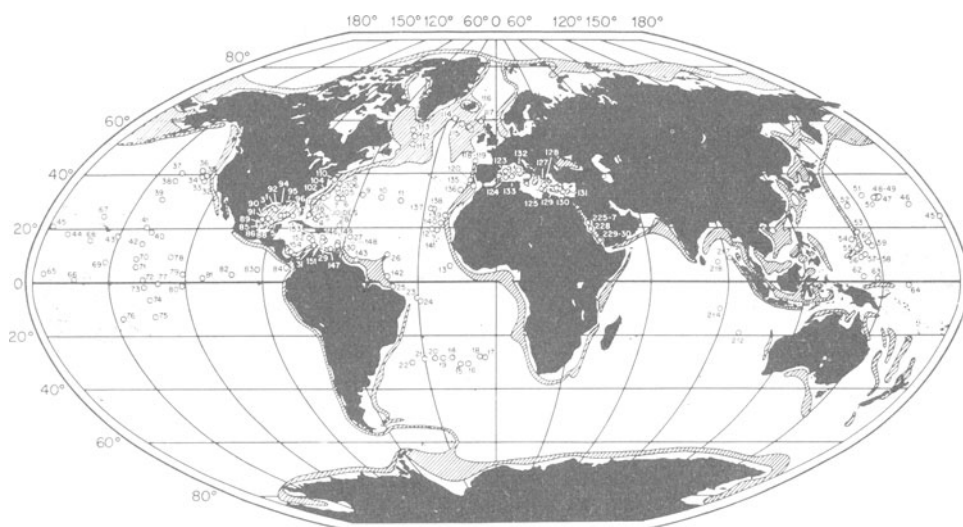


Fig. 1. Distribution of deep sea drilling project sites from which interstitial water data are available (see *Initial Reports*, volumes of the Deep Sea Drilling Project, U.S. Government Printing Office).

composition close to that of oceanic or local bottom water, where reasonable constancy in bottom water has prevailed.

Interstitial waters from all manner of sediments in over 150 drill sites in the world oceans have been studied in the course of the Deep Sea Drilling Project (DSDP) (Fig. 1). Interstitial chloride ion in samples approaching the sea floor generally approaches agreement with concentration in bottom waters within 1 per cent. Major cations in surficial sediments from both DSDP and piston core investigations approach agreement to within the reproducibility of the analytical data, if allowance for difference between *in situ* temperature and temperature of extraction on board ship is made (Mangelsdorf *et al.*, 1969, and subsequent investigators). Indeed, it was the accuracy in establishing interstitial water composition, made possible by pressure filtration methods, that partly led to discovery of the temperature effects (Mangelsdorf, 1969). Interstitial water data on Pacific Ocean sediment, reported by Shishkina (1966 and reference cited) revealed consistent but inexplicable enrichments in K (*ca.* 10 per cent), occasional enrichments in Na (<1 per cent) but depletion in Mg (*ca.* 3 per cent) with respect to standard ocean water, even in near-surface sediments. Diffusive fluxes calculated for K on a whole-ocean basis, using the observed concentration differences, yielded an input to the oceans as much as 10 times as great as the total input of all rivers and streams. Laboratory experiments showed, however, that the above differences were attributable to the rise in temperature as cores

were raised from the ocean floor (1–2°C) to shipboard and shipboard laboratory temperatures, where extraction took place. Reversible changes in strength of exchange binding evidently act to release monovalent ions from sediments (clays) and absorb divalent ions with increasing temperature. Subsequent studies showed that similar (and more pronounced) enrichment effects occurred for interstitial Si (Fanning and Pilson, 1971), Sr and B, although changes for interstitial Cl and SO₄ were below 0.5 per cent of total interstitial concentrations (Sayles *et al.*, 1973). The DSDP data are available in the *Initial Reports* volumes of the Deep Sea Drilling Project Legs 1–14, 16–19 published at this writing, and 22–23 (in press) (Government Printing Office, Washington, D.C.), and summarized in Manheim and Sayles (1974). Data from piston cores are available in dozens of recent, as well as older investigations, e.g. Berner *et al.* (1970); Bischoff and Sayles (1972), Nissenbaum *et al.* (1972), Shishkina (1972) and references cited in these publications.

