

Review

Fractionation of surface sediment fines based on a coupled sieve–SPLITT (split flow thin cell) method

Laurent Coppola^{a,b,*}, Örjan Gustafsson^b, Per Andersson^a, Pär Axelsson^b

^aSwedish Museum of Natural History, Laboratory for Isotope Geology, Box 50007, 10405 Stockholm, Sweden

^bStockholm University, Department of Applied Environmental Science (ITM), 10691 Stockholm, Sweden

Received 5 April 2004; received in revised form 12 January 2005

Abstract

In traditional sediment grain-size separation using sieve technique, the bulk of the organic matter passes through the smallest mesh size (generally 38 μm) and is not further fractionated. In this study, a common sieve separation has therefore been coupled with an extra high capacity split flow thin cell fractionation (EHC-SPLITT) instrument to separate the bulk surface sediment not only into size-based sieve fractions (>100, 63–100, 38–63 and <38 μm) but particularly to further fractionate hydrodynamically the fine fraction (<38 μm) using the EHC-SPLITT. Compared to the few previous studies using a smaller high capacity (HC) SPLITT cell, the EHC-SPLITT evaluated in detail here has several advantages (e.g., 23 times higher throughput and allowance for large particle diameters). First, the EHC-SPLITT was calibrated with particle standards. Then, its ability to fractionate fine surface sediments hydrodynamically was demonstrated with material from biogeochemically distinct regimes using two cutoff velocities (1 and 6 m d^{-1}). The results from particle standards indicated a good agreement between theory and experiment and a satisfactory mass recovery for the sieve–SPLITT method (80–97%) was observed for sediment samples. The mass distributions revealed that particles <38 μm were predominant (70–90%), indicating the large need for a technique such as the EHC-SPLITT to further fractionate the fine particles. There were clearly different compositions in the EHC-SPLITT-mediated sub-fractions of the sediment fines as indicated by analyses of organic and inorganic parameters (POC, Si, Fe and Al). The EHC-SPLITT technique has the potential to provide information of great utility to studies of benthic boundary layer transport and off-shelf export and how such processes fractionate geochemical signals.

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Keywords: Colloids; Gravitoids; Size separation

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*Corresponding author. Swedish Museum of Natural History, Laboratory for Isotope Geology, Box 50007, 10405 Stockholm, Sweden. Tel.: +46 851954234.

E-mail addresses: laurent.coppola@nrm.se (L. Coppola), orjan.gustafsson@itm.su.se (Ö. Gustafsson).

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1. Introduction

Studying composition of coastal surface sediment allows us to understand the fate of the terrestrial organic matter exported to the continental shelf as well as the factors controlling the content and preservation of organic matter in marine sediments (Keil et al., 1994; Hedges and Keil, 1995; Bianchi et al., 2002). Suspended particles and aggregates in the river and estuarine waters are hydrodynamically sorted during transport across the continental shelf. When the particles/aggregates reach the ocean and the horizontal transport rate decreases, the gravitational forces increase in importance, forcing the particulate matter to settle and sediment. This suggests that different particles/aggregates sediment at different distances from the river mouth to the open ocean (Gibbs et al., 1989). Settling velocity is governed by shape, size and density of the particles/aggregates under gravitational force. The properties that control the settling velocity also control hydrodynamic sorting and resuspension/resedimentation processes.

It is suggested that there is a strong relationship between sediment grain size and total organic matter due to the greater sorption capacity of fine sediments with large surface areas (Mayer, 1994a). This indicates that the size distribution of sediment is important for the study of the carbon content in sediments. Especially, it is important to examine the fine particles (<38 μm) because most of organic and inorganic compounds are concentrated in this fraction (Mayer, 1994a; Bergamaschi et al., 1997; Keil et al., 1997).

In previous studies, most techniques used to separate particles depend on size only, which is unlikely to satisfactorily simulate the natural fractionation occurring at the outlet of most rivers. Other methods have been used but they included lack of precise particle separation in size classes <20 μm , low throughput, contamination, loss of material to the separation medium and reliance on density or size alone to isolate the discrete classes (Keil et al., 1994).

In this paper, we evaluated and tested in detail a little-used method called SPLITT (split flow thin cell fractionation) for further separation of sieve-passing

fine (<38 μm) particles. The SPLITT method is based on hydrodynamic fractionation of the particles based on the size, density, shape and settling velocity. It seeks to imitate the natural settling of particulate matter and large volumes can be processed rapidly.

The SPLITT technique was first introduced by Giddings (1985) and Springston et al. (1987). SPLITT fractionation has been applied to some natural aquatic systems such as hydrodynamic sorting of bottom sediments (Keil et al., 1994), river particles (Contado et al., 1997) and surface ocean particles (Gustafsson et al., 2000), as well as characterization of dust and ground water particulates (Lee et al., 2001) and more recently to separate quantitatively diatom frustules from lake sediments (Rings et al., 2004).

However, previous studies in water research used only a so-called high capacity (HC) SPLITT cell (Keil et al., 1994). It is the objective of this present study to thoroughly test and demonstrate the feasibility of the extra high capacity (EHC) SPLITT to aquatic particles. The EHC-SPLITT cell enables processing 20 times more sample in a given amount of time compared to the previously applied HC-SPLITT cell and, further, pre-concentration of particles before the EHC-SPLITT fractionation is not required for geochemical measurements. In this study, the method was first tested with silica beads to calibrate the EHC-SPLITT cell. Then, its applicability to real aquatic sediments was demonstrated by analysis of organic and inorganic components (POC, Al, Si and Fe) in separated fractions of surface sediment samples from five biogeochemically distinct regimes.

