



# Measuring sediment grain size across the catchment to reef continuum: Improved methods and environmental insights

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## ABSTRACT

Sediments collected within freshwater, estuarine and marine habitats were used to trial various chemical and physical pre-treatments to develop a systematic protocol for grain-size analysis using laser diffraction. Application of this protocol mitigates the influence of bio-physical processes that may transform grain-size distributions, enabling the characterisation and quantification of 'primary' mineral sediments across the complex freshwater-marine continuum to be more reliably assessed. Application of the protocol to two Great Barrier Reef (Australia) river catchments and their estuaries reveals the ecologically relevant <20 µm fraction comprises a larger component of exported sediment than existing methods indicate. These findings are highly relevant when comparing measured data to grain-size-specific modelled sediment loads and water-quality targets. Finally, adoption of the protocol also improves the environmental interpretation of the influence of 'terrigenous sediment' in marine settings, including quantification of newly-delivered flood plume sediment.

## 1. Introduction

Grain size is a fundamental measure in the field of sedimentology. It is used to quantify sediment size, composition and texture, to understand geochemical properties, and to infer sediment transport and sorting dynamics (Sperazza et al., 2004; Walling et al., 2000; Blott and Pye, 2012; Owens et al., 2016). Within the context of a catchment to reef continuum setting, grain-size analysis (GSA) is critical to determine the sediment size fractions that may be transported furthest from the stream mouth into the marine environment, and to define the particles likely to influence water clarity during both flood plume and subsequent resuspension events (Bartley et al., 2014; Bainbridge et al., 2018). Research focused on understanding sediment transport processes and tracing the source of eroded sediments to prioritise remediation sites requires a grain size methodology that can be applied to suspended sediment samples collected across the complex freshwater-marine continuum. This includes particles suspended in freshwater through the catchment, the estuarine mixing zone, and finally within the marine environment. Within each of these zones, physical and biogeochemical processes

interact which can alter the composition, nature and transport potential of suspended sediments (Bainbridge et al., 2018).

Several well-established techniques are available to measure sediment grain size, including wet sieving and settling methods (e.g. hydrometer, pipette, rapid sediment analyser), image analysis and laser diffraction (Eshel et al., 2004; Sperazza et al., 2004; Özer et al., 2010; Fisher et al., 2017; Bittelli et al., 2019). Laser diffraction measures the diameter of particles according to the intensity of scattered light (using Mie or Fraunhofer theory) from a dispersed particulate sample (International Organisation for Standardisation, 2020). Wider access to improved laser diffraction instruments has increased their use in recent years, with advantages over traditional settling techniques including the speed of analysis and the capacity to precisely measure a complete grain-size distribution of the fine sediment fraction (Eshel et al., 2004; Özer et al., 2010; Fisher et al., 2017; Bittelli et al., 2019). While standardised GSA procedures and protocols are well-established for analysis of soils, suspended sediment and sediment cores (Walling et al., 2000; Snyder et al., 2004; Özer et al., 2010), the details of sample pre-treatment and GSA procedures for marine sediments are more

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complex and varied (Loring and Rantala, 1992; Rodríguez and Uriarte, 2009; Spencer, 2017; Romano et al., 2018; Hunt and Jones, 2019). Marine sediment pre-treatment and GSA procedures will also vary depending on whether the grain sizes of agglomerated in situ particles or the primary constituent particles of which they are composed are the focus (Allen and Thornley, 2004; Gray et al., 2010; Spencer, 2017; Hunt and Jones, 2019).

Grain size analyses of suspended sediment in streams are regularly performed to determine sediment behaviour, transport and likely sediment fate (Walling et al., 2000). Analytical methods are straightforward, with little pre-treatment of sample typically required as the medium is freshwater, with similar properties to the deionised water routinely used as the dispersing medium that delivers the sediments into the laser diffraction instrument. Measuring the grain size of marine sediment is far more complex due to changes in ionic strength and the influence of additional components such as microscopic organisms and other organic matter (e.g. plankton, detritus, biogenic silica) and marine carbonates (e.g. coral, mollusc, coralline algae fragments) (Loring and Rantala, 1992; Reynolds et al., 2010; Romano et al., 2018; Latrille et al., 2019). Low sediment concentrations ( $<5 \text{ mg L}^{-1}$ ) in offshore flood plume waters present further challenges to the collection of the necessary sample volumes required to satisfy obscuration thresholds for reliable GSA (Bainbridge et al., 2012). The in situ, particle aggregate sizes may be required when examining marine sediment transport dynamics, whereas terrigenous sediment transport and source tracing studies require an understanding of the hydrodynamic properties of individual 'primary' sediment grains; investigations of the latter will involve removing the marine components through various sample pre-treatments (Spencer, 2017). Undertaking concurrent in situ and treated analyses provides a more thorough understanding of the source, transport, and fate of suspended sediments, and when considered together can provide insights on the bio-physical processes that influence sediments across the catchment to marine continuum.

This study sought to develop a standardised protocol for the pre-treatment and analysis of sediment grain size in both freshwater and marine environments for application in catchment to reef studies. We use two case study rivers from the Great Barrier Reef (GBR), Australia, where suspended sediment is characterised along transects encompassing freshwater (end-of-river), estuarine, organic-rich flood plume and inshore reef and seagrass habitats. We quantify the influence on sediment grain-size distributions of several standard pre-treatments commonly applied (e.g. dispersal agent, HCl digestion) and identify significant differences in grain size traits according to treatment and sample location. Understanding the nature and significance of these differences is critical to environmental interpretations and management. Quantifying and managing sediment loads is a key component of water-quality management on the Great Barrier Reef (GBR), Australia. An improved characterisation of sediment and associated loads being exported from rivers, and a better understanding of the nature and significance of varying particulate organic matter contents and dominant grain size on sediment transport are required to reduce uncertainties in load estimations, and to improve how this sediment is monitored and modelled from catchment to reef (Bainbridge et al., 2018).

## 2. Methods

### 2.1. Study region

This study focused on two GBR catchment to reef transects, including the Tully River catchment ( $1480 \text{ km}^2$ ) located in the Wet Tropical region and discharging into the northern GBR, and the larger, seasonally Dry Tropical Burdekin River catchment ( $130,000 \text{ km}^2$ ), that drains into the central GBR (Fig. 1). High rainfall and discharge events, concentrated during the wet season and often associated with tropical cyclones and depressions, dominate the flow regimes of both rivers (Lough et al., 2015). Detailed catchment descriptions are provided in Bainbridge et al.

(2009) and (2014). Briefly, the long-term median annual discharge for the Tully River Basin is 3536 GL (1986–2018 inclusive; Gruber et al., 2019), with discharge from this river characterised by elevated dissolved inorganic nitrogen loads (Bainbridge et al., 2009; McCloskey et al., 2021b). This catchment has a tropical rainforest climate (Af) as defined by the Köppen-Geiger climate classification scheme (Peel et al., 2007). The mostly natural (rainforest) upper catchment drains into a coastal floodplain extensively modified by agricultural expansion (sugarcane, horticulture) in the late 20th century (Lewis et al., 2021). In contrast, the Burdekin River has a 'hot semi-arid' climate (Köppen-Geiger classification BSh; Peel et al., 2007), with highly pronounced intra-annual (coefficient of variation 1.3) and also relatively high interannual (coefficient of variation 1.1) river discharge variability (Lewis et al., 2013). The Burdekin long-term median annual discharge is 6290 GL (range: 246–54,031 GL), calculated over a 93-year gauge record to 2014 (Lough et al., 2015). This catchment is dominated ( $>90\%$ ) by cattle grazing across open woodland pastures. Ground cover is typically low, with soils exposed and erosion prone over much of the catchment (Bartley et al., 2014). The Burdekin catchment is the largest single contributor of suspended sediment to the GBR, exporting an average of four million tonnes of sediment per annum (range 0.004–15.74 over the period 1986 to 2010) (Kuhnert et al., 2012). Targeted management efforts are underway to reduce the suspended sediment load exported from the Burdekin catchment to improve turbidity regimes and water clarity in inshore and mid-shelf GBR environments (Bartley et al., 2014; Fabricius et al., 2014, 2016; Bainbridge et al., 2018).

### 2.2. Sampling design and collection

This study forms part of a larger Australian Government National Environmental Science Program (NESP) research project aimed at characterising and tracing the origin(s) and fate of environmentally detrimental sediment across two catchment-to-reef transects of the GBR, including the Tully and Burdekin Rivers. Field sampling campaigns in each transect location targeted:

- 1) end-of-river (freshwater) suspended sediment sampled during wet season high flow discharge events;
- 2) surface water suspended particulate matter (SPM) in the adjacent estuary and resultant flood plume generated by these high-flow discharge events; and
- 3) SPM captured over time in marine sediment traps situated at key inshore reef and seagrass habitats within the inshore GBR. These sediment traps capture a combination of newly delivered flood plume SPM as it is transported across the inshore lagoon and settles from the water column as well as sediments delivered to the traps when benthic deposits composed of previously deposited materials are resuspended during turbulent high-energy periods (Lewis et al., 2020).

Prior to this project, field logistical, technological and analytical constraints have restricted the collection of sufficient sample material for the full characterisation and tracing of terrigenous sediment transported in flood plumes and during subsequent resuspension events within the GBR lagoon. The novel application of the patented SediPump® high-volume filtration system (Stevens, 2019) and SediSampler® sediment trap (Stevens, 2013; Stevens et al., 2020) have been critical in achieving the collection of sufficient sediment mass to perform the required analyses from:

- end-of-river and flood plume surface waters, in situ during high flow events. The SediPump® high-volume filtration system (Integral Aqua Pty Ltd) collects necessary masses ( $>1 \text{ g}$ ) of sample material from low sediment concentration water types (refer to Section 2.2.4). These included low SPM concentration plume waters (typically  $<5$

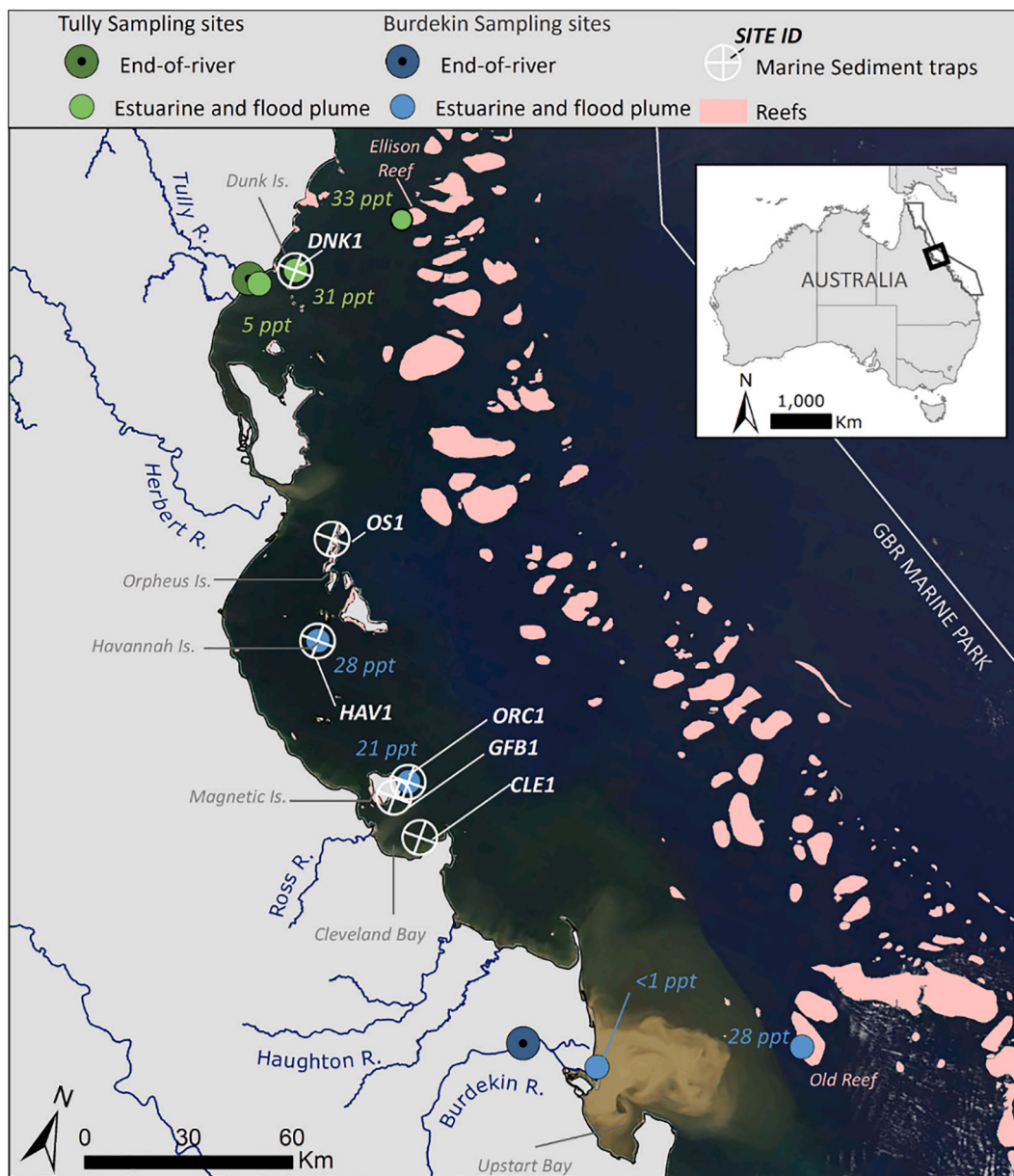


**Fig. 1.** MODIS (Aqua true-colour) satellite image captured on the 12th February 2019 following peak discharge at the Burdekin River mouth, highlighting a visible turbid sediment plume and less turbid secondary waters moving northwards along the Queensland coastline. Reefs shown as pink polygons. End-of-river, estuarine and flood plume sampling sites are shown for the Tully (green circles) and Burdekin (blue circles) Rivers, following a salinity gradient from each river mouth to the respective sediment trap sites (crossed circles) within the inshore Great Barrier Reef. Off the Burdekin River mouth, a sample was also collected from flood plume waters which covered Old Reef on the 15th February 2019. Note the Tully River to Dunk Island sampling sites were collected in a flood plume event during February 2018 (not captured in this image, refer to Supp. Fig. 1c). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

mg L<sup>-1</sup> in salinities 10 PSU or greater; Bainbridge et al., 2012), and rivers of the northern GBR Wet Tropics region with lower sediment concentrations, including the Tully River.

- the subsequent deposition of plume SPM and resuspended material captured within SediSampler® traps fixed at reef/seagrass substrates, with material accumulating over 2–3 month deployment periods. The SediSampler® traps surround an in situ instrument array comprising a nephelometer (turbidity), pressure, light and current meters at each site. The traps and arrays were continuously deployed, on rotating 2–3 month deployments between June 2016 and March 2020 (Lewis et al., 2020).

The sediment samples collected during this project have been used to characterise the physical, chemical and biological traits of the fine

sediments, and to trace their source and transformation, at a level of detail not possible in previous GBR research. Developing a consistent and reproducible methodology for the pre-treatment and analysis for primary grain size of mineral particles in samples collected across this catchment to reef continuum was also essential.

#### 2.2.1. End-of-river (freshwater) samples

Our dataset includes EoR suspended sediment samples from the Burdekin and Tully Rivers captured during high flow events that occurred over the 2016–2017 and 2017–2018 water years, and also the 2018–2019 water year for the Burdekin. Opportunistic, representative samples were also collected from the neighbouring South Johnstone River (Wet Tropics) in 2018, and the Herbert (Wet Tropics), Ross and Haughton (Dry Tropics) Rivers in 2019 (refer to Table 6). In total 15

samples were analysed for the Burdekin, three for the Tully, two for the Ross and one each for the neighbouring South Johnstone, Herbert and Haughton Rivers. The Herbert River sample was a composite of daily sampling across the discharge peak from 4th to 7th February 2019. The grain size methodology development component of this paper focuses mainly on samples collected across the 2019 Burdekin and 2018 Tully transects (EoR, flood plume and marine sediment trap samples).

The EoR sample for the Burdekin River was collected in February 2019 during a prolonged and 'large flood event', with an approximate 1 in 5-year flood return (volume) interval. The Burdekin River discharged 14.5 million ML in the 3-week period between 30 January and 19 February, and peaked at the moderate flood river height classification (Gruber et al., 2020). A manual 'grab' sample collected on the 6th February at Inkerman Bridge (Fig. 1) during the first of multiple flood peaks that occurred during this event had a total suspended solids (TSS) concentration of  $470 \text{ mg L}^{-1}$ .