In short, Donnan equilibria are not necessarily 'the major controls on the distribution of ions in clayey sediment–water systems'. In salty solutions (e.g. sea water) the Donnan forces are repressed and operate only close to the exchange sites, within the double layer-adsorbed water zone. This zone encompasses only a small proportion of the associated fluid in unconsolidated–semi consolidated sediment and great pressures are required to remove solutions from it. Virtually all of the pressure filtration devices now used

Table 2. Representative pressure filtration devices. Asterisk indicates those models capable of extracting fluid from relatively consolidated sediments, as well as unconsolidated materials

Type	Description	Reference
1 Hand (screw) squeezer	Large volume sediment, low pressure (few tens of atmospheres max.), steel.	Siever (1962)
2 Disposable syringe squeezer	Small volume, low pressure for few ml fluid.	Manheim (1970)
3 Hydraulic squeezers* (used with hydr. press)	Various steel squeezers with from 1000 to 20,000 kg/cm ² capacity, first publ. 1947, now used by most Soviet workers. Employs ingenious self-adjusting pressure gasket system. Kriukov also describes special centrifuge systems.	Kriukov (1971)
4 Hydraulic squeezer* (used with hydr. press or large C clamp)	Stainless steel squeezer, modified from Kriukov type, employing disposable syringes as gas-tight fluid receivers. Pressure to 2,500 kg/cm ² .	Manheim (1966)
5 Hydraulic squeezers* (or used with C clamp)	Modified from (3) to simplify filter support and extend size range: standard models employed for shipboard use on Deep Sea Drilling Project. Depicted in and operation described in noted references.	Manheim and Sayles (1974) and Waterman (1970)
6 Hydraulic squeezer	All-plastic squeezer designed to be used on whole core-liner sections to avoid air contact. Note, however, that external (contaminated?) portions of cores are thereby included. Pressure up to 76 kg/cm ² .	Kalil and Goldhaber (1973)
7 Gas-mechanical squeezer	Pressure applied by gas (10 kg/cm ²) to rubber membrane in commercial mud filter press (see 6). Aluminum, rubber and steel construction.	Hartmann (1965)
8 Gas-mechanical pressure	All-plastic device similar in principle to (7).	Reeburgh (1967)
9 Gas-pressure (displacement)	Commercial press employing CO ₂ cartridge or tank gas to expel fluid from drilling mud; used for unconsolidated natural sediments. (Baroid Co., Houston, Texas)	Luczynski (1961)
10 Gas-pressure (displacement)	Teflon-lined squeezer employing nitrogen gas, up to 70 kg/cm ² to obtain fluids for trace metal determination.	Presley <i>et al.</i> (1967)
11 Fluid displacement	Used silicone fluids to displace pore water from sands and permeable sediments.	Scholl (1963)

Note that some of the earliest investigators of soil solutions used liquid displacement (e.g. Schloesing, 1866) techniques.

(Table 2) operate within 'safe' pressure limits that extend as much as two orders of magnitude or more beyond the 'suspect' pressure of 100 psi (7 kg/cm²) used for extraction by Murthy and Ferrell (1972). Although pressure filtration methods may be simple and effective, they, of course, do not exclude errors due to improper sampling, storage, handling of sediment or fluid, or analytical problems. Most inaccuracies or discrepancies noted in interlaboratory or methods checks (e.g. Manheim *et al.*, 1971; Hammond, 1973) can be traced to such sources. Owing to diffusion, interstitial concentrational changes are usually gradual and smooth with depth. Erratic fluctuations may be a tipoff that extraneous errors are affecting the system, at least for major constituents. Finally, in cases where reduced

ionic strength solutions are to be extracted, one should establish the threshold of pressure influence by successive pressure increments.

CURRENT STATE OF INTERSTITIAL WATER STUDIES

I wish to mention here only a few of the basic points that have been established: brief reviews of the subject are available in Manheim (1972) and Glasby (1973); more extensive reviews, not including most DSDP data, are provided in Bruevich (1966), Kriukov (1971), and Shishkina (1972); English language reviews are in press (Manheim and Sayles, 1974; Manheim, 1974).