2. Materials and methods

2.1. The SPLITT principle

The SPLITT is a separation method where particles are gently sorted during laminar fluid flow in a thin channel based on a combination of their size, wet density and diffusion coefficient. The SPLITT has an

easily adjusted “cutoff” separation based on particle settling velocity (Giddings, 1985; Fuh et al., 1992).

The SPLITT cell is composed of laminated plastic and glass boards with a metal sheet in the middle in order to create a thin cell within the system. There is a pair of inlets on one side of the cell and a pair of outlets on the other side (Fig. 1). The sample suspension, which is referred to as the feed, is introduced at the upper inlet (called a') and a carrier solution free of particles (here: deionized water) at the lower inlet (called b'). These liquid media are continuously introduced by pumps, which can be set to different flow rates and cutoffs. It is important that the particles are disaggregated and the concentration of suspended solids is low. Keil et al. (1994) estimated that the optimum concentration of particles in the feed sample is ~1.4 weight percent suspended solids. This means that up to 14 g suspended solids per liter is allowed to not change the viscosity of the solution.

Inside the SPLITT, the inlet splitting plane separates the sample and carrier solutions as the particles are advected laterally in the cell with the laminar flow; they are affected by the gravitational pull (Fig. 1). Depending on the horizontal velocity of the suspension in the cell and the particle properties, particles above a certain settling velocity cutoff will sink before reaching the outlet splitting plane. Particles passing above the outlet splitting plane are termed large colloids and exit through the upper outlet (a; Fig. 1). Those that sink beneath are termed gravitoids and exit through the lower outlet (b). The gravitoids and the large colloids are then collected separately. Different sedimentation rate cutoffs can be obtained by adjusting inlet and outlet flow rates.

The cutoff sedimentation rate (U_{cutoff} in m d^{-1}) is defined as the rate when half of the particles exit as gravitoids and the remaining as large colloids in the SPLITT. The SPLITT flow rates required to achieve a desired settling velocity cutoff (m d^{-1}) is calculated from the equation:

$$U_{\text{cutoff}} = \frac{V_a - 0.5V_{a'}}{b \times L}, \quad (1)$$

where V_a and $V_{a'}$ represent the flow rates (ml min^{-1}) at the upper outlet and inlet, respectively. The terms b and L (cm) correspond to the cell breadth and the cell length between the two splitter ends. In this study, the flow rates were set to test the SPLITT technique at 1 and 6 m d^{-1} cutoff.

2.2. The advantages of the sieving–SPLITT fractionation method

The bulk sediment samples were first fractionated by wet sieving, which separates large particles due to size only (Fig. 1). The sieving fractionation is determined by the mesh size of the sieve. In this study, we used a shaker (Retsch AS200 Control) with stainless-steel sieves (Retsch 100 mm dia-

meter) with mesh sizes 500, 250, 100, 63 and $38 \mu\text{m}$. The fraction that passes through all sieves ($<38 \mu\text{m}$ i.e. fine fraction) was collected in a glass bottle (around 10 l) and subsequently used in the EHC-SPLITT. A small part (20 ml) of this fine fraction was filtered through a Nucleopore filter ($0.45 \mu\text{m}$) to estimate the concentration of particles. Each of the sieve fractions and filters were dried in oven (60°C) during 24 h and the mass was weighed to assess the mass recovery of the sieve procedure.

In this study, we tested an EHC-SPLITT cell (FFFractionation LLC, Salt Lake City, UT, USA) to separate fine sediment particles. The EHC-SPLITT cell dimensions were breadth 14 cm, length 100 cm and height $500 \mu\text{m}$ and consequently has a volume capacity of 70 ml which is 23 times higher than the HC-SPLITT cell (Keil et al., 1994; Gustafsson et al., 2000; Rings et al., 2004). The feed solution ($<38 \mu\text{m}$) was kept in a glass beaker on a shaking table and agitated at 50 rpm. The sample and carrier solutions were introduced to the SPLITT cell, passing through a pulse dampener (bubble trap), by peristaltic pumps using silicone tubing. The inlet flow rates V_a and $V_{b'}$ were adjusted by changing the settings on the two inlet pumps. The SPLITT fractionated solutions (gravitoids and large colloids) were collected in pre-weighed glass bottles and the mass of both fractions was determined after centrifugation and drying in the oven at 60°C .

The large size of the EHC-SPLITT cell is advantageous for the separation of particles in sediment samples. For example, the high capacity of the EHC-SPLITT cell and the appropriate particle concentration in the feed solution (10 g/l) allow to fractionate rapidly a large volume of sample (4 l in 160 min). In the present study, the feed concentration was around 0.4 g l^{-1} .

2.3. The EHC-SPLITT calibration

In contrast to most previous applications, in this work, the EHC-SPLITT was explicitly calibrated by using spherical monodisperse particle standards (silica beads of $7.8 \pm 1.0 \mu\text{m}$, 2.52 g cm^{-3} ; Sigma Chemicals). These dense inorganic particles have settling velocity (4.89 m d^{-1}) similar to detrital particles predominant in surface sediments. The silica beads were mixed in deionized water and fractionated by the EHC-SPLITT cell operated at varying cutoff velocity. To verify the EHC-SPLITT cutoff, quantification of two SPLITT fractions were evaluated and compared with theoretical predictions by Stoke's Law.