The Tully EoR sample was collected from the freshwater upstream limit of the estuary on the 9th February 2018 (Fig. 1) during a catchment wide, moderate-sized flood discharge event. TSS concentrations ran at  $17 \text{ mg L}^{-1}$  at the time of sample collection; TSS for this smaller Wet Tropics River are clearly much lower than those produced by the larger and drier Burdekin system. To ensure sufficient sediment was collected to perform all analyses outlined below, the standard manual grab sample (for nutrients and suspended sediments) was augmented with another collected using the SediPump® filtration system.

Additional wet season sampling details for the Burdekin (March 2017, March 2018), Tully (January 2017 and March 2018) and the ancillary rivers sampled are provided in Supp. Table 1.

## 2.2.2. Flood plume samples

Flood plume sampling along the estuarine salinity gradient from the river mouth was conducted immediately following EoR sample collection. Given the large size and duration of the Burdekin flood plume, sample sites targeted the movement of the plume over a number of days guided by near real-time MODIS satellite imagery. Samples were collected when the plume passed over sediment trap and environmental logger sites (HAV1, ORC1) as it moved northwards (11–12th Feb 2019), and later as it moved directly offshore to the mid shelf (Old Reef, 15th Feb 2019) (Fig. 1). Suspended particulate matter (sediment and organic matter) samples were collected using a 10 L container in plume surface waters (i.e. top 0.5 m) at the low salinity ( $<1 \text{ ppt}$ ), high concentration ( $590 \text{ mg L}^{-1}$ ) turbid plume site (Fig. 1). For the three offshore sites where greater mixing with seawater had occurred (i.e. 21–28 ppt), samples were collected using the SediPump® high-volume filtration system to ensure adequate samples sizes to complete the analyses below. Pump durations at each site were 2–3 h, which includes the time to collect both a surface and depth sample (1–2 m above local benthic depth). Only the Old Reef depth sample is discussed in this paper.

Sampling of the plume surface water at the shorter Tully River transect occurred on the 9th February 2018. Sites were located along a salinity gradient transect extending from the freshwater reaches of the estuary out to the sediment trap and environmental instrument array installed at a nearshore reef off Dunk Island (DNK1) (Fig. 1). At this most offshore site, surface waters were well mixed and had a salinity of 31 ppt. Using real-time MODIS satellite imagery to track plume movement and guide sampling, an outer plume transect was repeated 5 days later (14th Feb). This sampling campaign targeted Ellison Reef on the mid-shelf, where the plume extent was clearly visible and surface water salinity was measured to be 33 ppt. The DNK1 trap site was also resampled on this second trip, with increased salinity (33 ppt) indicating further mixing of plume surface waters with seawater during the intervening five days. Because SPM concentrations were low across the entire transect the SediPump® filtration system was used to collect all samples.

Additional wet season flood plume sampling of Burdekin (March 2017, March 2018) and Tully (January 2017 and March 2018) river discharge events are reported in Lewis et al. (2018) and are also referred

to in this study. Flood plume sampling transect locations and associated wet season water-type mapping (e.g. Devlin et al., 2015; Petus et al., 2019) for each event are provided in Supp. Fig. 1a–e. Wet season water type maps are produced using MODIS true colour satellite imagery reclassified to six distinct colour classes defined by their colour properties (Álvarez-Romero et al., 2013). The wet season water types are regrouped into three water types: primary, secondary and tertiary, and characterised by different concentrations of optically active components (suspended sediment, colour dissolved organic matter and chlorophyll *a*), which control the colour of the water and influence the light attenuation, and different contaminant concentrations (Petus et al., 2019). In the GBR, the primary water type is typically found in nearshore areas with resuspended sediments and/or inshore regions of river flood plumes and the secondary waters are typical of coastal waters or the mid-region of river plumes. Sampling targeted both primary (brownish to brownish-green turbid water masses) and secondary (greenish to greenish-blue water masses with increased algal content) water types in flood plumes (hereafter 'water type'). Water types are described in further detail in Supp. material 1.

## 2.2.3. Marine sediment traps

Six marine trap sediment samples were chosen for grain size method development to represent a range of inshore GBR environmental conditions, including reefal and open sediment field locations, relatively low to high swell exposure and bottom currents, as well as seasonal conditions (Table 1). Five SediSamplers® were located in Cleveland and Halifax Bays, on an approximately north-south longitudinal gradient extending from the Burdekin River mouth (Fig. 1). The most northern SediSampler® was deployed at Dunk Island, ~13 km from the Tully River mouth. Collected samples include mineral sediment, organic matter and marine carbonates deposited within the trap over a ~90 day deployment period. Samples from the five Burdekin sites represent wet season flood (ORC1, CLE1, HAV1) and post wet season (GFB1, OS1) deployments between December 2018 and June 2019. The Dunk Island trap (DNK1) was deployed from December 2017 to February 2018. This site is located on a reef-slope on the western side of the island to capture flood plume material exported from the Tully and nearby rivers.

Representative sub-samples were extracted from each highly-concentrated and well-mixed field sample for the analyses outlined in Section 2.3 below.

## 2.2.4. Suitability of the SediPump® sample collection method for grain-size analysis

The SediPump® high-volume filtration system (Stevens, 2019) was configured and initially applied in this study to process large volumes of water ( $>6 \text{ KL}$ ) to collect sufficient fine particles for sediment source tracing analyses. The SediPump® houses a replaceable  $1 \mu\text{m}$  string filter, where particulate matter in the water column is filtered and captured within the carousel. Fixed to a small vessel ( $<6 \text{ m}$ ), several thousand litres of water are pumped from a targeted section of the water column in situ over a short time period (e.g. 2 h) where a  $20 \pm 0.5 \text{ L min}^{-1}$  filter $^{-1}$  processes  $2400 \pm 60 \text{ L}$ . Using this SediPump® set up, 2–4 g of sediment are typically recovered from plume surface and depth waters with TSS concentrations  $<5 \text{ mg L}^{-1}$  within 2 h. This capability presented the opportunity to potentially capture the sample volumes required for additional analyses, including GSA and the larger sample material required for multiple pre-treatment steps. Low concentrations and field logistics have in the past limited sample sizes and the scope of analyses. Full GSA including pre-treatment for organic removal on low SPM concentration flood plume waters has been notoriously difficult, limited by grab samples containing high organic contents (~50%) and relatively low TSS concentrations (i.e.  $<5 \text{ mg L}^{-1}$ ). Previous investigations of Burdekin flood plume SPM samples found the grain sizes were mostly larger than  $1 \mu\text{m}$  (Bainbridge et al., 2012), and hence the  $1 \mu\text{m}$  filter used in the SediPump should capture all particulates in the plume.

To confirm the SediPump® captures the full representative grain-size

distribution of sampled waters, point-in-time ‘grab’ samples were also collected during SediPump® sampling at three sites for comparison. These sites included the Tully flood plume estuarine zone (5 ppt) in 2018 (1-hour pumping time), and the Orchard Rocks (ORC1) and Old Reef sites sampled in the 2019 Burdekin flood plume transect (each pumped for 2 h). Grain-size results are presented for the analysis that included a salt-removal pre-treatment.

### 2.3. Sample pre-treatments

Pre-treatment steps for the samples collected from the EoR, flood plume and marine sites are outlined in Fig. 2. They include mechanical and chemical dispersion of flocculated particles, and the removal of salt, organic and carbonate materials which may influence the measured sediment grain size. Representative aliquots of each sample were prepared for the number of pre-treatment steps required to process each sample type (i.e. freshwater, estuarine or sediment trap). Each pre-treatment is described further below.

**Removal of organic matter [all samples]:** Hydrogen peroxide ( $H_2O_2$ ) is commonly used for wet oxidation of organic matter, with minimal damage to the mineral grains of the sample (Vaasma, 2008; Romano et al., 2018). Concentrated  $H_2O_2$  (30%) was added to a small aliquot of each sample (i.e. 50–100 mL sample) at a 50:50  $H_2O_2$

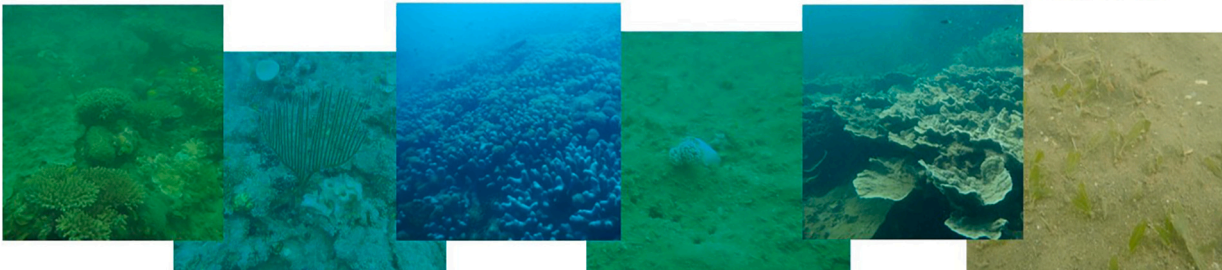
to water dilution. Sub-samples were placed in a fume hood and heated in a water bath at 80 °C until the active reaction (i.e. bubbling) had ceased. The active reaction duration varied between sample types; organic-rich flood plume samples required repeated  $H_2O_2$  additions and heating time over a number of days whereas those with lower organic contents were usually completed within 24 h. Finally, while remaining in the water bath samples were rinsed and decanted with RO water until  $H_2O_2$  concentrations were  $<1 \text{ mg L}^{-1}$  (measured using Quantofix® peroxide test strips).

**Salt removal [salt-influenced samples]:** Dissolved salts were removed using pre-rinsed cellulose membrane visking dialysis tubing. Sediment samples were placed and sealed into the tubing and continuously rinsed with RO water until conductivity  $<200 \mu\text{S}\cdot\text{cm}^{-1}$  was achieved (measured using an Orion Star A215 meter and 013005MD conductivity cell).

**Marine carbonate removal [marine trap samples only]:** Following  $H_2O_2$  treatment above, carbonate present in sediment recovered from the marine traps was digested using HCl acid (1 M active concentration) in a water bath at 80 °C for 3 to 4 h until the reaction ceased. Samples were then repeatedly rinsed ( $\geq 3$  times) and decanted with RO water ( $\geq 1 \text{ L}$ ) to flush the acid (Vaasma, 2008; Fisher et al., 2017). Samples were left to settle for at least 24 h prior to decanting a clear supernatant to ensure fine material was not lost.

Table 1

Environmental settings for the six marine sediment trap sites shown in Fig. 1.

| Environment Key                                                                      | DNK1                                                                                                                                       | OS1                                                                                                                                         | HAV1                                                                                                                                                               | ORC1                                                                                                              | GFB1                                                                                                   | CLE1                                                                                                                                                        |
|--------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Trap depth below LAT and position                                                    | 6 m, lower reef slope                                                                                                                      | 10.5 m, mid reef slope                                                                                                                      | 8 m, mid reef slope                                                                                                                                                | 15 m, open sediment field                                                                                         | 3 m, sub-tidal reef flat                                                                               | 3.5 m, open sediment field                                                                                                                                  |
| Proximity to dominant catchments or coastline                                        | <ul style="list-style-type: none"> <li>13 km → Tully River</li> <li>6 km → main coastline</li> </ul>                                       | <ul style="list-style-type: none"> <li>20 km → Herbert River</li> <li>14 km → main coastline</li> </ul>                                     | <ul style="list-style-type: none"> <li>~160 km → Burdekin River</li> <li>23 km → main coastline</li> </ul>                                                         | <ul style="list-style-type: none"> <li>~115 km → Burdekin River</li> <li>19 km → Ross River</li> </ul>            | <ul style="list-style-type: none"> <li>~115 km → Burdekin River</li> <li>13 km → Ross River</li> </ul> | <ul style="list-style-type: none"> <li>~110 km → Burdekin River</li> <li>13 km → Ross River</li> </ul>                                                      |
| Turbidity (NTU) <sup>1</sup>                                                         | Moderate                                                                                                                                   | Low                                                                                                                                         | Low                                                                                                                                                                | High                                                                                                              | Moderate                                                                                               | Very High                                                                                                                                                   |
| Exposure to swell                                                                    | Exposed to South-Easterly swell                                                                                                            | Sheltered                                                                                                                                   | Sheltered                                                                                                                                                          | Exposed                                                                                                           | Exposed to South-Easterly swell                                                                        | Semi-exposed                                                                                                                                                |
| Benthic current (m/s) <sup>2</sup>                                                   | Low                                                                                                                                        | High                                                                                                                                        | Moderate                                                                                                                                                           | High                                                                                                              | Moderate                                                                                               | Very high                                                                                                                                                   |
| Substrate description and image                                                      | Weak reef framework with undulating substrate. Thin dynamic mud layer embedded with coral rubble. Abundant turf algae on hard dead corals. | Weak reef framework with massive and encrusting live and dead corals, large undulating reef slope; high current low deposition environment. | Unconsolidated fine carbonates. Complex undulating morphology (intermittent grooves filled with fine sediment), high coral cover (>80% live <i>Goniopora</i> sp.). | Unconsolidated, flat, exposed outer bay mobile sediment field. Soft deep mud with poorly sorted large shell grit. | Weak reef framework. Poorly sorted terrigenous/carbonate mix. Abundant macro algae.                    | Unconsolidated, flat mid-bay site. Thick layer of mobile mud covering poorly sorted shell fragments. Often covered with moderate ephemeral sea grass cover. |
|  |                                                                                                                                            |                                                                                                                                             |                                                                                                                                                                    |                                                                                                                   |                                                                                                        |                                                                                                                                                             |

<sup>1</sup> Mean site turbidity = Low ( $<2 \text{ NTU}$ ) Moderate (2–5 NTU) High (5–15 NTU) Very High ( $>15 \text{ NTU}$ ) from Lewis et al. (2020) for the period June 2016 to March 2020 for all sites, except DNK1 (June 2016 to March 2019).

<sup>2</sup> Mean site benthic current (~60cm above substrate) using ten minute average data discontinuous record for the period June 2016 to March 2020 for all sites, except DNK1 (June 2016 to March 2019) from Lewis et al. (2020). Categories = Low ( $<0.04 \text{ m/s}$ ) Moderate (0.04–0.8 m/s) High (0.8–0.12 m/s) Very High ( $>0.12 \text{ m/s}$ )

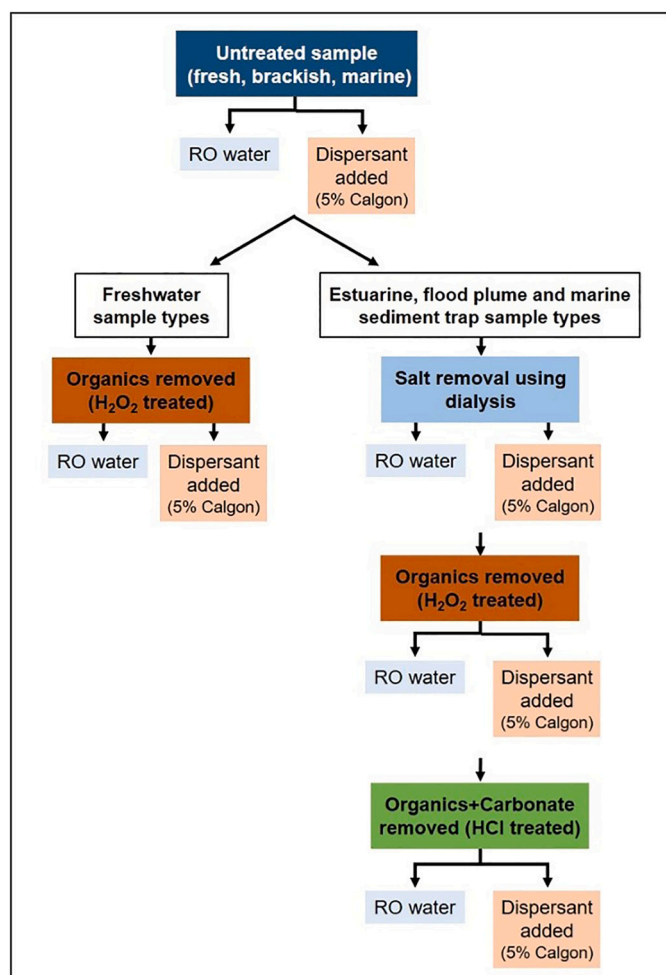


Fig. 2. Flow chart illustrating the pre-treatment steps undertaken in this study prior to grain-size analysis for end-of-river freshwater sample types (left) and estuarine, flood plume and marine sediment traps (right).