Table 3. Interstitial water composition from piston core, Sta. 3152, Pacific Ocean, water depth 5660 m (from Shishkina, 1958, cited in Shishkina, 1972). Core is chiefly red clay with occasional manganese-iron concretions and microconcretions

Depth below sed. surf. (cm)	m-equiv/kg										
	Na	K	NH ₄	Ca	Mg	Cations	Cl	SO ₄	Br	Alk.	Anions
16-30	479	10.9	<0.10	20.3	105	615	552	60.5	0.83	1.9	615
130-150	475	11.0	<0.10	19.3	102	607	545	60.8	0.82	2.0	609
260-281	474	11.2	<0.10	19.8	100	605	544	58.6	0.81	2.0	605
350-370	474	11.2	<0.10	19.1	101	604	545	56.5	0.83	2.0	604
415-435	474	10.6	<0.10	20.0	104	609	548	59.5	0.83	1.8	609
Standard sea water	468	9.9	<0.10	20.6	106	604	543	56.5	0.83	2.0	602

Pelagic sediments

For large areas of the world oceans the interstitial water composition of hemipelagic clays and slowly deposited biogenic oozes remains relatively constant with depth, and similar to standard sea water composition, at least with respect to major ionic constituents. Table 3 shows relationships in a typical piston core of Pacific red clay, but similar conditions have been shown in the DSDP program to extend throughout the sediment column to basaltic basement. Most of the significant deviations from standard sea water ion/Cl ratios are attributable to the temperature-of-squeezing effects, discussed in the text. Trace constituents may show larger variations (Brooks *et al.*, 1968; Presley and Kaplan, 1971 and other DSDP reports).

Terrigenous-biogenic sediments

In terrigenous or rapidly deposited biogenic sediments, weakly reactive interstitial species such as chloride vary only to a minor degree (except in special cases, below) but diagenetic reactions typically alter ionic ratios far beyond levels affected by temperature or other analytical artifacts. Sulfate is often depleted, especially in organic-rich nearshore sediments, magnesium potassium and sodium are all taken up by solid (authigenic) phases, especially silicates, and are therefore depleted in pore solutions. Calcium is often enriched and strontium may reach over 100 mg/kg where strong recrystallization of biogenic calcite has occurred. Boron is depleted, when its strong temperature-of-squeezing effect is compensated for. These reactions become more prominent as sediments reach increasingly consolidated stages. An example of pronounced 'reverse weathering' of authigenic reactions (MacKenzie and Garrels, 1966) and their influence on interstitial waters is provided in Fig. 2. Differences in ionic constituents of interstitial waters from standard sea water with depth are depicted for a station on the

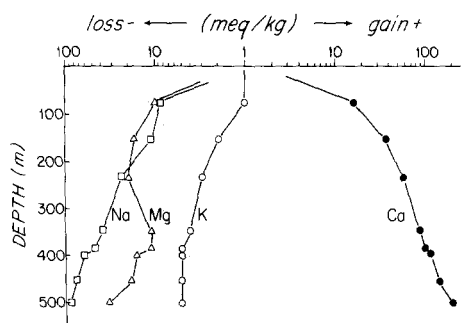


Fig. 2. Change in interstitial constituents with depth, Site 217, DSDP, Ninetyeast Ridge, Indian Ocean. Location of site is 8° 55.6' N, 90° 32.3' E, at water depth 3020 m. Values refer to difference between standard sea water and observed concentration of indicated elements, in mg-equiv/kg. Values at 7 m are very close to standard ocean water. Depth in figure is with reference to sea floor. The sediments penetrated are generally clayey biogenic oozes (foram, coccolith and radiolarian rich), and are Upper Cretaceous in age for the lowest samples reported here. Water content decreases from more than 50 per cent near surface to 18 per cent at 502 m. Chloride increases from 19.3 to 22.0 at 502 m. Data from Initial Reports volume for Leg 22 (in press).

E. flank of Ninetyeast Ridge, Indian Ocean, in 3020 m of water.

Submarine aquifers—buried salt deposits

In certain areas, usually relatively near continents, major changes in total interstitial salinity and chlorinity as well as ionic ratios occur. Submarine discharge from aquifers in continuity with meteoric water sources on land may freshen submarine strata as much as 100 km from shore. Above buried salt deposits diffusion of dissolved salts creates characteristic enrichments in the composition of surrounding pore fluids; these 'halo' effects may penetrate as far as 3 km or more through sedimentary strata, given sufficient geologic time. Where late stage evaporite minerals are in-

volved unusual concentrations of magnesium, potassium, boron and bromine have been observed in overlying pore fluids. These phenomena have demonstrated the effectiveness of diffusion through sediments as well as provided a prospecting method for buried salt domes.