2.4. Demonstration of sieving/EHC-SPLITT to surface sediment samples

To evaluate the sieving–SPLITT method, a variety of different surface sediment samples were tested. The samples were collected from the surface layer of

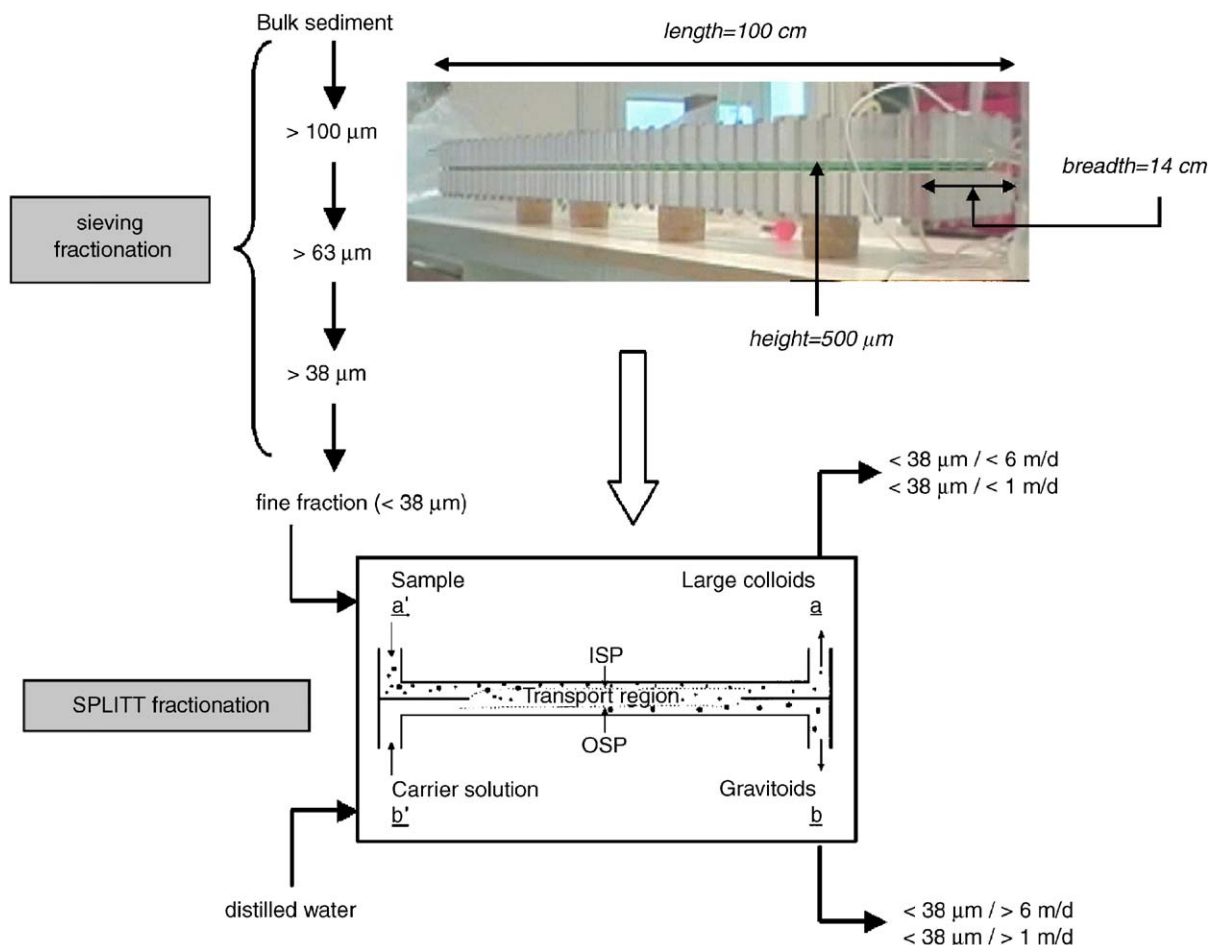


Fig. 1. The sieving–SPLITT fractionation process. The SPLITT assembled cell dimensions were breadth 14 cm, length 100 cm and height of the cell channel 500 μm . The ISP and OSP represent the inlet and outlet splitting planes.

sediments (0–2 cm) underlying different biogeochemical regimes (Fig. 2): Alaska region (Yukon River and Prudhoe Bay), Peru Margin and Swedish coast (Koster Fjord and Örserum Bay).

2.5. Chemical analysis

Subsamples of sieving–SPLITT fractions were analyzed for the concentration of particulate organic carbon (POC) by Europa IRMS (Isotope Ratio monitoring gas Mass Spectrometer in cooperation with UC Davis Laboratory, USA). The preparation of sieving–SPLITT fractions for IRMS analysis was identical to that of Gustafsson et al. (1997). Subsamples of sieving–SPLITT fractions (10–20 mg dry weight) were also subjected to inorganic element analysis (Al, Si, Fe) using inductively coupled plasma-atomic emission spectrometry (ICP-AES) in cooperation with SGAB Analytica AB (Luleå,

Sweden). For the SPLITT fractions, the subsamples were obtained after filtration onto nitrocellulose filters (0.45 μm). All samples were acidified with ultra-pure acid before analyses for a complete digestion of particles and filter. Measurements were done on each fraction without pre-concentration following previously described method by Ingri et al. (2000).