## 2.4. Sample analysis

### 2.4.1. Sediment grain-size analysis

Grain-size distributions were determined using a Mastersizer 3000 (Malvern Panalytical) laser diffraction analyser with a lens range of 0.01–3500  $\mu\text{m}$  following the general ISO 13320:2020(E) guidelines (International Organisation for Standardisation, 2020). The Mie diffraction theory was applied, which is most applicable to measuring sediments <50  $\mu\text{m}$  in diameter, typical of our sample types (Sperazza et al., 2004; Özer et al., 2010). A refractive index of 1.52 was used and all samples were ultrasonically dispersed (at 15% power) for 30 s in the introduction unit prior to measurement. Each sample was measured by the Mastersizer three times, and following checks for quality assurance (e.g. data quality pass, weighted residual, signal to noise ratio) the average of these measurements was used for reporting. Samples were sieved through a coarse 1400  $\mu\text{m}$  wet mesh sieve before measurement on the Mastersizer to remove large carbonate (shell or coral) fragments in marine samples and leaf/woody debris in freshwater samples. No residual sediment was captured by this coarse sieve from any of our samples.

A small quantity (~5 mL) of the common dispersal agent, Calgon (5% solution sodium hexametaphosphate) was initially trialled and added to a subset of all sample types, following the flow chart in Fig. 2 developed from the literature.

Microscope analysis (see Section 2.4.2 below) established the presence of long cylindrical or large circular diatoms in flood plume sediment samples. These diatoms were removed by repeatedly sieving

samples through 38  $\mu\text{m}$  sieves to progressively remove these rod-shaped particles before they were re-analysed for mineral grain size on the Mastersizer. In these cases, captured sieve material were carefully checked to ensure no mineral grains were captured, with further confirmation through viewing sub-samples of this material under microscope. The Mastersizer determined that most mineral grains were <20  $\mu\text{m}$  in diameter.

Results are presented as grain-size distribution plots (% distribution by volume), and classified underneath each plot as one of four size classes based on the Udden-Wentworth sediment grain size scale (Leeder, 1982): (1) clay (<3.9  $\mu\text{m}$ ); (2) very fine and fine silt (3.9–15.6  $\mu\text{m}$ ; hereafter referred to as fine silt); (3) coarse silt (15.6–63  $\mu\text{m}$ ); and (4) sand (63–2000  $\mu\text{m}$ ). Grain-size results are also provided in tabular form as D-values (D10, D50, D90) representing the median particle diameter, in  $\mu\text{m}$  (i.e. D50 = 50% by volume of the particles is smaller than this diameter), and the particle diameter at the 10th and 90th percentiles (AS 4863.1-2000). The proportion of sediment grain size for each measured sample representing the ecologically relevant <20  $\mu\text{m}$  fraction is also reported (see Bainbridge et al., 2018). This <20  $\mu\text{m}$  fraction is often referred to as 'fine sediment' within current Great Barrier Reef science and policy documents (e.g. Brodie et al., 2017; McCloskey et al., 2021a).

### 2.4.2. Microscope analysis

Light microscopy was used to visually assess the effect of each pre-treatment on grain size, flocculation and separation in each sample. A small aliquot (i.e. 1–2 pipette drops) of sub-sample collected at each pre-treatment step was placed on a microscope slide (diluted with RO if required) for visual comparison of a sample across each pre-treatment step undertaken. The ToupView software program (Touptex photonics) was used to capture these images.

### 2.4.3. Loss-on-ignition (LOI) for organic matter and carbonate content

Sample organic matter content was determined using Standard Method 2540E (APHA, 2012). Briefly, a glass fibre filter paper (flood plume samples) or crucible (sediment trap samples) containing a pre-weighed quantity of each sample (dried at 105 °C to remove moisture) was ignited in a Carbolite (AAF1100) ashing furnace at 550 °C for 4 to 5 h, before being reweighed. The weight lost during ignition represents the total volatile solids, an approximation of the organic matter content of each sample (weight % LOI, 550 °C).

Marine sediment trap carbonate content (weight % LOI, 950 °C) was determined through an additional igniting step to the process above, as described by Heiri et al. (2001). Briefly, following LOI at 550 °C, sample crucibles were returned to the ashing furnace and heated at 950 °C for at least 6 h, with the weight lost on ignition representing carbon dioxide release from calcium carbonate. The ratio between CO<sub>2</sub> and CaCO<sub>3</sub> molecular weights can then be used to determine the percentage of CaCO<sub>3</sub> in the original sample following Eq. (1).

## 3. Results

### 3.1. Systematic protocol for sediment grain-size analysis across the catchment to reef continuum

#### 3.1.1. Freshwater samples

EoR samples collected in freshwater were uncontaminated by salt water or marine carbonates and required the least pre-measurement preparation. Measurement of the untreated (as collected) sample produces the grain-size distribution (Fig. 3b, d dark blue series) of all particulate matter carried in suspension at the EoR, including discrete mineral grains, freshwater diatoms and flocculated particles held together by organic matter (Fig. 3a and c). Flocs and freshwater diatoms were more common in the Tully River EoR samples (e.g. Fig. 3c) than

$$\%CaCO_3 = \frac{[\text{Sample weight post } 550^\circ\text{C ash} - \text{sample weight post } 950^\circ\text{C ash}]}{[\text{Dried sample weight post } 100^\circ\text{C}]} \times 2.274 \times 100 \quad (1)$$

those from the Burdekin, where they were only observed in samples collected during the rising stages of the 2017 and 2019 flood events.

Pre-treatment to remove organic matter reduced the number of larger particles. As a result the grain-size distributions of both samples shifted toward the finer size fractions (Fig. 3b, d orange series) and the D10, D50 and D90 values all decreased (Tables 2–3). The organic-rich Tully sample (18% organic content) changed the most, with the distribution curve shifting toward the finer fractions and the grain-size ranges narrowing from 1.45 to 400  $\mu\text{m}$  and 1.13 to 111  $\mu\text{m}$  in the untreated and treated samples respectively. The D10, D50 and D90 grain-size statistics all decreased by  $\geq 50\%$  (the D50 reduced from 14.6 to 6.6  $\mu\text{m}$ ; the D90 from 62 to 22  $\mu\text{m}$ ). The untreated Burdekin EoR sample's multi-modal distribution persisted post-treatment, but the proportion of sediments in the main finer-fraction peak increased as coarser sediments dominated by organics were removed by the treatment procedure. Overall the

distribution shifted toward the finer fractions; the untreated D50 of 7.4  $\mu\text{m}$  changed to 5.6  $\mu\text{m}$  after treatment (Fig. 3b).

Calgon addition did not improve measurement precision or alter the grain-size distributions of EoR samples (e.g. Supp. Fig. 2a); the relative standard deviation (RSD) was generally  $<5\%$ , especially for the D10 and D50 statistics. Accordingly, EoR samples were not pre-treated with Calgon.

### 3.1.2. Estuarine and flood plume samples

Measurement reproducibility for the untreated (salt-present) estuarine and flood plume samples was generally poor (Supp. Fig. 2d). Salt (ionic) interference with the Mastersizer's background or measurement readings, and the low obscuration (i.e. low particle concentrations in the instrument's measurement cell and poor signal-to-noise ratio at the detector) typical of flood plume samples may explain this instability. Additional sonication during measurement or the addition of Calgon (e.

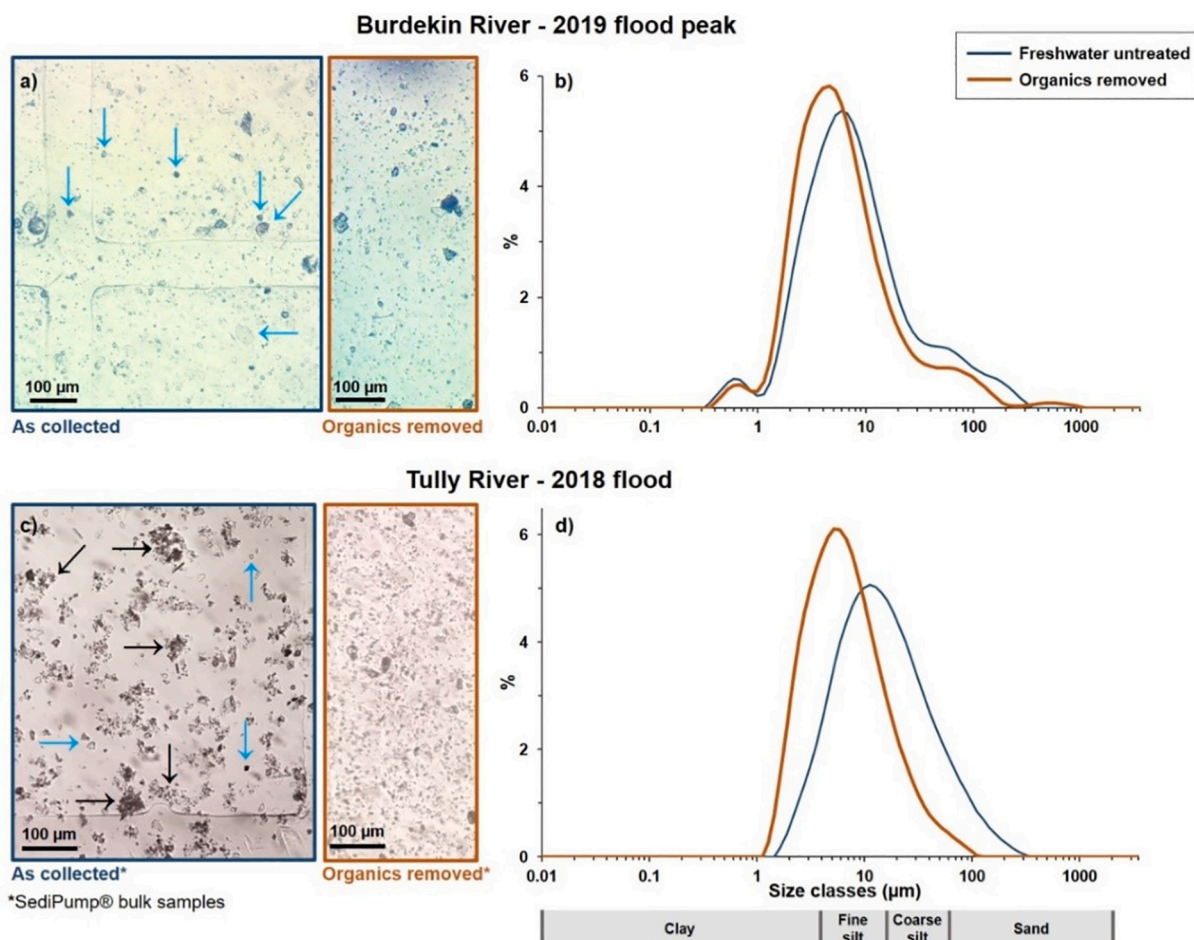


**Fig. 3.** Micro-imagery of Burdekin (a) and Tully (c) end-of-river (freshwater) samples collected during respective flood peaks (dark blue border), highlighting the presence of discrete clay, silt and sand-sized mineral grains in both samples (blue arrows), as well as small flocs (black arrows) present in the Tully River sample. The right-hand (orange border) image in each set is the sample post treatment for organic-removal, and shows the removal of binding floc organic matter in the Tully sample, with fine-grained mineral particles now separated. Note the bulk SediPump® sample has been used for this Tully EoR sample analysis, compared to a standard 'grab sample' captured at the Burdekin EoR, with an approximate 25-fold higher TSS concentration ( $470 \text{ mg L}^{-1}$  compared to  $17 \text{ mg L}^{-1}$  for the Tully EoR). Grain-size distributions for the untreated and organic-removed sub-samples for the Burdekin (b) and Tully (d) EoR samples are also displayed. Mastersizer analysis ranges from 0.01 to 2000  $\mu\text{m}$  (log scale), and represents each distribution as percent volume. A fine, colloidal fraction ( $<1 \mu\text{m}$ ) is present in the Burdekin multi-modal distribution (b). A vertical scale below the plot represents the proportion of grain sizes within the clay, fine silt, coarse silt and sand size fractions. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

**Table 2**

Grain-size results for sediment samples collected along the Burdekin River flood plume salinity gradient (6–15/02/19) during a major discharge event in February 2019. Grain-size results including the proportion of sample sediments <20 µm and the full grain-size distribution represented by the D10, D50 and D90 (standard deviation in brackets) are presented for the various pre-treatment options for each site including: as collected (i.e. untreated) and salt-, organic- and carbonate removed sub-samples. An additional measurement run for the plume samples that were repeatedly sieved through 38 µm mesh is also included. The set of final results used for each sample (i.e. best treatment option) is highlighted.

| Sample location from catchment to reef |                                                         | % organic | Treatment                                        | <20 µm % | Grain size (µm)    |                     |                 |
|----------------------------------------|---------------------------------------------------------|-----------|--------------------------------------------------|----------|--------------------|---------------------|-----------------|
|                                        |                                                         |           |                                                  |          | D10                | D50                 | D90             |
| <b>Freshwater</b>                      | End-of-river                                            | 12%       | <i>As collected</i>                              | 81       | 2.4 (±0.01)        | 7.4 (±0.05)         | 43 (±5)         |
|                                        |                                                         |           | <b>Organics removed</b>                          | 87       | <b>2.1 (±0.01)</b> | <b>5.6 (±0.09)</b>  | <b>29 (±5)</b>  |
| <b>Plume – turbid primary waters</b>   | Estuarine mixing zone <1 ppt                            | 15%       | <i>Salt removed</i>                              | 80       | 2.3 (±0.01)        | 7.7 (±0.02)         | 36 (±1)         |
|                                        |                                                         |           | <b>Salt + Organics removed</b>                   | 89       | <b>2.2 (±0.00)</b> | <b>6.1 (±0.02)</b>  | <b>21 (±0)</b>  |
| <b>Plume – secondary waters</b>        | Northern plume – 21 ppt surface waters @ ORC1 trap site | ~19%      | <i>Salt removed</i>                              | 50       | 4.6 (±0.03)        | 19.9 (±0.35)        | 142 (±8)        |
|                                        |                                                         |           | <b>Salt-Organics removed</b>                     | 55       | <b>2.6 (±0.01)</b> | <b>15.9 (±0.27)</b> | <b>101 (±2)</b> |
|                                        |                                                         |           | <b>Salt + Organics removed + &lt;38µm sieved</b> | 83       | <b>1.4 (±0.01)</b> | <b>6.6 (±0.14)</b>  | <b>34 (±3)</b>  |
| <b>Plume – secondary waters</b>        | Northern plume – 28 ppt surface waters @ HAV1 trap site | ~50%      | <i>Salt removed</i>                              | 41       | 6.5 (±0.05)        | 26 (±0.12)          | 123 (±3)        |
|                                        |                                                         |           | <b>Salt + Organics removed</b>                   | 44       | <b>4.3 (±0.01)</b> | <b>25 (±0.12)</b>   | <b>97 (±0)</b>  |
|                                        |                                                         |           | <b>Salt + Organics removed + &lt;38µm sieved</b> | 68       | <b>2.4 (±0.01)</b> | <b>11.5 (±0.06)</b> | <b>63 (±1)</b>  |
| <b>Plume – secondary waters</b>        | Mid-shelf – 28 ppt (Old Reef)                           | ~50%      | <i>Salt-Organics-Carbonate removed</i>           | 50       | 3.6 (±0.03)        | 19.9 (±0.36)        | 82 (±2)         |
|                                        |                                                         |           | <i>Salt removed</i>                              | 59       | 4.9 (±0.04)        | 15.3 (±0.25)        | 84 (±2)         |
|                                        |                                                         |           | <b>Salt + Organics removed</b>                   | 58       | <b>1.3 (±0.06)</b> | <b>13.0 (±0.40)</b> | <b>81 (±3)</b>  |
|                                        |                                                         |           | <b>Salt + Organics removed + &lt;38µm sieved</b> | 79       | <b>0.7 (±0.00)</b> | <b>6.6 (±0.11)</b>  | <b>31 (±1)</b>  |

