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a representative sample of suspended sediment
in terms of organic matter and particle size:
A study on the US M-512 Manual Centrifuge system

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Signed

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PLYMOUTH UNIVERSITY

UNDERGRADUATE DISSERTATION

BSc GEOGRAPHY WITH GEOLOGY

An evaluation of continuous flow centrifugation to collect a representative sample of suspended sediment in terms of organic matter and particle size:

A study on the US M-512 Manual Centrifuge system

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Abstract

Continuous flow centrifugation (CFC) is used in water studies for the bulk collection of suspended sediment, thus allowing a broad range of sediment quality analysis. However, various studies have demonstrated issues associated with CFC such as: particle distribution change; contamination of sediments from instrument components or a restricted cleansing ability; and a lack of comparable efficiency rates of sediment collection. Experiments were undertaken to establish the efficiency of collection and potential changes of organic matter (OM) and particle size (PS) by CFC using the US M-512 manual centrifuge. The experiment included three different sediment types with two varied concentrations that were completed in a controlled experiment. Samples of the water-sediment mixture were collected systematically throughout the experiments and processed for various characteristics. The collection efficiency was evaluated using suspended sediment concentration variation between the influent and effluent samples collected. OM change was calculated using loss-on-ignition of the filtered sediment and PS was analysed using a LISST-ST (Laser *in Situ* Scatter and Transmissometry) particle size analyser. Sediment collection efficiency recorded for all sediments was < 50%, a value much lower than previous literature for different manufacturer's centrifuges. Furthermore, time-series of sediment retention indicated consistent collection efficiency. The centrifuge preferentially retained the mineral component of fluvial sediment but there was no significant change of OM in kaolinite or agricultural sediments caused by centrifugation. An increase in the fine fraction (<63 µm) of the PS distribution for all sediments was observed. Given the usefulness of CFC to collect adequate quantities of sediment for analysis, even in low concentration water bodies, the continued use of the US M-512 centrifuge instrument is acceptable when the associated limitations are acknowledged. As a consequence of this paper, OM content values and effective PS distributions must be considered carefully whilst there are actions that may be taken to improve the efficiency of sediment collection in future applications.

Keywords: Continuous flow centrifugation; sediment collection; organic matter; particle size.

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To my grandfather, John Anderson, who loved the river

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1. Introduction

Continuous-flow centrifugation (CFC) is a technique that separates suspended particles from water for analysis of the particulates physical and chemical characteristics (Burrus, et al., 1989; Comba & Kaiser, 1990; Douglas, et al., 1993; Horowitz, et al., 1989; Leenheer, et al., 1989; Moody & Meade, 1994; Ongley & Thomas, 1989; Rees, et al., 1991; Rossé, et al., 2006). Researchers continue to implement CFC systems from various manufacturers in sampling tasks that require the collection of a relatively large amount of suspended solids and consequently improve the analytical opportunities and their reliability (Parker, et al., 2007). Despite continued use of CFC, there has been a hiatus of studies evaluating CFC's ability to collect a representative sample.

1.2 Fine sediment study

Fluvial ecosystems play an essential role in the hydrological cycle to deliver freshwater. They are also responsible for the transport of sediment to oceans, which is vital for the geomorphological, ecological and habitat functions of rivers (Owens, et al., 2005). Sediment within water bodies is often viewed as an umbrella term that includes several parameters: coarse and fine grained sediment, bed load and suspended load, and within solution as the dissolved fraction consisting of $<0.45 \mu\text{m}$ particles (Owens, et al., 2005). Each parameter of sediment has ecological and biological importance, as well as associated detrimental effects on drainage basins. Furthermore, an increase in sediment load means a potential change in the aquatic ecosystems (Hancock, 2012). Schubert et al. (2012) mention the sensitivity of the particulate phase to anthropogenic impacts, that may disturb the natural state of water bodies. River flow conditions and particle size, shape and density determine whether sediment is transported as bed load or suspended load.