3. Results and discussion

3.1. Calibration result

The EHC-SPLITT fractionation of standard mono-disperse silica beads is in agreement with theoretical predictions by Stoke's Law (Fig. 3). The calibration lines are theoretical fractograms predicted by Stoke's Law taking into account the non-Gaussian mass distribution. This experiment confirms that a laminar flow regime was

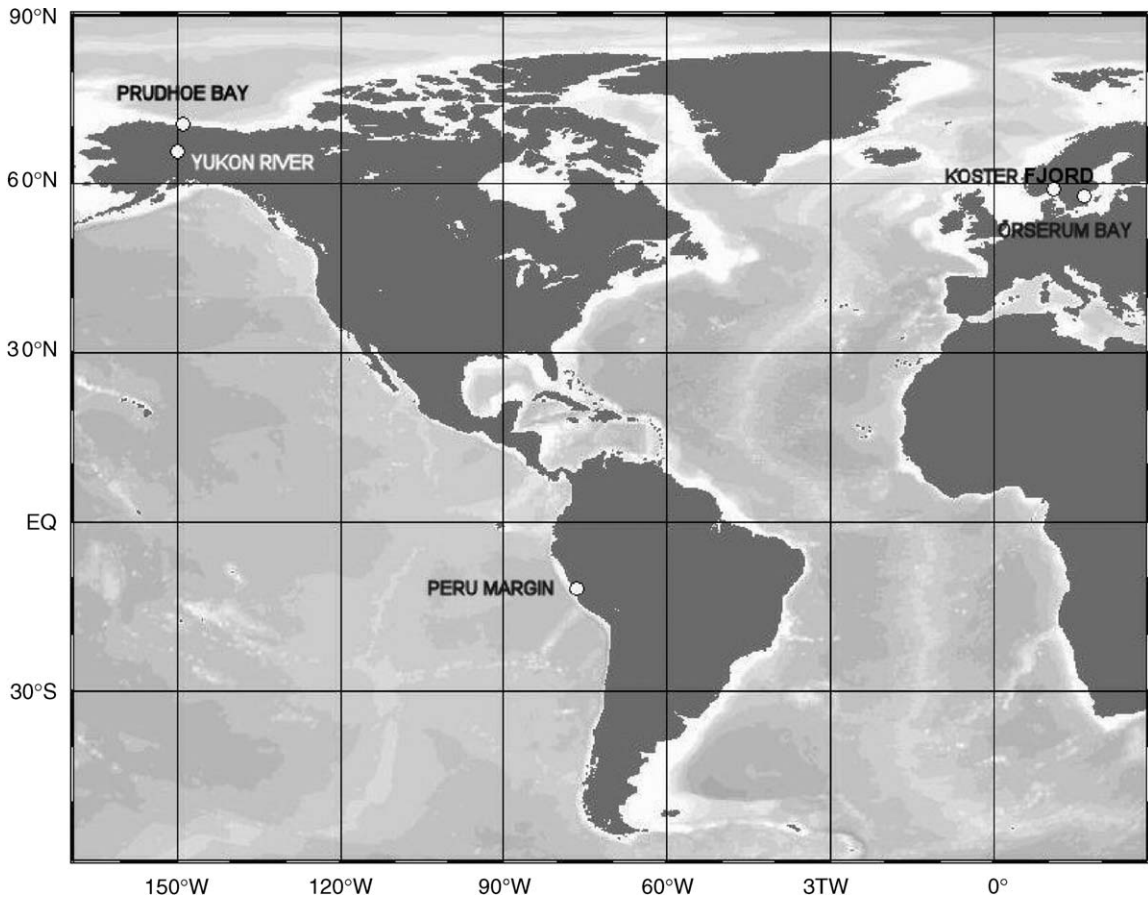


Fig. 2. Location of the surface sediment test samples (Yukon 65.9°N/149.7°W, Prudhoe 70.8°N/149.0°W, Peru 12°S/77.3°W, Koster 58.9°N/11.1°E and Örserum 57.8°N/16.7°E).

established in the EHC-SPLITT cell. The cutoff velocities generated by the fractions in the exit of the EHC-SPLITT (gravitoids and large colloids) were in the same range as predicted by the theoretical model (from 1 to 6 m d^{-1}). The cutoff velocity data was estimated graphically from the intersection of the correlation lines plotted for the gravitoids and colloids mass data, respectively, and compared to the value predicted by the intersection of the Stoke's Law curves. The experimental value was 5.0–5.5 and 4.4 m d^{-1} from the Stoke's Law model for 7.8 μm silica beads. Similar SPLITT calibration results have previously been reported for this and other standard particles for the HC-SPLITT instrument (Gustafsson et al., 2000).

3.2. Distribution of the sieve–SPLITT fractions

To be able to estimate the recovery of the sieve fractionation, each of the sieve fractions were dried and weighed. The sum of these masses was compared to the

mass of the bulk sediment introduced to the sieve system. Before sieving, the dry/wet ratio of the bulk sediment was determined. The recovery was calculated according to Eq (2), where m_i represents the dry mass of the sieve fraction i :

$$\text{Recovery} = \frac{m_{500} + m_{250} + m_{100} + m_{63} + m_{38} + m_{<38}}{m_{\text{tot}}} [\%]. \quad (2)$$

The results of the sieve fractions indicated a good mass recovery for all five sediments ranging from 75% to 99% (Fig. 4). The mass distributions suggest that there is a significant variation between these different types of sediment (Fig. 4). The fine fraction ($<38 \mu\text{m}$) represents more than 70% of the total mass for Peru, Koster and Örserum samples. For Yukon and Prudhoe samples, the fraction 63–100 and 38–63 μm are higher even if the fine fraction stays the predominant fraction in the mass sieve distribution. On average for all sediment samples, the fine fraction represents 64% of the bulk sediment, which indicates that sieve fractionation alone

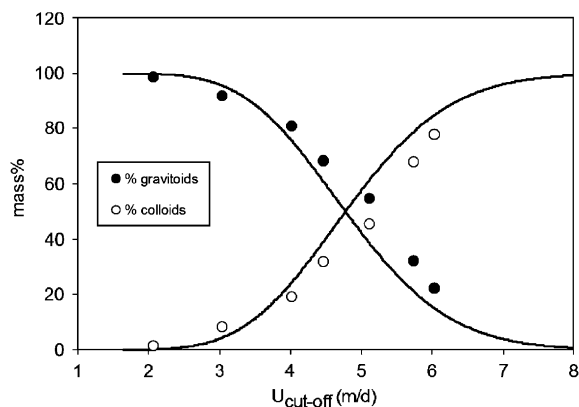


Fig. 3. Cutoff calibration graph. The lines are the theoretical fractogram predicted by Stoke's Law. The separation is based on differential settling of a spherical monodisperse particle standard (silica beads of $7.8 \mu\text{m} \pm 1.0 \mu\text{m}$).

is not providing sufficient separation of the fine fraction. Thus, there is clearly a need for a technique that is able to separate among different fine fractions with a high throughput.