**Table 3**

Grain size-results for sediment samples collected along the Tully River flood plume salinity gradient (9–14/02/18) during a major discharge event in February 2018. Grain-size results including the proportion of sample <20 µm and the full grain-size distribution represented by the D10, D50 and D90 (standard deviation in brackets) are presented for the various pre-treatment options for each site including: as collected (i.e. untreated) and salt- and organic removed sub-samples. The set of final results used for each sample (i.e. best treatment option) is highlighted.

| Sample location from catchment to reef    |                                                      | % organic | Treatment                        | <20 µm % | Grain size (µm)    |                     |                |
|-------------------------------------------|------------------------------------------------------|-----------|----------------------------------|----------|--------------------|---------------------|----------------|
|                                           |                                                      |           |                                  |          | D10                | D50                 | D90            |
| <b>Freshwater</b>                         | Freshwater estuary – 0.05 ppt                        | 18%       | <i>As collected – freshwater</i> | 62       | 4.6 (±0.01)        | 14.6 (±0.10)        | 62 (±2)        |
|                                           |                                                      |           | <b>Organics removed</b>          | 88       | <b>2.6 (±0.01)</b> | <b>6.6 (±0.01)</b>  | <b>22 (±0)</b> |
| <b>Plume – primary waters</b>             | Estuarine mixing zone – 5 ppt                        | 20%       | <i>As collected – 5 ppt</i>      | 68       | 4.4 (±0.01)        | 13.6 (±0.06)        | 40 (±0)        |
|                                           |                                                      |           | <i>Salt removed</i>              | 72       | 2.9 (±1.53)        | 10.5 (±0.60)        | 39 (±1)        |
|                                           |                                                      |           | <b>Salt + Organics removed</b>   | 71       | <b>3.2 (±0.01)</b> | <b>10.4 (±0.10)</b> | <b>45 (±1)</b> |
| <b>Plume – secondary waters</b>           | Midway to Dunk Is. – 22 ppt                          | 33%       | <i>Salt removed</i>              | 61       | 3.1 (±0.03)        | 13.4 (±0.20)        | 103 (±6)       |
|                                           |                                                      |           | <b>Salt + Organics removed</b>   | 91       | <b>0.7 (±0.01)</b> | <b>4.3 (±0.02)</b>  | <b>18 (±1)</b> |
| <b>Plume – secondary waters</b>           | Outer plume – 31 ppt surface waters @ DNK1 trap site | 38%       | <i>As collected – 31 ppt</i>     | 80       | 3.0 (±0.00)        | 7.9 (±0.02)         | 41 (±1)        |
|                                           |                                                      |           | <i>Salt removed</i>              | 82       | 2.6 (±0.01)        | 7.2 (±0.03)         | 35 (±0)        |
|                                           |                                                      |           | <b>Salt + Organics removed</b>   | 84       | <b>0.7 (±0.00)</b> | <b>5.4 (±0.01)</b>  | <b>29 (±0)</b> |
| <b>Plume – secondary waters (+5 days)</b> | Outer plume – 33 ppt surface waters @ DNK1 trap site | 40%       | <i>Salt removed</i>              | 63       | 3.6 (±0.01)        | 12.3 (±0.10)        | 111 (±2)       |
|                                           |                                                      |           | <b>Salt + Organics removed</b>   | 95       | <b>1.5 (±0.02)</b> | <b>5.2 (±0.04)</b>  | <b>16 (±1)</b> |
| <b>Plume – tertiary waters (+5 days)</b>  | Mid-shelf – 33 ppt (Ellison Reef)                    | 29%       | <i>Salt removed</i>              | 41       | 6.2 (±0.05)        | 26 (±0.15)          | 163 (±12)      |
|                                           |                                                      |           | <b>Salt + Organics removed</b>   | 79       | <b>3.1 (±0.04)</b> | <b>8.0 (±0.09)</b>  | <b>43 (±3)</b> |

g. Supp. Fig. 2b) had negligible impact. Many of the untreated measurement results did not achieve the precisions required by ISO 13320:2020(E) (International Organisation for Standardisation, 2020) and were thus discarded. However, the precisions were met for several samples from the Tully case study which are compared below with other treated measurements.

Salts were removed using dialysis tubing (see Section 2.3) and all plume samples were lightly sonicated (15% for 30 s) prior to

measurement. Calgon addition did not improve measurement precision or alter the grain-size distribution for flood plume samples (e.g. Supp. Fig. 2b) and was therefore not included in the processing of flood plume samples. The salt-removed grain-size distributions of flood plume sediments collected from the Burdekin and Tully transects are presented in Figs. 4–6 (light blue series). Replicate measurements on salt-removed samples were generally more stable than for untreated samples. However, the high particulate organic matter content (15–50%) of flood

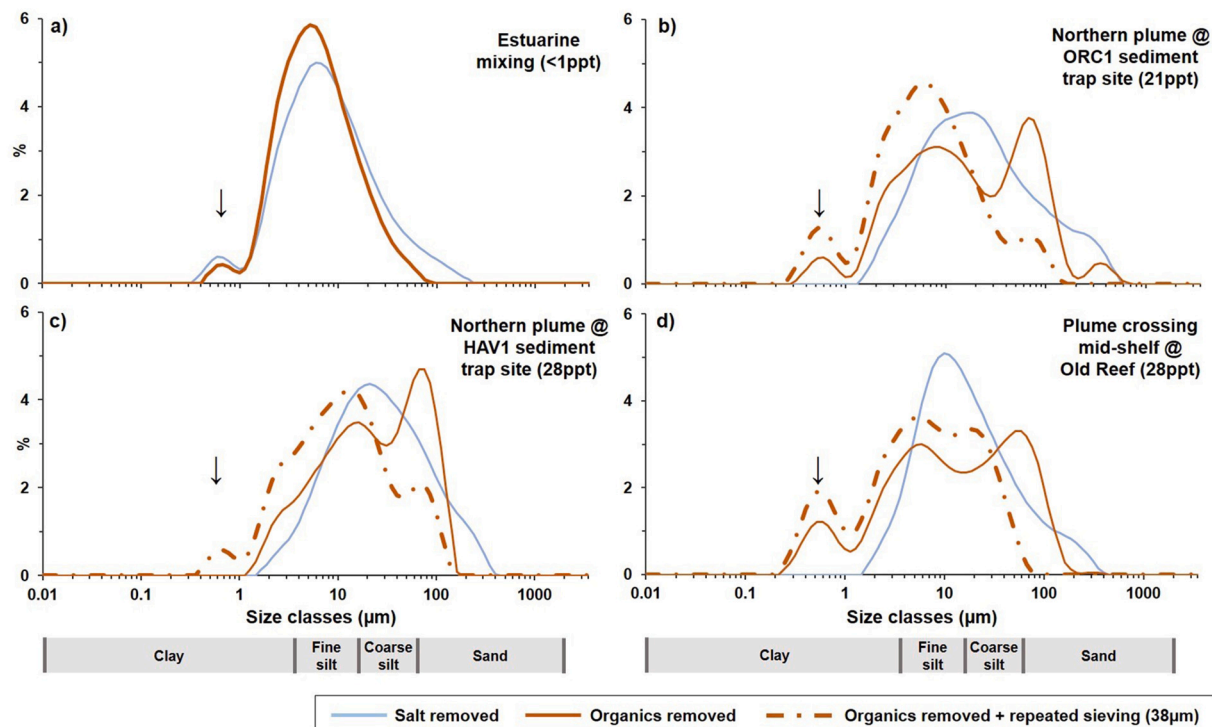


**Fig. 4.** Grain-size distributions of samples collected from the Burdekin River flood plume event, 11–15th February 2019 including: (a) an inner plume sample from the estuarine mixing zone (<1 ppt); northern plume transect collected from surface waters above the Orchard Rocks (b) and Havannah Is. (c) marine sediment trap sites; and (d) the eastern edge of the plume crossing the GBR Mid-shelf (Old Reef). Grain-size distributions analysed on the salt- and organics-removed and additional sieved (38  $\mu\text{m}$ ) sub-samples are presented for each site. Arrow on each graph highlights the colloidal fraction present along the plume gradient. (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article.)

plume samples often bound primary particles together, producing a peaked unimodal distribution (see Figs. 4b–d and 6d). These samples generally transformed into multi-modal distributions (refer to orange series in the plots) when treated to remove organics, reducing the D90 for each estuarine and flood plume sample across both case study sets (Tables 2 and 3). For example, removal of the organic content in the Burdekin estuarine sample (<1 ppt) increased the proportion of particles falling within the main distribution peak (Fig. 4a), and reduced the D90 value from 36 to 21  $\mu\text{m}$  (Table 2).

The remaining Burdekin flood plume samples were collected further offshore in secondary waters characterised by well-mixed salinities (21–28 ppt), low SPM concentrations (<5  $\text{mg L}^{-1}$ ), and organic matter comprising ~20–50% of measured sample particulate matter by mass (Table 2; Petus et al., 2019). Accurately measuring grain size in these samples is particularly challenging and time-intensive, requiring additional treatments beyond standard organic removal. Light microscopy confirmed siliceous diatoms (long cylindrical and disc shaped cells shown in Fig. 5b–c) persisted in many samples after  $\text{H}_2\text{O}_2$  treatment. Siliceous diatoms can be abundant in samples collected during flood plume associated phytoplankton blooms (see sample depicted in Fig. 5a). Where this occurs the diatoms comprise a coarser grain-size peak around 65–90  $\mu\text{m}$  within the total sample grain-size distribution (Fig. 5a). Similar peaks of coarse particle fractions were also measured in the other secondary water samples (Fig. 4b and d orange series). Repetitive sieving of these samples was required for diatom removal, and light microscopy confirmed they were mostly removed prior to re-measurement (e.g. Fig. 5d). Application of this series of pre-treatments greatly refined the grain-size distributions produced for these samples so that they better represent the suspended sediments delivered from the catchments. The peak associated with coarser particles was reduced (orange dashed series in Fig. 4b–d) and the D90 grain-size value declined by >50% in all three samples (Table 2).

Within the Tully transect samples, the grain-size distributions for two

untreated samples are included (Fig. 6a and c). These samples represent two endmembers of the salinity gradient (5 ppt within the estuary and 31 ppt in the outer secondary waters of the plume) and were more stable than the majority of untreated sample measurements. The grain-size distribution curves are similar for the two measurement runs (i.e. untreated versus salt removed) for each sample (see Fig. 6a, c dark blue series (untreated) and light blue series (salt-removed)), with a slightly finer D90 value (35 compared to 41  $\mu\text{m}$ ) in the salt-removed measurement of the outer plume sample (Table 3).

Large flocs binding mineral particles and phytoplankton were again observed using light microscopy in the Tully flood plume samples collected in secondary waters (with salinity >22 ppt and organic contents of 29–40%). Treatment to remove organics shifted the main distribution peak to finer grain sizes, and for two samples, created bi-modal distributions with a smaller colloidal peak (Fig. 6b–d). Similar to the Burdekin secondary water samples, the D90 reduced considerably in three of these secondary water samples, from >100  $\mu\text{m}$  to <50  $\mu\text{m}$  (Table 3). However, unlike the Burdekin samples, the Tully samples were collected a year earlier and were not treated to remove the diatoms. These results therefore include coarse, non-mineral particles (such as diatoms) that would normally be removed, and the proportion of finer mineral grains represented would be larger if pre-treatment to remove diatoms had been applied.

HCl-digestion treatment to remove marine carbonates was also trialled on one of the Burdekin secondary water samples (surface waters at HAV1 site) and two Tully plume samples as a potential alternative method to the repeated sieving and microscopy. Light microscopy revealed diatoms were still present after HCl-digestion, and thus this treatment was not applied further to plume samples. Fig. 5a provides an example of this carbonate-removed distribution (green) curve, which is similar to the organic-removed (orange) measurement. The D10, D50 and D90 results are provided in Table 2.

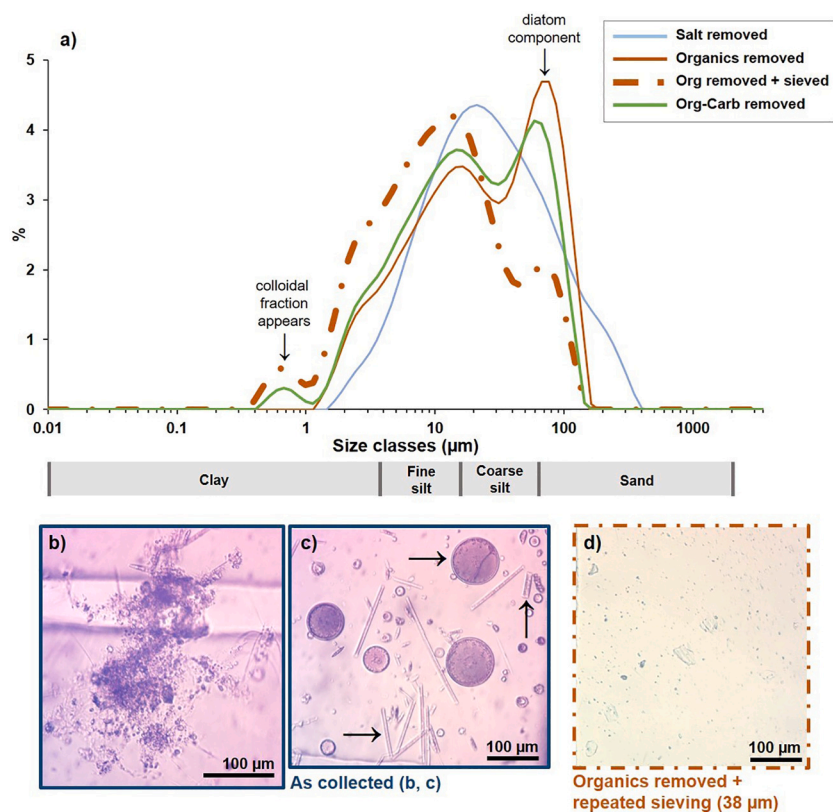


**Fig. 5.** Grain-size distributions (a) of northern Burdekin flood plume surface water SPM (12th Feb 2019) collected above the Havannah Island marine sediment trap site following multiple pre-treatments. Associated light microscopy images show this sample: (b) and (c) as collected; (d) post organic-removal and multiple sieving (<38 μm) to further remove siliceous diatoms. This plume sample captured a phytoplankton bloom providing a good case study for removal of both organic matter and siliceous diatoms. Image (b) highlights a large floc comprising organic matter binding diatoms and small discrete mineral particles. Diatoms dominate SPM in this water (c), with arrows highlighting disc-shaped (?*Coscinodiscus*) and long cylindrical diatom cells. Most of these diatoms have been removed through pre-treatment steps in (d), highlighting the need for careful sieving in a sub-set of these difficult diatom-rich samples. (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article.)

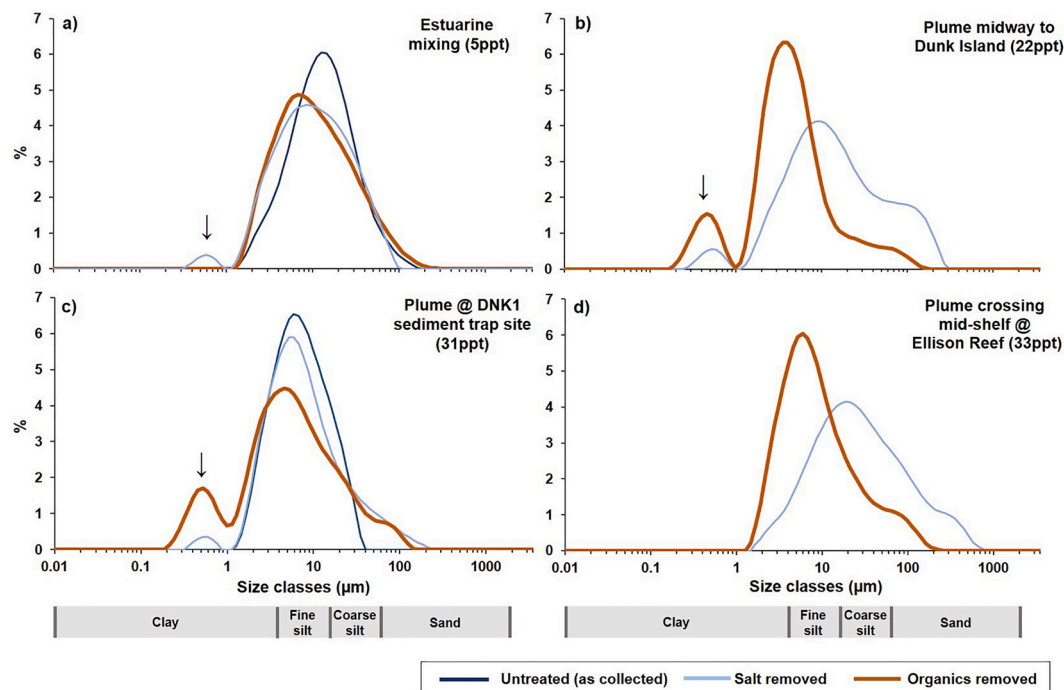


**Fig. 6.** Grain-size distributions for samples collected from the Tully River flood plume surface waters on the 9th February 2018 following EoR peak discharge including: (a) an inner plume sample from the estuarine mixing zone (5 ppt); (b) midway to Dunk Island (22 ppt) and (c) plume surface waters (31 ppt) above the inshore reef (Dunk Is.) marine sediment trap site. Sample (d) was collected 5 days later (14th February) as the plume crossed the mid-shelf at Ellison Reef (33 ppt). Grain-size distributions analysed on the salt- and organic removed sub-samples are presented for all sites. Additional as collected (i.e. untreated) distributions are also presented for (a) and (c). (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article.)