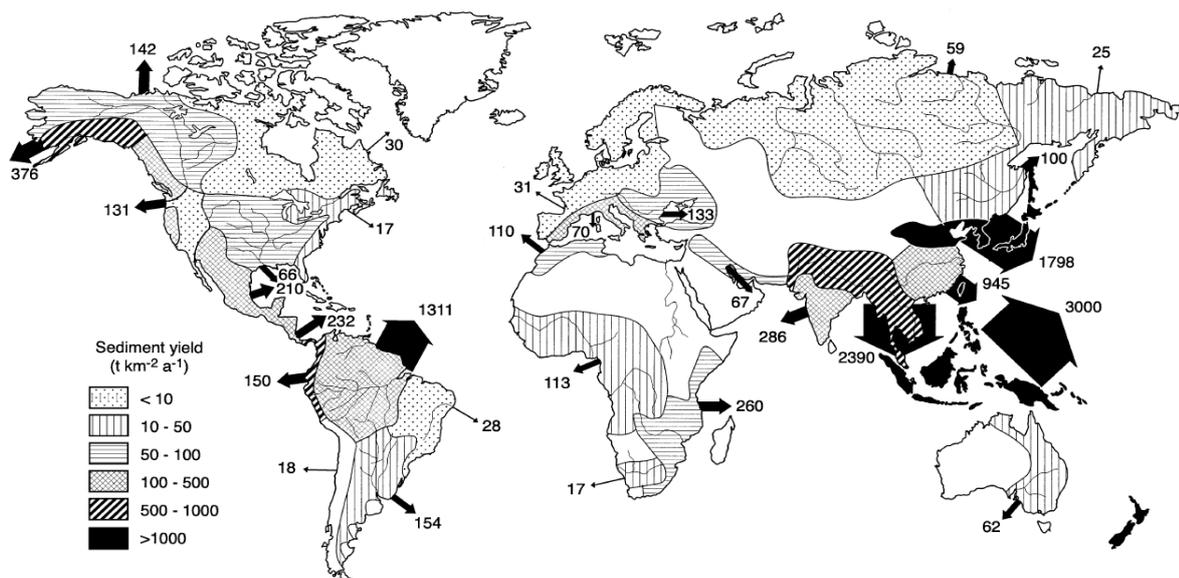


Figure 1: Annual fluvial sediment yields and sediment flux from large drainage basins to the oceans. Arrow sizes are proportional to sediment flux (in 10^6 tonnes year $^{-1}$, shown as numbers (Dearing & Jones, 2003)).

1.3 Pressures on sediment

An understanding of the spatial and temporal (present and future fluxes) patterns of water flow through a drainage basin (Figure 1) have become even more crucial for the persistently increasing human population, who are dependent on the natural resource of freshwater (Emmott, 2013). A healthy freshwater ecosystem provides life, water, energy and transportation along with many more vital ecosystem services that humans depend on (SIWI, 2009). In an unhealthy freshwater ecosystem, human health can be at potential risk from contaminants and bacteria that are transported by sediment (U.S Environmental Protection Agency, 2013).

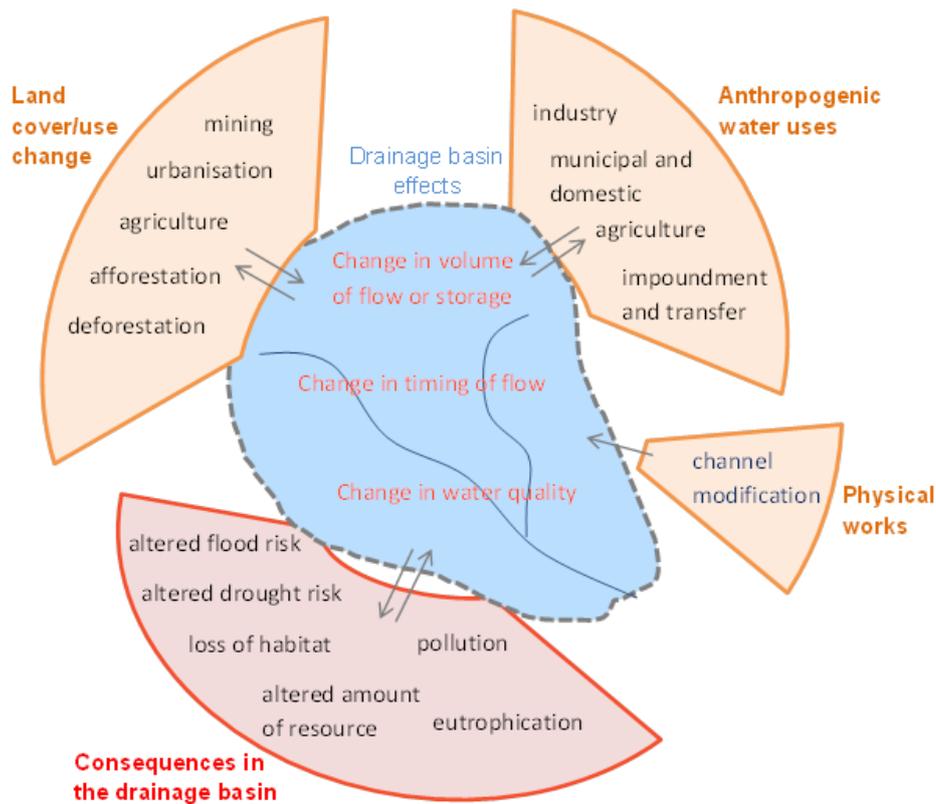


Figure 2: Effects and consequences of changes within a drainage basin, adapted from Arnell (2002).