The SPLITT mass recovery ($R\%$) was calculated as the ratio between the amounts of an element (n) found in the sum of the large colloid and the gravitoid fractions to that in the feed fraction (bulk particles $<38 \mu\text{m}$):

$$R\% = \frac{n_{\text{colloids}} + n_{\text{gravitoids}}}{n_{\text{feed}}} \quad (3)$$

The SPLITT fractionation was tested for cutoff velocities 1 and 6 m d^{-1} (Table 1) estimated from Eq (1) and from the flow rates at the upper outlet and inlet. The gravitoid fraction with velocity higher than 6 m d^{-1} represents particles $<38 \mu\text{m}$ and $>6 \text{ m d}^{-1}$ whereas the ultra-fine large colloid fraction with velocity lower than 1 m d^{-1} corresponds to particles $<38 \mu\text{m}$ and $<1 \text{ m d}^{-1}$. Between these two fractions, there is an intermediate fraction at $<38 \mu\text{m}$ and $1\text{--}6 \text{ m d}^{-1}$.

The experiments demonstrate that good mass balance is obtainable with the EHC-SPLITT (Fig. 4). The mass recovery averages 88% and the two SPLITT runs for each sediment show a similar mass fractionation. For Yukon and Prudhoe samples, the mass fraction is higher for particles ranging from $63 \mu\text{m}$ to $<38 \mu\text{m}$ – $>6 \text{ m d}^{-1}$. This feature is characteristic to coarse river and shelf sediments. In the opposite, the mass fraction is very high for fine particles lower than $<38 \mu\text{m}$ – $<6 \text{ m d}^{-1}$ for marine and brackish sediments (Peru, Koster and Örserum). This case demonstrates the importance to further fractionate the fine particles.

Comparing these gravimetric results with theoretical curves from Stoke's Law, the diagram in Fig. 5 gives a complete picture of the settling velocity of the sediment fines. There are clearly different sediment fine properties depending on the sample location. For the Alaska samples (Yukon and Prudhoe), 47% of the particle size $<38 \mu\text{m}$ (fine particles) settle faster than 6 m d^{-1} whereas only 19% of them settle lower than 1 m d^{-1} . Peru and Scandinavian (Koster and Örserum) samples, more influenced by the marine environment, have only 7% of the fine particles settling faster than 6 m d^{-1} . Between 40% and 50% of the fine particles have a sinking rate between 1 and 6 m d^{-1} and an equal fraction is found in the $<1 \text{ m d}^{-1}$ settling pool.

3.3. Carbon data

The weight percent of organic carbon ($\% \text{ POC}$) was measured in the sieve and SPLITT fractions and the carbon recovery was calculated for both fractionation methods (Fig. 6). The total recovery for the organic carbon ranged from 71% to 107% . This demonstrates the efficiency of both fractionation methods for the organic carbon in all sediment fractions.

One main characteristic of the POC distribution is the large difference between the sieve and SPLITT fractions. This aspect is clearly evident for Yukon, Peru and Koster sediments where the ultra-fine fractions have a very high organic carbon content (from 1.5% to 8%). For the marine sediments (Peru and Koster), the ultra-fine fractions ($<38 \mu\text{m}$ – $<1 \text{ m d}^{-1}$) had $\% \text{ POC}$ 10 times higher than the $38\text{--}63 \mu\text{m}$ and the $<38 \mu\text{m}$ – $>6 \text{ m d}^{-1}$ fractions. For Örserum sample, the large particles ($>100 \mu\text{m}$) have the highest POC content. These results are in overall agreement with the origin of sediments. For example, a high carbon value was also observed by Bergamaschi et al. (1997; $1.5\text{--}21.6\%$) in the Peru Margin confirming the validity of the sieve-SPLITT method. This also indicates the importance of the fine particles, where the major carbon content is located and thus the motivation to focus biogeochemical studies on this fraction of particles. An ambition that is now attainable with the EHC-SPLITT technique is to study the influence of grain size on organic carbon preservation in marine sediments (Canfield, 1994; Bergamaschi et al., 1997) and the relationships between mineral surfaces and organic carbon concentrations in sediments (Mayer, 1994b).