### 3.1.3. Marine sediment trap samples

The grain-size distributions determined for untreated and salt-removed samples collected from the six marine sediment traps were similar (Fig. 7a–e dark blue and light blue series), but differed most in the coarser fractions as demonstrated by divergent D90 values and large standard deviations within each treatment set (Table 4). The coarser grained CLE1 sample was an exception, with more variable bi-modal distributions between the untreated and salt-removed measurements (Fig. 7f). For each of these treatment types Calgon addition did not improve measurement reproducibility (e.g. Supp. Fig. 2c). However, for samples dominated by coarser grain sizes (i.e. CLE1), the reproducibility of distribution curves was improved by light sonication (15–20%), especially at the coarse end of the curve (i.e. D90).

Pre-treatment to remove organic matter binding primary particles in the marine trap samples (i.e. the flocs visible in the untreated DNK1 sample images Fig. 8a–b) shifted all grain-size distributions toward the finer size fractions (Fig. 7a–f orange series), and lowered the D10, D50 and D90 values for all sites except ORC1 (Table 4). The combined pre-

treatment  $H_2O_2$  and HCl steps in Fig. 2 to remove both organics and marine carbonate (e.g. Fig. 8c) generally increased the height of the main distribution peaks (Fig. 7a–f green series) and further reduced the D90 values in all samples (Table 4). Combined with Calgon-drop additions and ultrasonic dispersal (at 15%) during measurement, this extensive pre-treatment improved the reproducibility of these sub-samples with improved standard deviations across the D10, D50 and D90 values compared to the less treated runs (Table 4). The grain-size reduction across treatments was greatest for DNK1, OS1, HAV1 and GFB1, where D90 values of 95–158  $\mu m$  in the salt-removed run were reduced to 24–46  $\mu m$  post-treatment (Table 4). As a result the proportion of total grain-size <20  $\mu m$  for each of these four samples increased substantially to 71–86%. In contrast, for the two open sediment field site samples (ORC1, CLE1) the proportion of grains <20  $\mu m$  changed little across the different pre-treatments. The coarser grained CLE1 sample showed the most variation across pre-treatment grain-size distributions (Fig. 7f), and had the largest decrease in D90 value from the salt-removed to full treatment run (163 to 71  $\mu m$ , respectively).

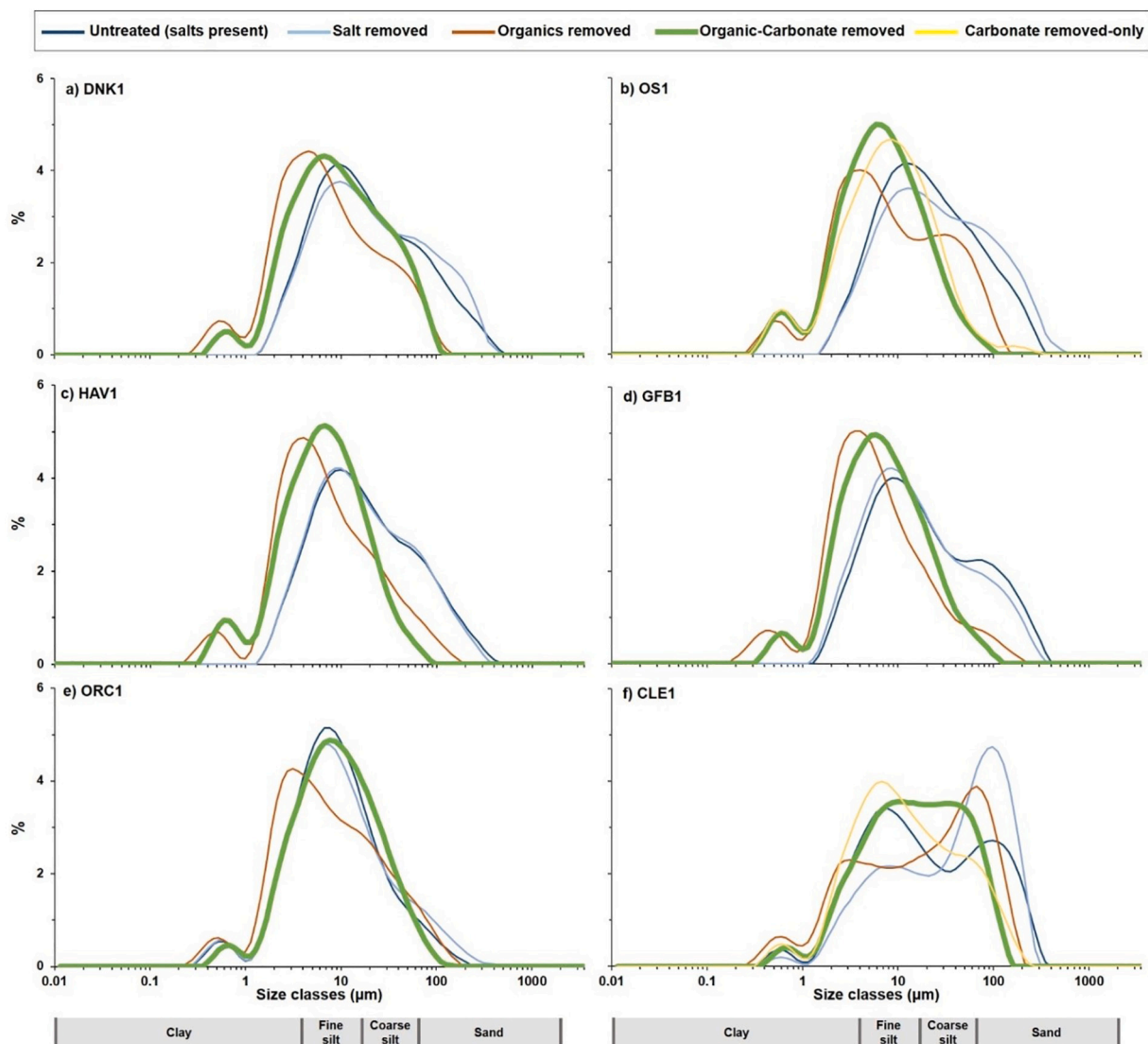


**Fig. 7.** Grain-size distributions of six samples collected from marine deployed SediSamplers® including: (a) Dunk Is. (DNK1) trap capturing the Tully River offshore gradient (Dec 2017 to Feb 2018); and five sites within Cleveland and Halifax Bays representing the longitudinal gradient from the Burdekin River mouth including (b) OS1, (c) HAV1, (d) GFB1, (e) ORC1 and (f) CLE1 collected during wet season flooding (c, e, f) and post wet season (b, d) deployments in the period December 2018 to June 2019. Grain-size distributions presented compare pre-treatments including as collected (i.e. untreated) and salt-, organic-, organics + <38  $\mu m$  sieving, organic + carbonate removed. For sites OS1 (b) and CLE1 (f) an additional distribution is presented for carbonate removal-only pre-treatment. (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article.)

**Table 4**

Sediment grain-size results for various treatments for six marine sediment trap sites during various wet and dry season deployments. The site environment, and proportions of organic and carbonate content for each sample are also provided. Grain-size results including the proportion of sample <20 µm and the full grain-size distribution represented by the D10, D50 and D90 (standard deviation in brackets) are presented for the various pre-treatment options for each site including: as collected (i.e. untreated) and salt-, organic- and carbonate removed sub-samples. The set of final results used for each sample (i.e. treatment option) is highlighted.

| Marine sediment trap site                                                                        | % organic | % carbonate | Environment                                                      | Treatment                                  | <20 µm % | Grain size (µm) |              |           |
|--------------------------------------------------------------------------------------------------|-----------|-------------|------------------------------------------------------------------|--------------------------------------------|----------|-----------------|--------------|-----------|
|                                                                                                  |           |             |                                                                  |                                            |          | D10             | D50          | D90       |
| <b>DNK1</b><br>Wet season deployment – 2018<br>Tully flood plume<br>(Dec'17 – Feb'18)            | 7.1%      | 23%         | <b>Lower reef-slope</b><br>Exposed in SE swell<br>Low current    | <i>As collected in marine trap</i>         | 55       | 4.3 (±0.01)     | 16.9 (±0.06) | 117 (±2)  |
|                                                                                                  |           |             |                                                                  | <i>Salt removed</i>                        | 57       | 4.1 (±0.12)     | 15.8 (±0.80) | 95 (±6)   |
|                                                                                                  |           |             | Weakly consolidated reef framework                               | <b>Salt + Organics removed</b>             | 77       | 1.9 (±0.01)     | 6.7 (±0.03)  | 43 (±1)   |
|                                                                                                  |           |             |                                                                  | <b>Salt + Organics + Carbonate removed</b> | 71       | 2.5 (±0.05)     | 9.7 (±0.02)  | 46 (±1)   |
|                                                                                                  |           |             |                                                                  |                                            |          |                 |              |           |
| <b>OS1</b><br>Post wet season deployment<br>(April-June'19)                                      | 7.1%      | 35%         | <b>Mid reef-slope</b><br>Sheltered<br>High current               | <i>As collected in marine trap</i>         | 50       | 5.1 (±0.02)     | 19.9 (±0.1)  | 112 (±1)  |
|                                                                                                  |           |             |                                                                  | <i>Salt removed</i>                        | 44       | 5.3 (±0.05)     | 25 (±0.85)   | 158 (±18) |
|                                                                                                  |           |             | Weakly consolidated reef framework                               | <b>Organics removed</b>                    | 70       | 1.9 (±0.01)     | 7.8 (±0.05)  | 54 (±1)   |
|                                                                                                  |           |             |                                                                  | <b>Salt + Organics + Carbonate removed</b> | 85       | 1.9 (±0.07)     | 7.0 (±0.06)  | 26 (±0)   |
|                                                                                                  |           |             |                                                                  | <i>Salt + Carbonate removed only (HCl)</i> | 79       | 1.9 (±0.23)     | 8.4 (±1.0)   | 32 (±8)   |
| <b>HAV1</b><br>Wet season deployment – 2019<br>Burdekin flood plume<br>(Jan-April '19)           | 7.2%      | 24%         | <b>Mid reef-slope</b><br>Sheltered<br>Low current                | <i>As collected in marine trap</i>         | 56       | 4.2 (±0.02)     | 16.3 (±0.17) | 102 (±3)  |
|                                                                                                  |           |             |                                                                  | <i>Salt removed</i>                        | 57       | 4.1 (±0.02)     | 15.8 (±0.23) | 95 (±3)   |
|                                                                                                  |           |             | Unconsolidated fine carbonates                                   | <b>Salt + Organics removed</b>             | 80       | 2.0 (±0.02)     | 6.3 (±0.05)  | 38 (±1)   |
|                                                                                                  |           |             |                                                                  | <b>Salt + Organics + Carbonate removed</b> | 86       | 1.9 (±0.03)     | 7.0 (±0.06)  | 24 (±0)   |
| <b>GFB1</b><br>Post-wet season deployment<br>(March-June'19)                                     | 8%        | 15%         | <b>Sub-tidal reef flat</b><br>Exposed in SE swell<br>Low current | <i>As collected in marine trap</i>         | 55       | 4.1 (±0.02)     | 16.6 (±0.32) | 130 (±8)  |
|                                                                                                  |           |             |                                                                  | <i>Salt removed</i>                        | 60       | 3.7 (±0.06)     | 14.1 (±0.53) | 105 (±7)  |
|                                                                                                  |           |             | Weakly consolidated reef framework                               | <b>Salt + Organics removed</b>             | 84       | 1.7 (±0.01)     | 5.4 (±0.03)  | 31 (±1)   |
|                                                                                                  |           |             |                                                                  | <b>Salt + Organics + Carbonate removed</b> | 83       | 2.2 (±0.04)     | 7.1 (±0.09)  | 28 (±1)   |
| <b>ORC1</b><br>Wet season deployment – 2019<br>Burdekin flood plume<br>(Dec'18-Mar'19)           | 7.5%      | 7%          | <b>Open sediment field</b><br>Exposed<br>Low to moderate current | <i>As collected in marine trap</i>         | 76       | 2.6 (±0.09)     | 9.2 (±0.28)  | 43 (±3)   |
|                                                                                                  |           |             |                                                                  | <i>Salt removed</i>                        | 71       | 2.6 (±0.08)     | 9.8 (±0.34)  | 71 (±5)   |
|                                                                                                  |           |             | Unconsolidated, flat, exposed outer-bay mobile sediment field    | <b>Salt + Organics removed</b>             | 76       | 1.9 (±0.02)     | 7.0 (±0.03)  | 45 (±1)   |
|                                                                                                  |           |             |                                                                  | <b>Salt + Organics + Carbonate removed</b> | 76       | 2.7 (±0.05)     | 9.4 (±0.11)  | 35 (±0)   |
| <b>CLE1</b><br>Wet season deployment – 2019<br>flood major resuspension event<br>(Dec'18-Mar'19) | 4.4%      | 8%          | <b>Open sediment field</b><br>Semi-exposed<br>Very high current  | <i>As collected in marine trap</i>         | 55       | 3.4 (±0.01)     | 16.4 (±0.10) | 134 (±2)  |
|                                                                                                  |           |             |                                                                  | <i>Salt removed</i>                        | 51       | 2.6 (±0.10)     | 19.3 (±0.84) | 163 (±9)  |
|                                                                                                  |           |             | Unconsolidated, flat mid-bay sediment field                      | <b>Salt + Organics removed</b>             | 49       | 2.1 (±0.04)     | 21 (±0.39)   | 102 (±3)  |
|                                                                                                  |           |             |                                                                  | <b>Salt + Organics + carbonate removed</b> | 56       | 3.2 (±0.08)     | 16.1 (±0.20) | 71 (±2)   |
|                                                                                                  |           |             |                                                                  | <i>Salt + Carbonate removed only (HCl)</i> | 64       | 2.8 (±0.06)     | 11.7 (±0.12) | 76 (±1)   |

A pre-treatment to remove carbonate only (i.e. organics remaining in the sample) was trialled with HCl acid digestion-only on the OS1 and CLE1 samples (see Fig. 7b and f yellow series). Measurements show a coarser D90 value for both samples than for the standard carbonate and organic removed treatment.

### 3.1.4. SediPump® filtration system for bulk sediment collection

The comparison of grain-size distributions from the SediPump® bulk sample and the manual point-in-time 'grab' samples concurrently collected at the three flood plume sampling sites show close agreement (Table 5). For all three samples the SediPump® sample had a slightly wider distribution than the point-in-time 'grab', confirming that this new collection method captures particles of all size ranges. For example, the grain-size range (D10 and D90) for the Old Reef site was 5.9 and 81 µm for the 'grab' sample, and 4.9 and 84 µm for the SediPump® sample. The proportion of total sample <20 µm in grain-size was also similar or slightly higher in the SediPump® sample compared to the grabs for all three sites e.g. 50% compared to 45% in the Orchard Rocks (21 ppt)

sample. The quantity of particulate material that can be captured within the SediPump® 1 µm filter compared to the 'grab' sample as collected is shown in Table 5.