In spite of their dependence on freshwater systems, humans have a long history of altering drainage basins. The quality of water in rivers across the world is under increasing pressure, as ecosystems are forced to struggle due to anthropogenic influences that have diverse and multiple impacts (Figure 2). This sometimes causes irreversible changes in the way those river systems function (Allan & Castillo, 2007; Owens, et al., 2005; Taylor & Owens, 2009). The global transport of sediment in fluvial systems through land use change and intensification has increased by 2.3 ± 0.6 billion metric tons per year whilst the flux of sediment reaching the coast has reduced by 1.4 ± 0.3 billion metric tons per year by sediment retention in reservoirs (Syvitski, et al., 2005).

Additionally, almost an 80% reduction of total sediment yield of drainage basins in Brazil and Thailand have been attributed to dam projects, yet drainage basins in Columbia and Papua New Guinea have experienced sediment load increases approximately 40% due to land use change and surface disturbance (Walling, 2006).

The challenge to mitigate water body contamination will intensify with the increasing global population growth estimated at 9.6 billion by 2050 (United Nations, 2012) (*Appendix*, Figure 20). Future food consumption and agricultural needs are complex topics and are affected by a range of factors, but inevitably a larger population requires greater agricultural production to provide adequate food supplies (Kearney, 2010). Pesticide use will continue to increase in conjunction with agricultural expansion that will likely cause an increase of sediment load and contaminant levels of fluvial systems. Tilman et al., (2001) estimate that 10^9 hectares of ecosystems will be converted for agricultural production by 2050 and agriculture will be a major driver of global environmental change.

1.4 Governance of river quality

As sediment and water quality are linked to public and fluvial ecosystem health, they are applied as functions for multi-parameter water body quality guidelines implemented by government institutions worldwide; examples include the EU Water Framework Directive (WFD) (European Union, 2000) and the U.S National Rivers and Streams Assessment (NRSA) (U.S Environmental Protection Agency, 2013). The aim of these water body quality guidelines and monitoring programmes is to inform their governments of potential human and ecological health risks due to contaminated water bodies. There is no single institutional body that has the ability to enforce monitoring or remediation efforts of polluted water bodies. However, the World Health Organization (WHO) has developed water quality guidelines for drinking water and has compiled an internationally accessible index of chemical hazards and remediation effort outlines that are sent to aide authorities in their management strategy (WHO, 2013). Without an international obligation to monitor and tackle water body pollution, it can be assumed that wealthy, developed countries of North America and Europe are at the forefront of studying remediation efforts targeted at improving sediment and water quality.

1.5 Project context

Considering the future population and agricultural dynamics along with possible associated impacts on a drainage basin, sediment and water quality monitoring and research projects are essential to improve understanding of the dynamic, vulnerable fluvial system. There are numerous methods for the collection of sediment from water bodies for further chemical analysis. Different sampling methods and various applications are determined by project objective; further descriptions can be found in sections 2.6 and 2.7, *Literature Review*. For bulk collection of suspended sediment from rivers, a continuous flow centrifugation (CFC) system is a well-established and evaluated technique.

Investigation of CFC systems from several different manufacturers to collect sediment samples have presented a variety of efficiency rates ranging from 65 - 85 per cent with an Alfa Laval centrifuge (Ongley & Blatchford, 1982) up to 92 - 99 per cent using a Westphalia centrifuge and also an Alfa Laval (Horowitz, et al., 1989). Continued implementation of CFC techniques for research at the Quesnel River Research Centre (QRRC), Canada, using an un-evaluated centrifuge (US M-512 centrifuge system) prompted an investigation of the centrifuge instrument and its efficiency.