3.4. Elemental ratios in the particulate SPLITT pools

In order to investigate the possible variation in composition of the three SPLITT pools (feed $<38 \mu\text{m}$, gravitoids and large colloids) as a function of the cutoff velocities (1 and 6 m d^{-1}), the distribution of the

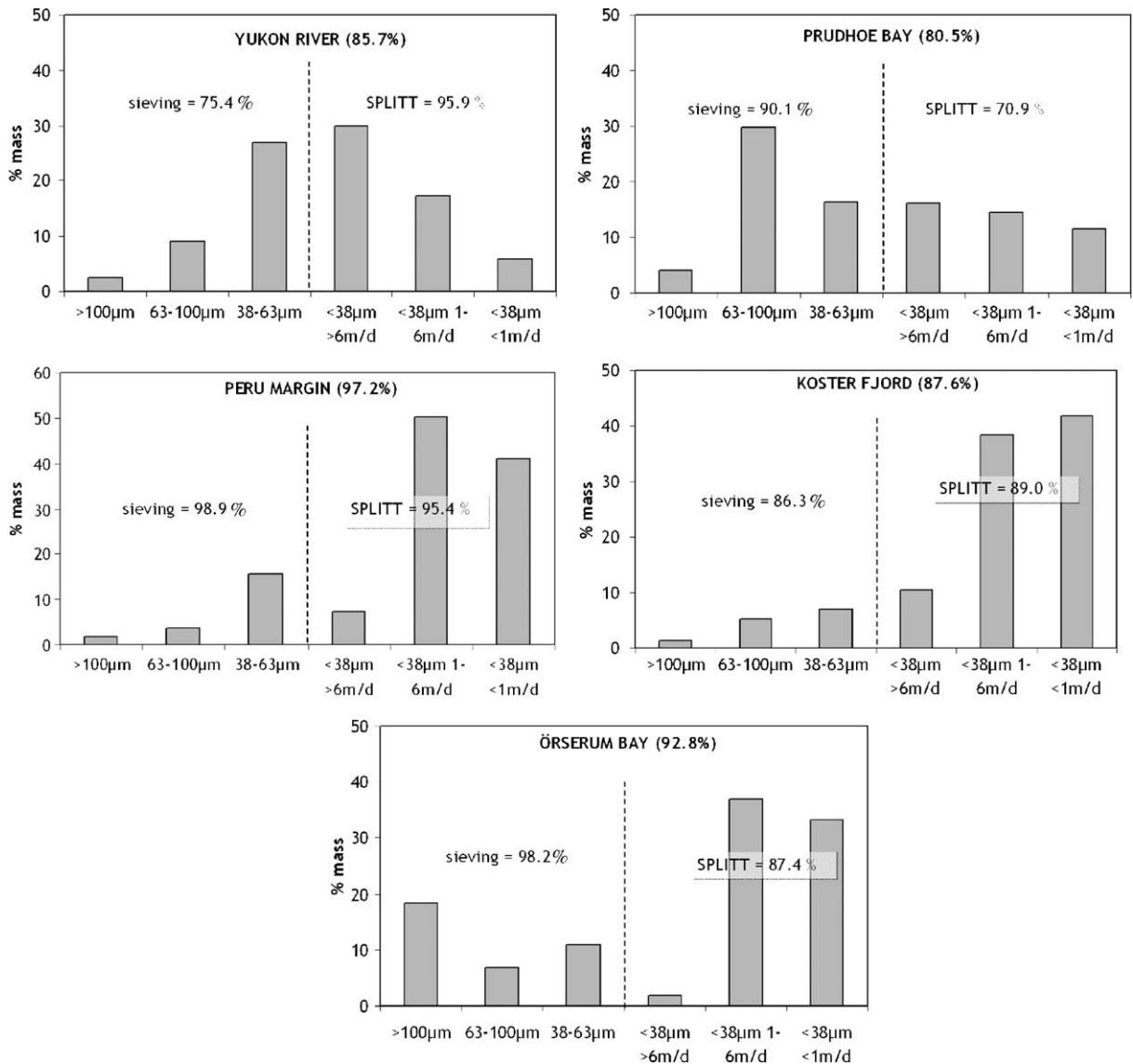


Fig. 4. Mass distribution of sieve-SPLITT fractions of sediment samples. The dashed line delimits the sieve and the SPLITT fractionation methods. The cutoff velocities used for the SPLITT fractionation were 1 and 6 m d⁻¹. Values listed above the bars are the mass recovery for sieve and SPLITT fractionations. The value in comma represents the total mass recovery.

Table 1
EHC-SPLITT flow rates and settling velocity cutoff used in this study

U_{cutoff} (m d ⁻¹)	$V_{a'}$ (ml min ⁻¹)	V_a (ml min ⁻¹)	$V_{b'}$ (ml min ⁻¹)	V_b (ml min ⁻¹)
1.0	30–46	115–120	95–108	21–23
6.0	110–120	630–650	650–660	125–130

inorganic elements iron (Fe) and silicon (Si) were normalized to that of aluminium (Al). Al is not very bioactive and can be used as a measure of detrital material and Si/Al and Fe/Al ratios can be compared to

the average content of the continental crust. Crustal sources are likely for the detrital material and element/Al ratios higher than the average crust indicating a non-detrital enrichment of the element in the analyzed