### 3.2. Sediment grain size across the GBR catchment to reef continuum

#### 3.2.1. Freshwater samples

Samples from the Wet Tropics rivers contained higher organic contents (16–23%) than those from Dry Tropics streams (11–14%), mostly due to more abundant flocculated particles and freshwater diatoms. Tully EoR samples were collected at peak-of-flow in three moderate-sized discharge events, including January 2017 (peak ~8.5 m) and February and March 2018 (peaks of 8 and 9 m, respectively, measured at the Euramo gauge: 113006A). An opportunistic sample was also collected on the South Johnstone River in 2018. Pre-treatment to refine these samples to primary particles considerably increased the measured proportion of fine sediment (<20 µm) (Table 6). For example, pre-treatment to remove organics from the sample collected at the peak of

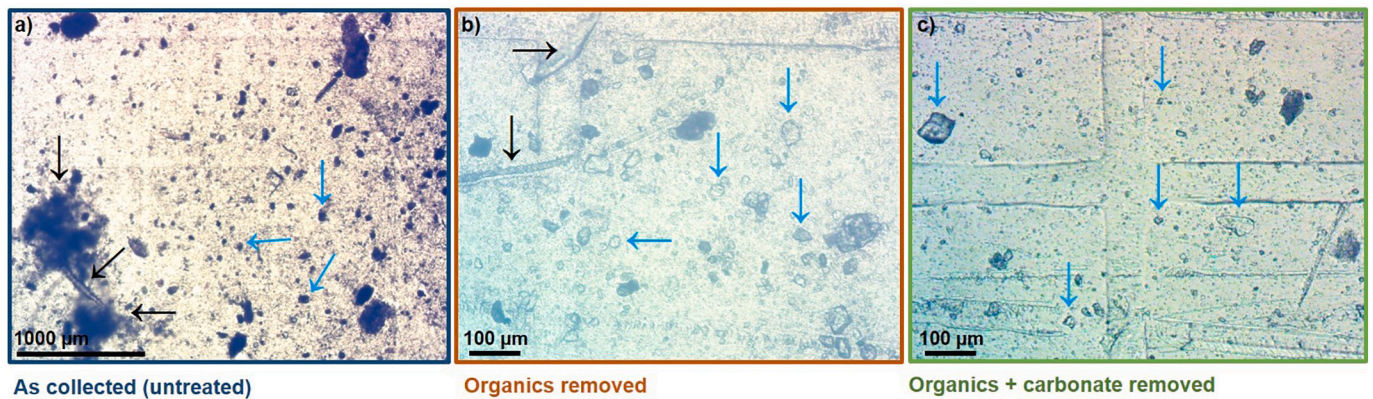


**Fig. 8.** Micro-imagery of the DNK1 sediment trap sample from the 2017–18 wet season flood period deployment (07/12/17–15/02/18): (a) as collected (untreated); and following (b) organic removal and (c) both organic-carbonate removed. A wider view of the untreated sample highlights the presence of large flocs, plankton (both black arrows) and grain particles (blue arrows), with organic matter binding particles removed in (b) and showing a magnified view (note change in scale) of discrete grains of various sizes, and the presence of some remaining plankton (black arrows). Finally, with carbonate grains and fragments removed, a large range of clay to coarse sized grains are observed (blue arrows). The cross-pattern of the counter cell is also clearly visible on (b) and (c). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

**Table 5**

Comparison of grain-size results for a SediPump® concentrated ‘bulk’ sample and the corresponding manual point-in-time ‘grab’ sample collected at three study plume samples. Sample characteristics, SPM concentration ( $\text{mg L}^{-1}$ ), organic content (%) and imagery of both the ‘grab’ sample and concentrated SediPump® sample are also included. Sites include the February 2018 Tully flood plume estuary site and two February 2019 Burdekin secondary water type flood plume samples collected at Orchard Rocks (ORC1) and Old Reef (refer to Fig. 1 and Supp. Fig. 1c). Grain-size results are from salt-removed sub-samples (i.e. organics present).

| Sample                                                     | Sample type                                                          | Sample collection method          | SPM conc. ( $\text{mg L}^{-1}$ ) | % Organic | % <20 $\mu\text{m}$ | Grain size ( $\mu\text{m}$ ) |      |     | Images of recovered sample |                |
|------------------------------------------------------------|----------------------------------------------------------------------|-----------------------------------|----------------------------------|-----------|---------------------|------------------------------|------|-----|----------------------------|----------------|
|                                                            |                                                                      |                                   |                                  |           |                     | D10                          | D50  | D90 | Grab (point in time)       | SediPump® bulk |
| Flood plume – 5 ppt in estuary surface waters              | Discrete particles, flocs (50–100 $\mu\text{m}$ ), some plant debris | Grab sample                       | 20                               | ~20       | 73                  | 3.5                          | 12.3 | 31  |                            |                |
|                                                            |                                                                      | SediPump® bulk sample (1 hr pump) | -                                | -         | 72                  | 2.9                          | 10.5 | 39  |                            |                |
| Flood plume – 21 ppt surface waters at ORC1 trap site      | High organic matter (plankton/detritus)                              | Grab sample                       | <3                               | ~19       | 45                  | 6                            | 22.8 | 135 |                            |                |
|                                                            |                                                                      | SediPump® bulk sample (3 hr pump) | -                                | -         | 50                  | 4.6                          | 19.9 | 142 |                            |                |
| Flood plume – 28 ppt surface waters at Old Reef, Mid Shelf | High organic matter (plankton/detritus)                              | Grab sample                       | <3                               | ~50       | 47                  | 5.9                          | 21.5 | 81  |                            |                |
|                                                            |                                                                      | SediPump® bulk sample (2 hr pump) | -                                | -         | 59                  | 4.9                          | 15.3 | 84  |                            |                |

the January 2017 flood increased the proportion of mineral grains <20  $\mu\text{m}$  from 44 to 77%. The treated grain-size range (bounded by the D10 and D90 statistics incorporating 80% of the sample grain sizes) for Tully sediments (2.6 to 30  $\mu\text{m}$ ) was smaller than for the South Johnstone sample (3.4 to 70  $\mu\text{m}$ ). The grain-size range for the Herbert sample collected across the discharge peak (composite from 4 to 7th February 2019) was 4.1 to 38  $\mu\text{m}$ .

The Burdekin EoR site was sampled across minor to large discharge events during the 2017, 2018 and 2019 wet seasons, capturing different flow stages and sediment sources within each year sampled (Table 6). For the fifteen samples analysed, organic content (11–14%) and the proportion of mineral grains <20  $\mu\text{m}$  (81–93%) remained relatively consistent for the treated samples (Tables 2 and 6). The largest increases in the fine sediment fraction between the untreated and treated samples

occurred in those from the March 2017 event, a drought-breaking and rapid flood associated with Tropical Cyclone Debbie. Treatment of the rising stage sample collected during this event increased the proportion of fine sediment (<20  $\mu\text{m}$ ) from 49 to 89% (Table 6). The grain-size distribution for all Burdekin samples ranged from  $1.9 \pm 0.5 \mu\text{m}$  at the D10 to  $24 \pm 7 \mu\text{m}$  at the D90 (average of annual averages in Table 6), with a slightly finer D10 measured in all 2018 event samples. The 2018 event exclusively originated from the Upper Burdekin catchment and passed through the Burdekin Falls Dam. All measured Burdekin suspended sediment samples exhibited a characteristic multi-modal distribution (e.g. Fig. 3b), with a small mini peak at  $\sim 0.5 \mu\text{m}$ , representing a colloidal fraction (<1  $\mu\text{m}$ ). A similar colloidal fraction component was also observed in suspended sediment samples from the neighbouring Haughton and Ross River EoR sites collected opportunistically for

**Table 6**

Grain-size distributions for samples collected at a number of Great Barrier Reef catchment end of river (EoR) freshwater sites collected under the broader NESP research project. Collection dates, hydrograph flow stage, total suspended solid concentrations ( $\text{mg L}^{-1}$ ) and organic content (%) are also provided. The proportion of fine sediment ( $<20 \mu\text{m}$ ) in freshwater samples analysed as collected and following treatment for organic-removal are presented, as well as the grain-size distribution on treated samples.

| GBR catchment              | Site coordinates (DD) | Date      | Hydrograph stage         | TSS ( $\text{mg L}^{-1}$ ) | Organic content | Proportion grain size $<20 \mu\text{m}$ |                  | Grain size ( $\mu\text{m}$ ) |     |     |
|----------------------------|-----------------------|-----------|--------------------------|----------------------------|-----------------|-----------------------------------------|------------------|------------------------------|-----|-----|
|                            |                       |           |                          |                            |                 | As collected                            | Organics-removed | D10                          | D50 | D90 |
| Burdekin EoR – 2017        | –19.64, 147.40        | 29/03/17  | Rising rapidly           | 748                        | 13%             | 49                                      | 89               | 1.9                          | 5.6 | 22  |
|                            |                       | 30/03/17  | Steady peak              | 1810                       | 11%             | 70                                      | 85               | 2.5                          | 7.4 | 26  |
|                            |                       | 31/03/17  | falling steadily         | 884                        | 12%             | 66                                      | 90               | 2.1                          | 6.5 | 20  |
|                            |                       | Mean      |                          |                            |                 |                                         | 88               | 2.1                          | 6.5 | 22  |
| Burdekin EoR – 2018        | –19.64, 147.40        | 03/03/18  | Rising                   | 998                        | 12%             | 77                                      | 90               | 1.8                          | 5.3 | 20  |
|                            |                       | 04/03/18  | Rising                   | 681                        | 12%             | 76                                      | 89               | 1.8                          | 4.9 | 21  |
|                            |                       | 04/03/18  | Rising                   | 644                        | 12%             | 76                                      | 82               | 1.0                          | 5.6 | 32  |
|                            |                       | 05/03/18  | Sustained peak           | 534                        | 12%             | 89                                      | 90               | 0.7                          | 4.4 | 21  |
|                            |                       | 06/03/18  | falling                  | 554                        | 13%             | 93                                      | 93               | 1.7                          | 4.4 | 15  |
|                            |                       | Mean      |                          |                            |                 |                                         | 90               | 1.4                          | 4.9 | 22  |
| Burdekin EoR – 2019        | –19.64, 147.40        | 11/01/19  | Local Bogie R source     | 820                        | 13%             | 88                                      | 92               | 2.0                          | 5.4 | 18  |
|                            |                       | 31/01/19  | Rising                   | 740                        | 14%             | 76                                      | 86               | 2.4                          | 6.7 | 25  |
|                            |                       | 05/02/19  | Rising                   | 500                        | 12%             | 89                                      | 86               | 2.0                          | 5.6 | 31  |
|                            |                       | 06/02/19  | Peak 1                   | 470                        | 12%             | 81                                      | 87               | 2.1                          | 5.6 | 28  |
|                            |                       | 07/02/19  | Rising to peak 2         | 504                        | –               | 87                                      | 92               | 2.1                          | 5.3 | 18  |
|                            |                       | 11/02/19  | Falling                  | 330                        | 12%             | 87                                      | 81               | 2.3                          | 6.7 | 40  |
|                            |                       | 14/02/19  | Falling                  | 137                        | 12%             | 75                                      | 85               | 2.4                          | 6.2 | 28  |
|                            |                       | Mean      |                          |                            |                 |                                         | 87               | 2.2                          | 5.9 | 27  |
| Haughton EoR - 2019        | –19.55, 147.11        | 06/02/19  | Falling after flood peak | 167                        | 13%             | 62                                      | 69               | 2.8                          | 11  | 48  |
| Ross EoR – 2019            | –19.28, 146.83        | 02/02/19  | Early dam release        | 170                        | 12%             | 44                                      | 67               | 2.7                          | 10  | 75  |
|                            |                       | 06/02/19  | Sustained dam release    | 79                         | 11%             | 66                                      | 63               | 1.9                          | 11  | 82  |
| Herbert EoR – 2019         | –18.63, 146.17        | 4–7/02/19 | Around peak              | –                          | –               | 50                                      | 69               | 4.1                          | 14  | 38  |
| Tully EoR – 2017           | –18.02, 146.05        | 11/01/17  | Peak                     | 39                         | 18%             | 44                                      | 77               | 3.6                          | 11  | 30  |
| Tully EoR – 2018           | –18.02, 146.05        | 09/02/18  | Around peak              | 17                         | 18%             | 62                                      | 88               | 2.6                          | 6.6 | 22  |
|                            |                       | 12/03/18  | Around peak              | 25                         | 16%             | 39                                      | 81               | 2.8                          | 8.9 | 28  |
| South Johnstone Arm – 2018 | –17.61, 145.99        | 08/02/18  | Falling after flood peak | 35                         | 23%             | 25                                      | 60               | 3.4                          | 14  | 70  |

sediment source tracing purposes. Organic contents of suspended sediment for the Haughton River (13%) and Ross River (11–12%) were similar to the Burdekin (11–14%), however fine sediments ( $<20 \mu\text{m}$ ) comprised only 63–69% of these samples (Table 6). Ross River sediments included a larger coarse component than Burdekin samples, with a D10 to D90 range of 10.5 to 75  $\mu\text{m}$ , and sand ( $>63 \mu\text{m}$ ) comprising around ~12% of all mineral grains.

### 3.2.2. Estuarine and flood plume samples

Along the 2019 Burdekin flood plume gradient, the pre-treatment refining these samples to primary particles resulted in significant increases in the measured proportion of fine sediment ( $<20 \mu\text{m}$ ), with the treated grain size ranging from 68 to 89% along this plume gradient (Table 2). The grain-size distribution of the sample collected in the seawater mixing zone ( $<1 \text{ ppt}$ ) was narrowest, with a D10 to D90 range from 2.2 to 21  $\mu\text{m}$  (Table 2, Fig. 4a). This sample lacked the smaller coarse peak (around ~100  $\mu\text{m}$ ) present in the EoR freshwater sample collected immediately upstream (Fig. 3b). The samples collected further along the salinity gradient from secondary water types had more variable and coarser grain-size distributions ranging from 0.7 to 63  $\mu\text{m}$  (Table 2, Fig. 4b–d). These traits probably reflect the presence of residual diatoms persisting after pre-treatment and sieving. This is likely the case for the phytoplankton bloom sample collected at the HAV1 site, which had the largest D90 value. Median grain size (D50) for the plume samples ranged from 6.1 to 11.5  $\mu\text{m}$ . The colloidal fraction ( $<1 \mu\text{m}$ ) characteristic of all Burdekin EoR samples was evident in each of the flood plume samples following sample pre-treatment but was not measured in the corresponding untreated salt removed samples (Fig. 4a–d black arrows).

Across the Tully transect, flood plume waters were characterised by relatively low SPM concentrations, generally  $<10 \text{ mg L}^{-1}$ , and high organic contents (20–40%) (Table 3). The plume sampled at the seawater mixing zone (5 ppt) had the highest SPM concentration (20  $\text{mg L}^{-1}$ ) and a treated grain-size range (D10 to D90) of 3.2 to 45  $\mu\text{m}$  (Table 3, Fig. 6). Treated grain-size distributions for secondary water samples collected further along the salinity gradient increased in salinity and organic content, with fine sediment ( $<20 \mu\text{m}$ ) comprising 71–95% of all samples. These results were consistent with sampling of 2017 Tully River secondary waters at Dunk Island (DNK1 trap site) and Bedarra Island, where treated samples were also dominated by the fine sediment fraction (92 and 99% respectively; Lewis et al., 2018).

### 3.2.3. Marine sediment trap samples

Across the six marine sediment trap samples, silt-sized grains (3.9–63  $\mu\text{m}$ ) dominated mineral sediment, comprising 69–78% of each sample (Fig. 7). Clay-sized grains ( $<3.9 \mu\text{m}$ ) comprised 14% in CLE1, 19% in ORC1 and 27–30% in GFB1, HAV1, DNK1 and OS1. The proportion of sand grains ( $>63 \mu\text{m}$ ) was highest for the CLE1 sample at 13%, but comprised only 1.0–2.6% in the other five samples. A colloidal fraction ( $<1 \mu\text{m}$ ) was present in all six treated samples and ranged from 2.4% (at CLE1) to 8.2% (at DNK1) of the grain size distribution across the sample set (Fig. 7a–f).