1.6 Research objectives

Despite the well-studied centrifugation technique, there is no evidence that the reported rates of efficiency and representativeness of sample collection are transferrable to another manufacturer's instrument. Previous work evaluating CFC systems has not evaluated the US M-512 centrifuge system. Centrifugation has been shown to cause sample contamination and sediment breakdown. In the present report, centrifugation and the US M-512 instrument are evaluated on their ability to collect a representative sample of organic matter and particle size of the suspended sediment. To achieve the aim three research objectives, questions and hypotheses were formulated and presented in Figure 3 below.

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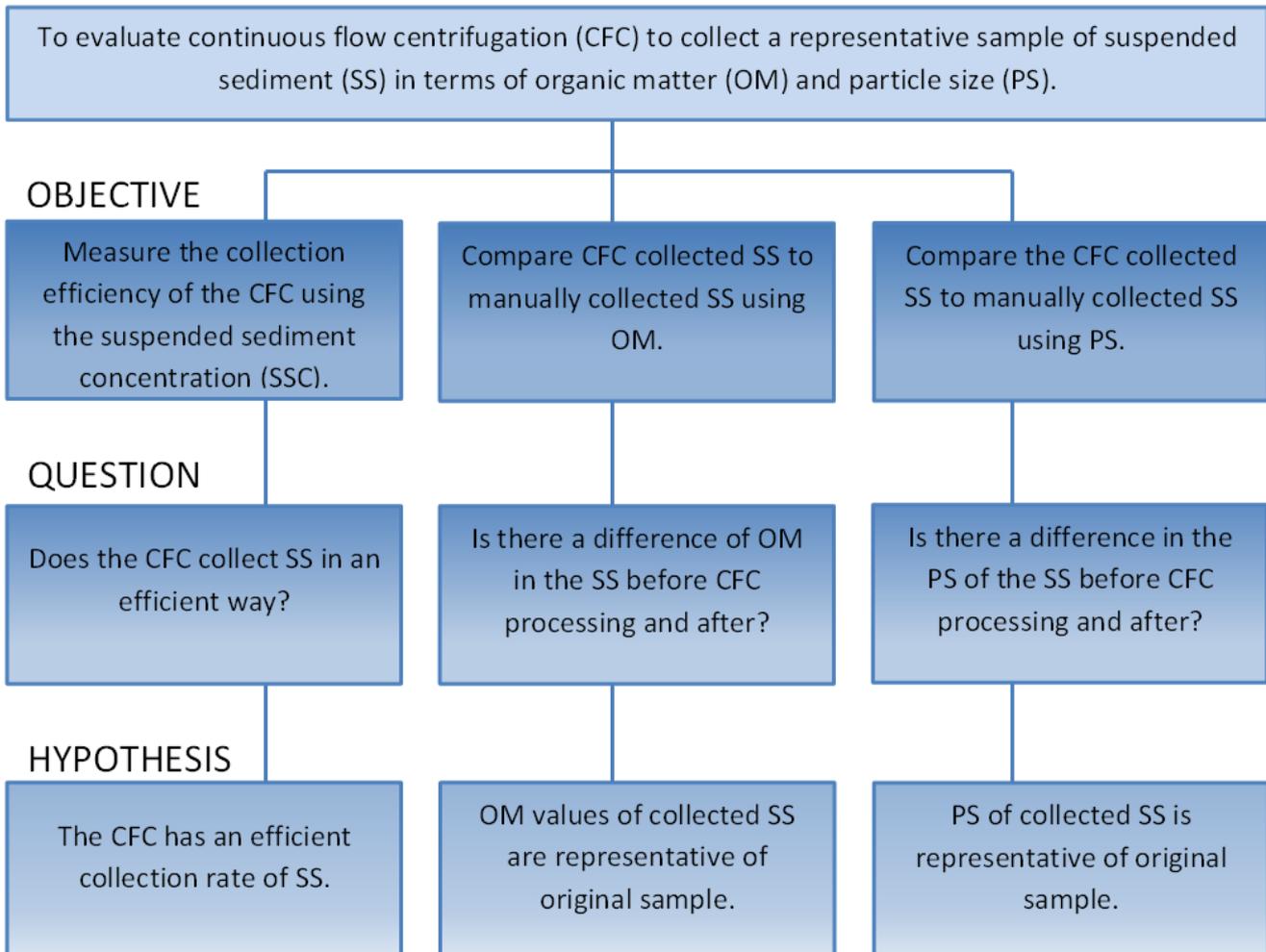


Figure 3: Project flow chart

2. Literature Review

The purpose of this literature review is to give context to the dissertation and to place the research undertaken within a wider context of the complex terrestrial aspect of the hydrological cycle and its associated sediment.