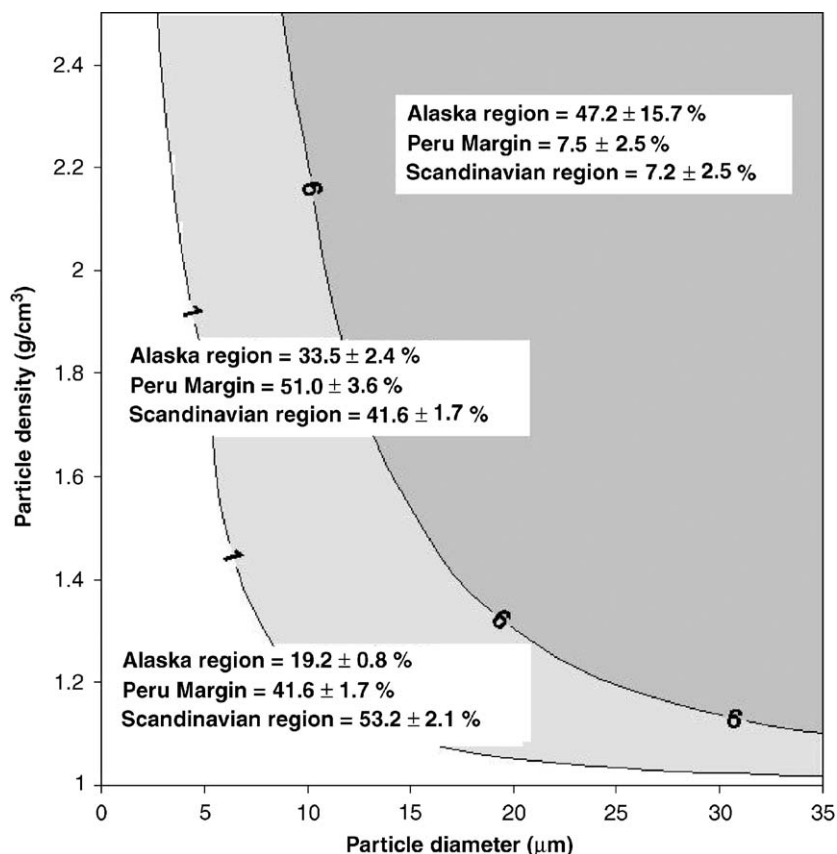


Fig. 5. The distribution of the fine particles (<38 μm) among three different settling velocity fractions. The curved lines represent 1 and 6 m d⁻¹ settling velocity modelling by Stoke's Law. The settling characteristics of the tested sediments grouped into three categories: Alaska region (Yukon and Prudhoe), Peru Margin and the Scandinavian region (Koster and Örserum).

sample. The relatively high Si/Al ratio for the particles <38 μm> 6 m d⁻¹ indicates a specific particle phase with high Si content (e.g. diatoms or sand) in the fast sinking fraction (Fig. 7a). The Si/Al ratios in the other SPLITT fractions are similar or lower than in the feed (<38 μm), which indicates that detrital material is likely to dominate.

The Fe/Al ratio in all fractions of the Alaska sediments is close to the average crust value (0.6) demonstrating that the particulate Fe predominantly existed in detrital particles (Fig. 7b). For the marine and brackish sediments, the SPLITT runs reveal that the highest value of the Fe/Al ratio is contained in the ultra-fine particles (<38 μm and <6 m d⁻¹).

4. Conclusion

The EHC-SPLITT is a non-destructive and simple instrument used to fractionate particles/aggregates in natural water into different settling velocities. The large capacity of the EHC cell compared to HC increases the

throughput (23 times higher), the maximum particle size and the volume of sample that it is practically possible to fractionate.

In the present study, the perspective to test the validity of the sieve-SPLITT method was afforded by the fractionation of surface sediments from five different regimes. Calibration using standard particles, never done before, clearly demonstrates that the EHC-SPLITT is well fitted to fractionate particle samples from 1 to 6 m d⁻¹ providing information about the ultra-fine particles distribution.

The fractionation of sediment samples demonstrated the preponderance of fine particles (<38 μm) in most sediment samples. Thus, the sieve separation combined with EHC-SPLITT can further separate the fines, which are important to elucidate biogeochemical processes. The carbon and inorganic elements confirmed this feature for all sediments and revealed differences could exist in between subcomponents of the SPLITT fractions. Finally, this paper suggests that many processes could be elucidated with the EHC-SPLITT