The organic contents of the six sediment trap samples were lower than established for the EoR and plume samples, comprising 7.1–8.0% of trapped material in five of the six samples (Table 4). The high energy CLE1 site was an exception, with organics comprising only ~4% of material trapped during the wet season deployment at this open sediment field site within Cleveland Bay. This sample had the widest and

coarsest mineral grain-size distribution of all six trap samples following full treatment for organic and carbonate removal, with a D10 to D90 range of 3.2 to 71  $\mu\text{m}$  (Table 4). This coarser grain-size distribution is clearly demonstrated by a unique distribution curve in Fig. 7f (green line). Pre-treatment to remove organics only also revealed a carbonate component notably coarser than observed at other sites (see Fig. 7f orange line), with a D90 value of 102  $\mu\text{m}$  (Table 4). Carbonate comprised 8% of total sediment accumulated over this deployment. The proportion (56%) of fine sediment ( $<20\ \mu\text{m}$ ) was smallest for this sample. A second open sediment field site (ORC1 situated off Magnetic Is.) displayed very different grain-size results (Fig. 7e). Unlike the CLE1 sample, pre-treatment of the ORC1 sample did not greatly reduce the untreated finer grain-size distribution, the D10 to D90 range shifting only slightly from 2.6 to 43  $\mu\text{m}$  (untreated) to 2.7 to 35  $\mu\text{m}$  (full treated) (Table 4). Fine sediments ( $<20\ \mu\text{m}$ ) comprised 76% of the ORC1 sample.

Mineral grain-size distributions were finest for the two sheltered mid reef-slope sites (OS1 and HAV1) and the sub-tidal reef flat site (GFB1), with D10 to D90 ranges of 1.9 to 26  $\mu\text{m}$ , 1.9 to 24  $\mu\text{m}$  and 2.2 to 28  $\mu\text{m}$ , respectively (Table 4). Carbonate contents were also highest for samples from the two mid reef-slope sites, comprising 35% of the post-wet season sample collected at OS1 and 24% of the wet season HAV1 sample. The northernmost trap (DNK1) was positioned on a lower leeward reef-slope at Dunk Island. The 2017–2018 wet season sample there had a grain-size range of 2.5 to 46  $\mu\text{m}$  (D10–D90; Table 4) and a carbonate content of 23%.

#### 4. Discussion

##### 4.1. A systematic protocol for measuring terrigenous sediment grain size across the catchment to reef continuum

The chemistry and biology of the water in which sediments are suspended varies from catchment to reef, and suspended sediments are influenced by a range of bio-physical processes and additions as they

move through the continuum. The measurement and interpretation of sediment grain size across river to reef gradients is therefore complex. Depending on the specific objectives of the research and where the samples are collected, particular sample pre-treatments will be required if the identification and characterisation of primary mineral grains are necessary. Here, suspended sediment samples collected from end-of-river (freshwater), flood plume (estuary) and marine habitats were used to develop a systematic protocol (Fig. 9) for the reproducible and consistent measurement of mineral grain size in marine-influenced sediment samples. Grain-size results produced using this procedure allow grain-size data derived from the analysis of marine-influenced samples to be meaningfully compared to those derived from the analysis of instream sediments. This advance improves the ability to trace mineral grains through the catchment to marine continuum, allowing better insights on sediment sources and redistribution patterns that can be strategically used for water-quality and catchment management purposes.

In freshwater environments, pre-treatment to remove the organic component eliminates the bias of flocculated particles revealing a true representation of primary particle size. This step is especially important where organic matter comprises  $>15\%$  of the total particulate content. The most complicated pre-treatment regime must be applied to samples collected from mixed salinity estuarine and flood plume settings. In addition to any organics, these samples may contain biogenic silica particles (i.e. diatom valves) that must be removed as they can distort the grain size signature of primary sediments. The removal of diatoms requires additional sieving and microscope checking (outlined in Fig. 9; see also Section 2.4.1). Chemical stripping has been used for this purpose elsewhere (Vaasma, 2008), but testing the efficacy of this approach in our setting was beyond the scope of this study. Fine sediments deposited in marine settings exposed to mixed carbonate and terrigenous inputs, such as the marine trap sites in this study, must be chemically and physically pre-treated to remove both organic and carbonate components (i.e. coral or shell fragments) if the primary mineral

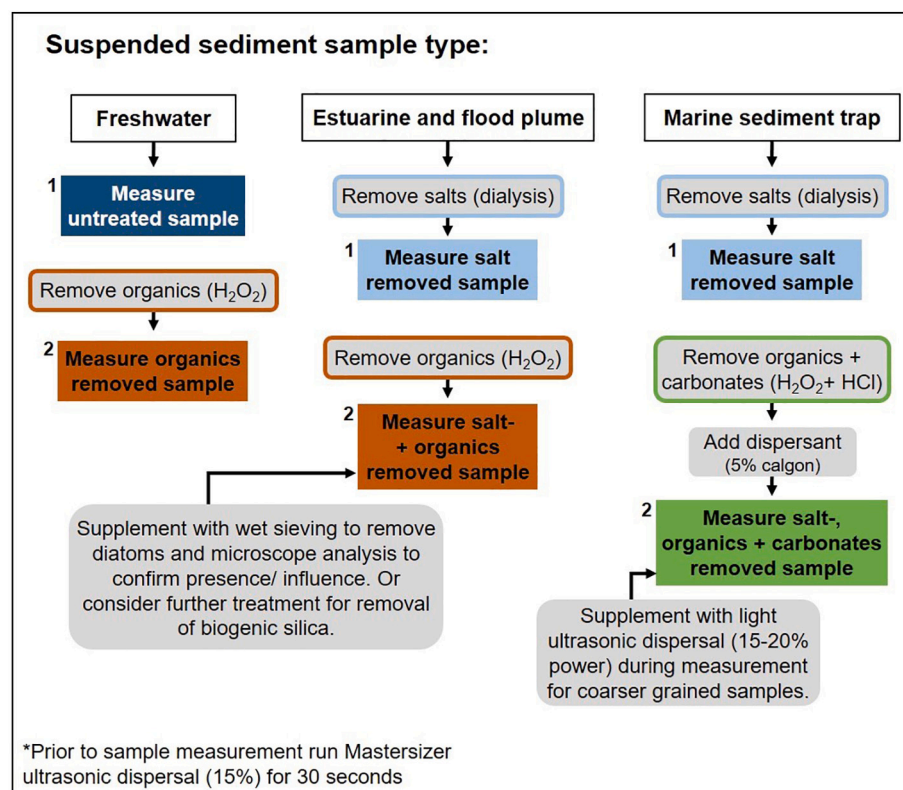


Fig. 9. Systematic protocol for sediment pre-treatment and grain-size analysis for end-of-river freshwater, estuarine and marine sediment trap samples. Each sample type has two proposed Mastersizer analysis runs, labelled 1 and 2 accordingly.

particles are the focus. Paired grain size analyses on corresponding untreated sub-samples for freshwater EoR samples and a salt-removed sub-sample for marine samples is also recommended to provide a measure of how the particles behave in transport, and to inform their potential settling capacity. This information can support modelling of sediment transport in estuarine and inshore marine waters (Manning et al., 2017), but cannot replace time-intensive and costly in situ or laboratory-based analysis of particle settling rates. Below we apply the procedures recommended above to demonstrate new insights on sediment transport and fate across the Great Barrier Reef catchment to marine continuum.

#### 4.2. New insights into fine sediment transport along the catchment to reef continuum

##### 4.2.1. Improved characterisation of GBR end-of-river fine sediment

Suspended sediments of the two case study rivers were determined to have distinct compositional differences that influence their measured grain-

size characteristics; this finding highlights the importance of adopting the recommended protocol for accurate and consistent reporting of mineral grain-size across rivers. The Wet Tropics rivers contain higher organic contents (>16% of SPM) that combined with mineral particles, form larger flocs within the freshwater reaches which distorts the grain-size distribution. Wet Tropics rivers typically carry higher dissolved inorganic nitrogen loads (Bainbridge et al., 2009; McCloskey et al., 2021b) that likely enhance instream floc formation by freshwater bacteria during downstream sediment transport (Bainbridge et al., 2018; Garzon-Garcia et al. 2018, in review). Grain-size results from rivers with high organic loadings (>16%) not pre-treated to remove this organic component will thus markedly underestimate the abundance of fine primary sediments in measured samples (e.g. 25–40% underestimation of the <20 µm fraction in Tully-Johnstone samples; Table 6). Such misrepresentation has significant implications for the assessment of water-quality risks and the evaluation of effective remediation.

In comparison, EoR suspended sediments measured in the Dry Tropics streams (Burdekin, Haughton and Ross Rivers) contained less

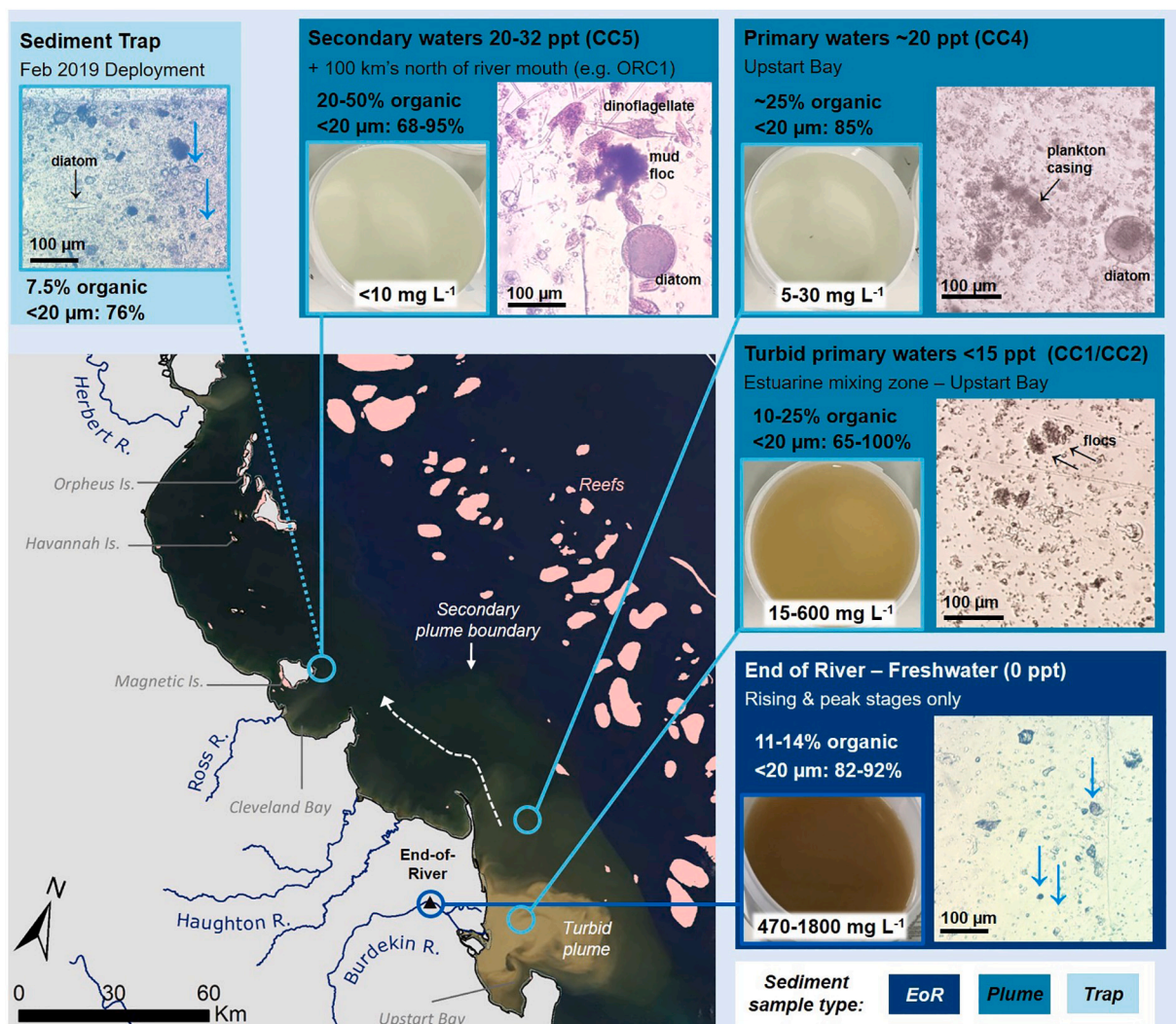


**Fig. 10.** Sediment characteristics across the Burdekin catchment to reef continuum, highlighting distinct flood plume water types and summarising data collected during river discharge events that occurred in 2017, 2018, and 2019. End of river (freshwater), flood plume water types and a sediment trap site that captured these flood plume events are all presented, anticlockwise from bottom right (refer to legend). The water type colour classification scheme from Petus et al. (2019) is used to describe plume waters from Colour Class 1 (CC1) to Colour Class 5 (CC5). Each water type box includes micro-imagery of a representative sediment sample (with features labelled and blue arrows highlighting mineral particles) and the ranges of salinity, organic content and proportion of fine sediment (<20 µm) measured within each zone. The suspended sediment concentration/suspended particulate matter range (mg L<sup>-1</sup>) is also displayed on an image of a typical “grab sample” collected at each of the end of river and flood plume water types (with increasing salinity and decreasing SPM concentration). The MODIS satellite image was captured on the 12th February and is used here to generally represent plume transitions along a salinity gradient from the river mouth. Refer to Supplement for tabulated data. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

(~11–14%) organic material (Table 6). An exception may be during drought-breaking and/or extreme, rapid floods, where EoR organic particulate loads may be elevated (e.g. Tropical Cyclone induced rapid rise on 29/03/17, Table 6). Pre-treatment for organic removal is therefore less critical here, however consistency in the pre-treatment protocol used for all samples of the same water type is recommended (Gray et al., 2010).

Burdekin EoR sediment was well characterised across three minor-large discharge events (2017–2019), with fine sediment ( $<20\ \mu\text{m}$  fraction =  $88\% \pm 4\%$ ) dominating export from this catchment. This finding is supported by earlier (2009–2011) Burdekin sampling during a wetter period (Bainbridge et al., 2012, 2014), highlighting the importance of remediation efforts within the catchment to target this fine sediment component. Despite the different catchment source contributions to each of the sampled discharge events during the 2017–2019 period, the average Burdekin EoR sediment grain-size range of each event was remarkably consistent (Table 6). Thus, grain-size of the Burdekin EoR suspended load component can be expected to be relatively consistent from all sub-catchment sources, despite the sediment trapping influence of the Burdekin Falls Dam (Lewis et al., 2013).

#### 4.2.2. Improved characterisation of fine sediment transported within the GBR

The collection and analysis of mineral grains in low SPM concentration estuarine and flood plume waters (i.e.  $<10\ \text{mg L}^{-1}$ ) have previously been difficult to collect and describe. Grain-size analysis and identification of the mineral component of SPM within plume waters is limited by the presence of organic matter binding sediment grains, including live bacteria and plankton, and detritus (Bainbridge et al., 2012; Manning et al., 2017). The recommended pre-treatment protocol used in this study overcomes these challenges, and in combination with the use of the novel SediPump® high-volume filtration system for initial sample collection, allows the grain-size distribution of mineral particles transported by rivers and exported into the marine environment to be reliably and accurately measured. For the first time in the GBR, this has facilitated the documentation of transformations in SPM traits during transport from EoR to flood plume, including secondary water types at seagrass and coral reef locations at both inshore and mid-shelf settings (e.g. Old Reef, Ellison Reef).