Unsolved problems

Not all methodical problems have been solved. For example, gas concentrations in interstitial waters are still inadequately known (Hammond *et al.*, 1973) and interstitial species influenced by gas exchange are sometimes problematical owing to events contingent on the raising of cores from *in situ* pressures and temperatures. The temperature-of-squeezing effect and its influence on exchange capacity must be reckoned with, and redox-active species such as iron have raised serious problems. Study of organic constituents is still in an early stage, and interstitial compositions sometimes raise complex and difficult problems regarding mineral equilibria.

One can agree with Murthy and Ferrell (1972) on one point, namely that good results can be assured only if sediments are extracted soon after their recovery from the natural environment. Indeed, *in situ* recovery of pore fluid may yield the next major advances in pore fluid studies (Sayles *et al.*, 1973).

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Résumé—La conclusion de Murthy et Ferrell (1972) selon laquelle les études sur l'eau interstitielle n'ont pas abouti à des résultats clairs, est critiquée en remarquant que les auteurs ne se sont pas appuyés sur une masse considérable de données constituée, en particulier, par les travaux russes depuis 1950 et par les résultats du Deep Sea Drilling Project. Les systèmes de filtration sous pression pour extraire les solutions interstitielles sont, en général, des méthodes de choix pour les études marines; ces méthodes ont atteint une qualité et une reproductibilité substantielles. Bien que des manques de connaissance et des problèmes subsistent encore, de nombreux aspects de la composition interstitielle des sédiments marins ont été clarifiés; ils portent sur la constance notable de la composition des solutions interstitielles dans les dépôts pélagiques en mer profonde, l'appauvrissement en cations interstitiels dû à la formation de minéraux authigènes dans les sédiments accumulés plus rapidement (en particulier les sédiments terrigènes), et sur des phénomènes spéciaux se passant dans des sédiments recouvrant des dépôts de sel.

Kurzreferat—Die Schlußfolgerung von Murthy und Ferrell (1972), daß sich Untersuchungen des Porenwassers im Zustand der Verwirrung befinden, wird kritisiert, weil die Autoren eine bemerkenswerte Anzahl von Ergebnissen, insbesondere sowjetischer Untersuchungen seit den 1950 Jahren sowie die Befunde des Tiefseebohrprojektes nicht berücksichtigt haben. Druckfiltrationssysteme zur Extraktion von Porenwasser sind gegenwärtig die Methodender Wahl für marine Untersuchungen und haben wesentliche Zuverlässigkeit und Reproduzierbarkeit erreicht. Obwohl Lücken und Probleme fortbestehen, wurden viele Aspekte der Zusammensetzung der Porenlösungen mariner Sedimente geklärt. Zu diesen gehören die wesentliche Konstanz der Zusammensetzung von Porenwässern in pelagischen Tiefseeablagerungen, die Verarmung an Porenkationen infolge der authigenen Mineralbildung in schneller abgelagerter (vorwiegend terrigenen) Sedimenten sowie spezielle Erscheinungen in Sedimenten über Salzablagerungen.

Резюме— Утверждение Мурти и Феррелла (1972 г.), что исследование промежуточной воды находится в хаотическом состоянии, критикуется на том основании, что авторы не рассмотрели значительную часть имеющихся данных, особенно исследований проведенных советскими работниками с 50-тых годов и не ознакомились с результатами проектов бурения на больших морских глубинах. В настоящее время для изучения морей и для извлечения промежуточной воды выбрали систему фильтрации под давлением и полученные результаты оказались надежными и воспроизводимыми. Хотя, имеются еще пробелы и проблемы, все таки многие стороны состава импренированных морских отложений были выяснены; как то: заметную постоянность состава промежуточных вод в отложениях глубоких морей, истощение промежуточных катионов, вследствие образования аутигенного минерала в более быстро скопившихся отложениях (особенно терригенных), и специальная особенность залегания над пластами соли.