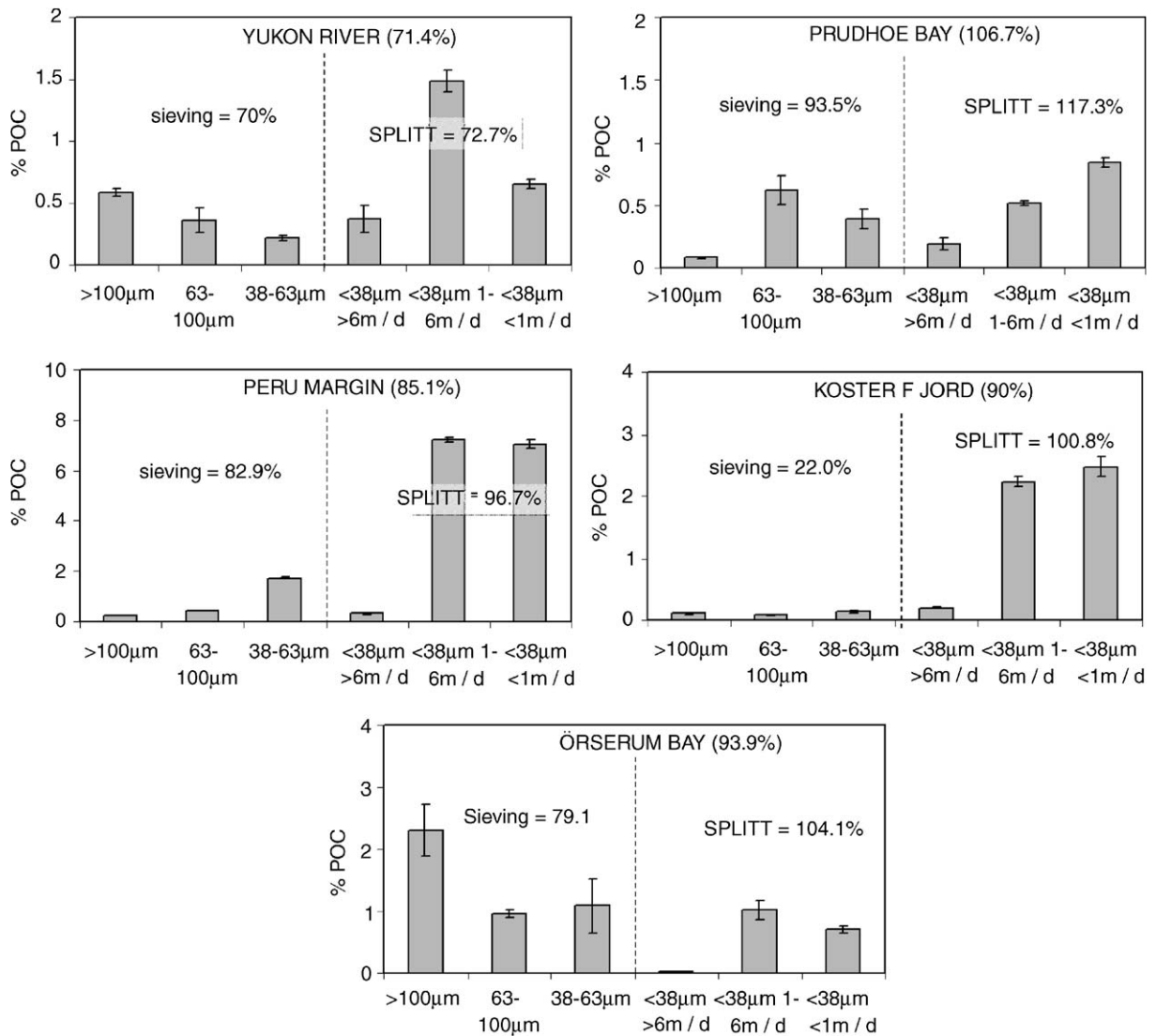


Fig. 6. Organic carbon concentration in sieve-SPLITT fractions. Values listed above the bars are the yield for sieve and SPLITT fractionation. The value in comma represents the total yield. Errors bars represent the standard deviation of the propagated analytical uncertainty derived from triplicate analyses.

fractionation of the fine particle, such as geochemical fractionation within the fine particle pool during off-shelf transport in the benthic boundary layer (Keil and Cowie, 1999). The hydrodynamic particles sorting in ocean margins is a topic that could benefit from the EHC-SPLITT emphasis as, for example, the lateral transport of terrestrial organic matter (Goñi et al., 1997; Goñi et al., 2000; Bianchi et al., 2002) and the dispersion of trace contaminants in the continental margins (Santschi et al., 1997).

Acknowledgments

We are grateful to Jenny Larsson for discussions about the SPLITT fractionation method. Zofia Kukulska is thanked for technical assistance in the laboratory and Johan Ingri for ICP-AES analysis. Thanks also to test sediment donors Drs. Laodong Guo and Tim Eglinton. This study has been carried out with financial support from the Marie Curie Individual Fellowship EVK3-CT-2002-50011 (Laurent Coppola), the Swedish Research Council (VR grant 1-AA/GB 12291-301 to Ö.G. et al.

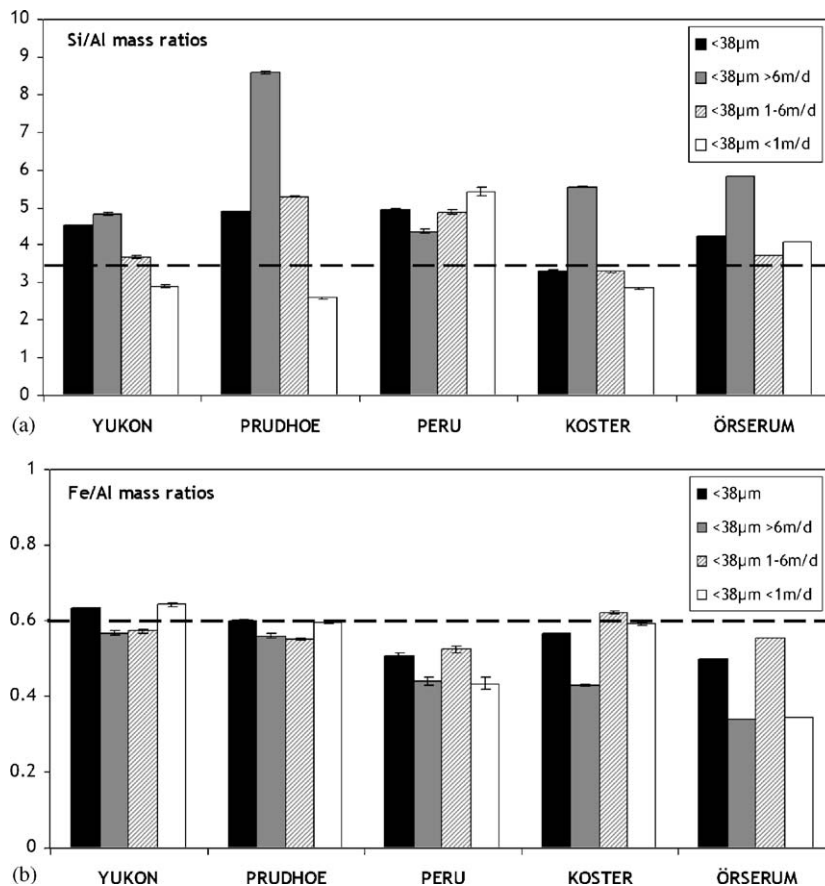


Fig. 7. Elemental mass ratios of the different fine particle SPLITT fractions. The dashed lines represent the continental crust average for Si/Al and Fe/Al mass ratios (3.4 and 0.6, respectively). Error bars represent the standard deviation of the propagated analytical uncertainty derived from triplicate analyses.

and G-AA/GU 6331-316,317 to P.A. et al.). Ö.G. also acknowledges a senior researcher grant from the Swedish Research Council (VR grant 629-2002-2309).

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