This new conceptual understanding of Burdekin and Tully catchment to reef continuum sediment characteristics is summarised in Figs. 10 and 11, respectively. These figures synthesise all data collected during

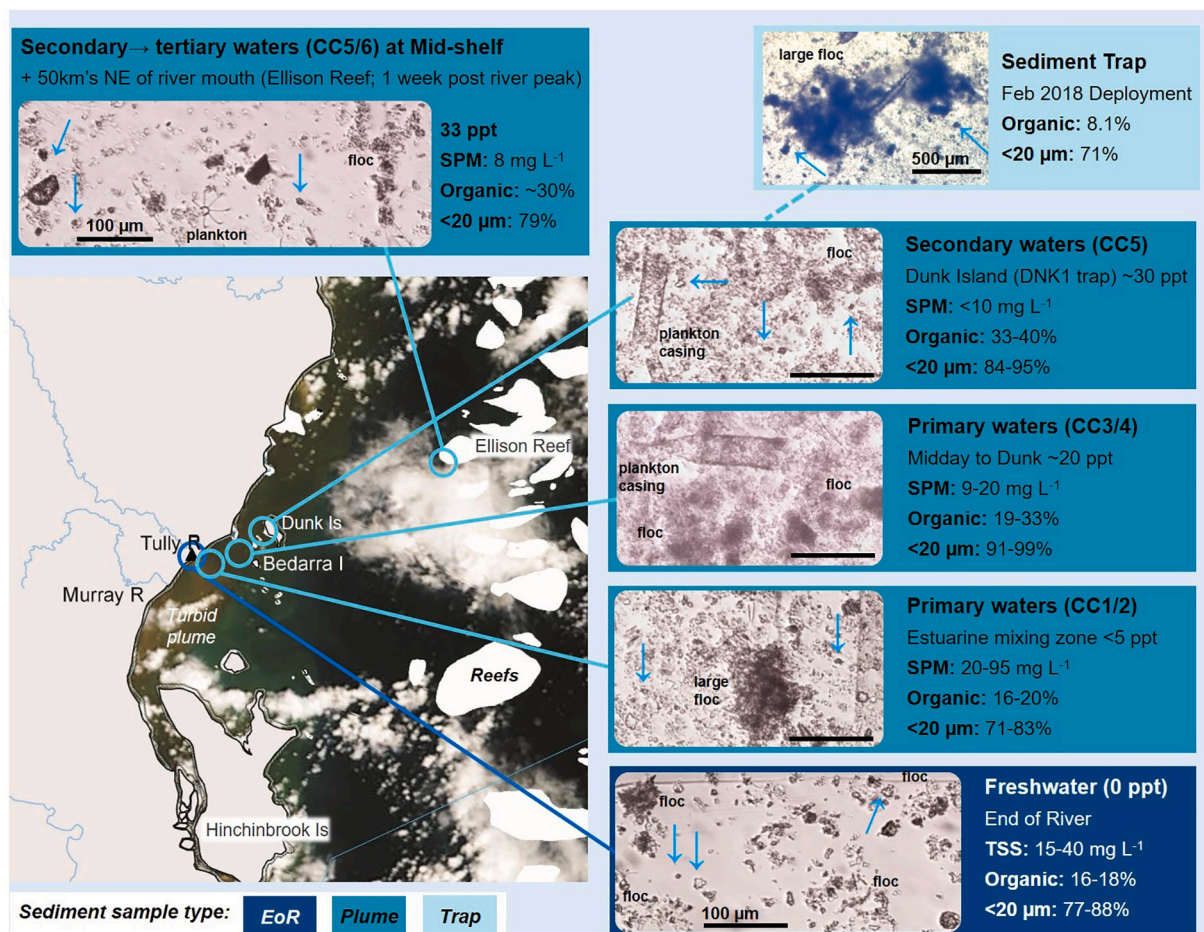


**Fig. 11.** Sediment characteristics across the Tully catchment to reef continuum, highlighting distinct flood plume water types and summarising data collected during river discharge events that occurred in 2017 and 2018. End of river (freshwater), flood plume water types and a sediment trap site that captured these flood plume events are all presented, anticlockwise from bottom right (refer to legend). The water type colour classification scheme from Petus et al. (2019) is used to describe plume waters from Colour Class 1 (CC1) to Colour Class 6 (CC6). Each water type box includes micro-imagery of a representative sediment sample and the ranges of salinity, suspended sediment concentration (TSS)/suspended particulate matter (SPM) range ( $\text{mg L}^{-1}$ ), organic content (%) and proportion of fine sediment ( $<20\ \mu\text{m}$ ) measured within each of these zones. All micro-imagery was captured from the concentrated SediPump® sample, with features labelled and blue arrows highlighting mineral particles. The scale bar represents 100  $\mu\text{m}$  unless otherwise indicated. The MODIS satellite image was captured on the 12th January 2017 and is used here to generally represent plume transitions along a salinity gradient from the river mouth. Refer to Supplement for tabulated data. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

discharge events from 2017 to 2019 (Burdekin) and 2017–2018 (Tully), and classified by water-type colour classes developed for the GBR (Petus et al., 2019). The summaries highlight the preferential transport of the clay and fine silt grains  $<20\ \mu\text{m}$  (i.e. fine sediment) within both Burdekin and Tully primary and secondary water types. While Tully EoR suspended sediment concentrations are an order of magnitude lower than for the Burdekin, once this terrigenous material is transported into the GBR lagoon, SPM characteristics (concentration, %  $<20\ \mu\text{m}$  minerals, organic content and presence of larger flocs) become comparable within both primary (colour classes 1–4) and secondary (colour classes 5) water types of the two river plumes. As SPM concentrations decrease, organic matter and flocculated particles increase in quantity and size, dominating within secondary water types. Comparison grain-size analysis on the salt-removed sub-set of these secondary water type samples (as recommended by the protocol) show these flocculated particles easily reach  $100\text{--}165\ \mu\text{m}$  in size (i.e. large floc aggregates), and that at least some finer mineral grains (i.e. clay and colloidal sizes) are bound together in transport (Tables 2 and 3). Mineral grains were measured in secondary water types transported as far as the GBR mid-shelf, including Old Reef and Ellison Reef, both  $\sim 50\ \text{km}$  off the coast from the Burdekin and Tully river mouths, respectively (Figs. 11–12). The concentrated

SediPump® samples collected within surface and depth (10 m) plume waters that reached Old Reef in February 2019 highlight the distribution of terrigenous SPM throughout the water column, down to the seafloor (Fig. 12). Micro-imagery of these samples (Fig. 12) also documents the presence of both discrete mineral grains and flocculated particles. In the context of water-quality guidelines, these low SPM concentrations (i.e.  $<5\ \text{mg L}^{-1}$ ) can still substantially impact benthic light availability; the Secchi disc depth measured at Old Reef during sampling was  $<1.5\ \text{m}$  (refer also to Lewis et al., 2020).

The marine sediment traps situated at offshore locations designed to intercept typical secondary water type movements of the Burdekin and Tully River plumes, including Orchard Rocks (115 km from Burdekin River) and Dunk Island (13 km from Tully River) have also been included for the associated wet season deployments (Figs. 10–11). Trap material contained lower ( $<10\%$ ) organic contents than measured within the secondary surface water types collected above these trap locations, with increased presence of diatoms and larger floc aggregates in the Dunk Island trap compared to other deployment periods outside of plume influence (Fig. 11, see also Lewis et al., 2020). Similar organic contents have been reported in the sediments on algal turfs associated with coastal mainland-attached coral reefs of the GBR

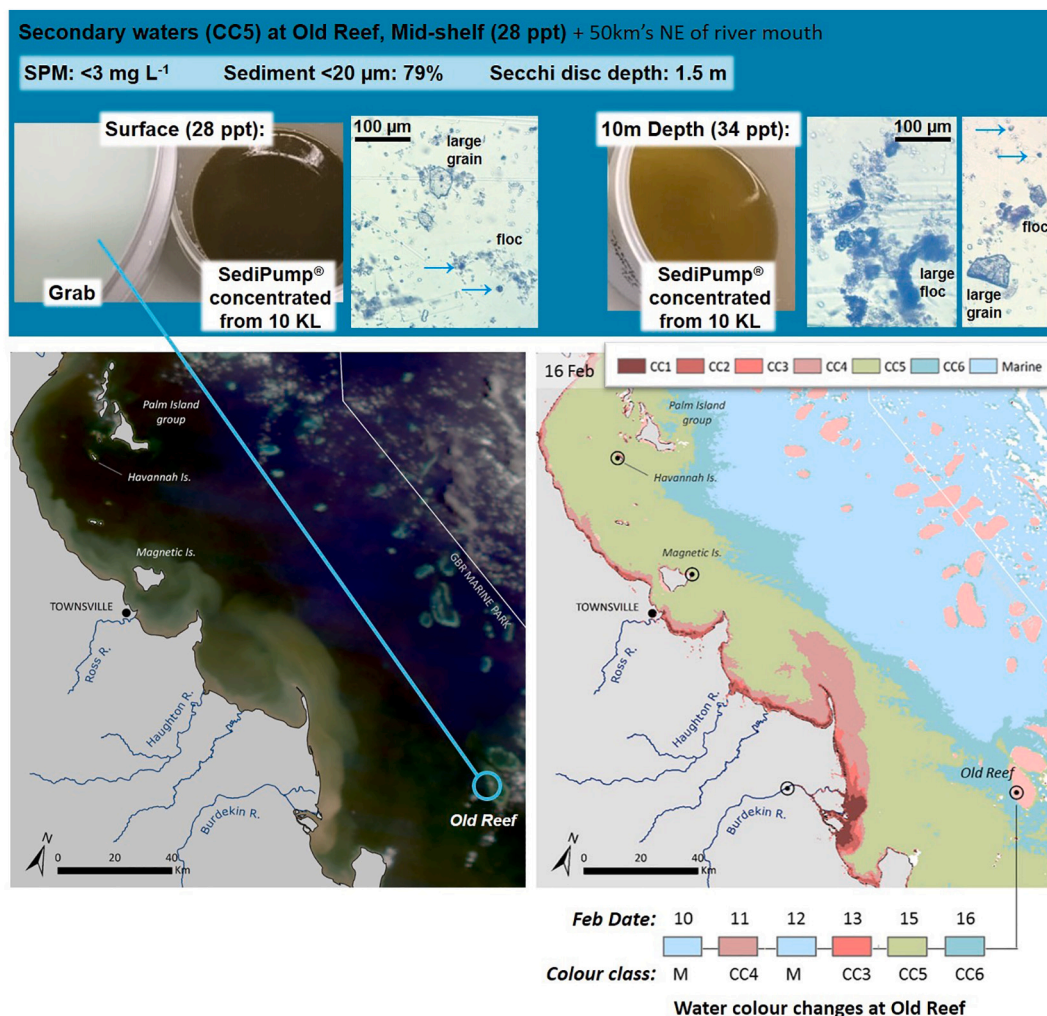


**Fig. 12.** Burdekin plume sediment characteristics in secondary waters at Old Reef, mid-shelf collected on the 15th February 2019, including SPM (suspended particulate matter), mineral sediment grain size  $<20\ \mu\text{m}$  and Secchi disc depth (m). Micro-images of sample collected at the plume surface (left, top panel) and at 10 m depth (right, top panel) illustrate the sediment and particulate organic matter present in the concentrated SediPump® sample, with blue arrows highlighting mineral particles. The surface “grab” sample is also shown for comparison. The MODIS satellite image (bottom left panel) captured on the 16th February shows the location of Old Reef NE of the Burdekin River mouth. The water type colour class map (bottom right panel) highlights the changes in water type over the period 10th to 16th February. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

(Tebbett et al., 2018). Application of this method to the sediment trap data show the accumulation rates of the <20 µm fine sediment fraction roughly double in the flood event deployment compared to dry season conditions (Lewis et al., 2020).

#### 4.2.3. Improved quantification of fine sediment influence and fate within the GBR lagoon

Clear spatial distinctions in the grain size and sediment composition were detected at the six environments targeted by sediment traps following application of the treatment protocol. Fine to coarse grained carbonate contents of trapped material was highest at the mid reef slope sites at Orpheus Island (OS1) and Havannah Island (HAV1) in the Palm Island Group. Samples collected from these sheltered locations also contained the greatest proportion of fine sediment (<20 µm) within the mineral component (85% and 86%, respectively; Table 4). In contrast, the sample collected at the higher energy mid-bay sediment field site within Cleveland Bay (CLE1) was characterised by coarse mineral and carbonate grains, with a larger sand fraction (13%) than other sites (Table 4). Sample pre-treatment had the greatest influence in refining grain-size distribution for this sample type. Sediment from the Orchard Rocks (ORC1) site, a deep (>15 m) and wave exposed outer bay mobile sediment field setting was much finer; fine sediment (<20 µm) comprised 76% of the mineral component, with sample pre-treatment having little influence on grain-size distribution shape or range. The compositions of sediments collected at each trap were associated with the type of sediment in the surrounding environment and its potential for resuspension. Factors affecting this include proximity to sediment source (i.e. river mouth versus coral reef/carbonate bank), the local exposure or resuspension energy, settling behaviours of the particles and other aspects such as local structure, benthos and bioturbation, and water depth.

Application of the marine sediment characterisation methodology outlined here will provide new insights into sediment composition and variation across a range of inshore GBR environments (for example see Lewis et al., 2020). This methodology builds on earlier studies of GBR sediments where the carbonate component has been removed (e.g. Orpin et al., 2004) and also highlights the additional influence of sample organic matter (4–8% measured in this study) in aggregating particles and skewing the grain-size distribution to coarser grain sizes. Removing the organics allows the seasonal influence of ‘terrigenous’ sediment and its character to be more accurately examined, and the addition of newly delivered flood plume sediments to be quantified through normalisation to mineral-only accumulation rates following LOI. It is this fine <20 µm terrigenous sediment that presents the greatest ecological risk to phototrophic benthos such as corals and seagrasses through a disproportional influence on light attenuation and the settling of these organic-rich and often sticky and difficult to disperse fine particles (Fabricius et al., 2003; Bainbridge et al., 2018). This methodology has benefit for understanding the relative role of marine ecosystem exposure to mineral grains exported from catchments and can assist with targeted management decisions, such as consideration of potential sites for restoration efforts. Inshore GBR trap material characterised in this study can also be used in the design of in situ and laboratory-based experiments focused on turbidity and sedimentation effects on marine species.

#### 4.3. Implications for reporting fine sediment transport across Catchment to Reef Programs

The improved characterisation of sediments being transported by two GBR rivers during wet season discharge events, including key mineral size fractions, provide important data for current GBR catchment modelling efforts under the Australian and Queensland Governments Paddock to Reef Program. The GBR Dynamic SedNet model (implemented in Source) generates fine sediment (<20 µm) loads for each of the 35 GBR river basins, including the Burdekin and Tully Rivers (McCloskey et al., 2021a). Correction for the analysis of the

‘mineral’ only grain-size distribution (i.e. organic removal) will identify variation in sediment grain-size being transported by each of these rivers. Normalisation to a true grain-size-specific suspended sediment load for GBR rivers allow direct comparison to sediment load targets, and for improved validation of modelled fine sediment loads. This is particularly important for the Wet Tropics rivers where the higher particulate organic content and aggregation of particles results in an over-estimation of coarser grain sizes for comparison against grain-size specific modelled loads. In the Dry Tropics, our research over multiple years provide quantification of the Burdekin EoR fine sediment (<20 µm) contribution ( $88 \pm 4\%$ ), for direct comparison against current modelled loads estimates. For example, the current best measured average TSS load of 4 million tonnes (Kuhnert et al., 2012) equates to approximately  $3.52 \pm 0.16$  million tonnes of fine sediment, which is close to the GBR Dynamic SedNet modelled load of 3.77 million (McCloskey et al., 2021a).

Our EoR, estuarine and flood plume sediment characterisation data will also provide valuable input parameters for the eReefs fine sediment transport and biogeochemical model components (Steven et al., 2019). While the current river basin load inputs into the eReefs model are derived from the Dynamic SedNet model which captures the variability of the <20 µm fraction, the discrimination of the finer fractions within this component is not well constrained by the model; this influences the specific modelling of the mud and dust particle sizes in the GBR. Hence such data can also be incorporated into further improvements of the ‘fluff/dust’-layer (Margvelashvili et al., 2018; Baird et al., 2021), and how this ‘new sediment’ is modelled over subsequent months within the inshore GBR.

This study demonstrates the need for standardised reporting of sediment grain size for samples collected across rivers, flood plumes, and marine settings, which is currently lacking. This will help deliver improved clarity for grain-size specific catchment loads, meeting water-quality targets, dredging compliance monitoring and offsets. Ideally at a minimum the treated <20 µm fraction and proportion of sample organic content are both required. Future research should examine additional sample pre-treatments to remove biogenic silica in flood plume samples as well as investigate the reproducibility of our method by applying other GSA techniques. Expansion of this work to quantify sediment grain size across all GBR rivers would also be highly valuable for validation of current modelled load estimates.

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#### CRediT authorship contribution statement

Zoe Bainbridge: Project conceptualisation, methodology development and laboratory analysis, figure and table production, original draft, writing, review and response to reviewer comments.

Stephen Lewis: Project conceptualisation; funding acquisition, field work, supervision, review and editing.

Tom Stevens: Field component, method development, review.

Caroline Petus: Satellite imagery analysis and image production, writing, review.

Emily Lazarus: Laboratory analysis, method development, writing, review.

Jessica Gorman: Laboratory analysis, field component, method development, review.

Scott Smithers: Project conceptualisation, supervision, review and editing.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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