2.1 Water quality and contaminant guidelines

Water quality is principally, but not exclusively dictated by sediment quality. Determined 'safe' concentrations of hazardous substances and other chemicals are identified and listed by the United Nations (UN Environment Programme, 1996) for the intended purpose of assessing drinking water quality. Parameters used for a more specific assessment of the water body's environmental status are identified in the European Union's Water Framework Directive (WFD) introduced in 2000 (European Union, 2000). They include nutrients, organic matter, hazardous substances and other chemical determinants and biological quality elements (Carere, et al., 2012; European Environment Agency, 2013).

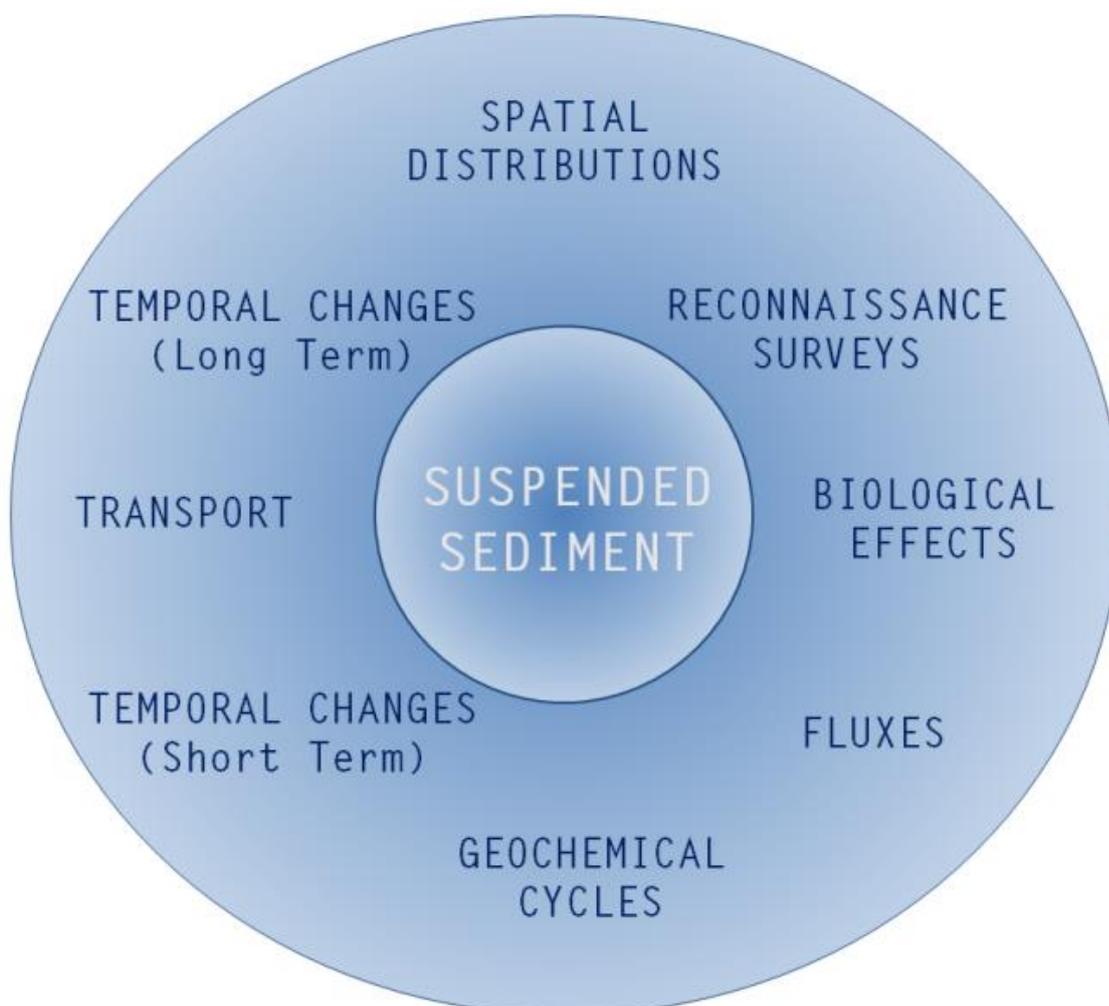


Figure 4: The various types of trace element studies in which suspended sediment sampling and analysis is used, adapted from Horowitz (1995).

The Society of Environmental Toxicology and Chemistry (SETAC) workshop, held in August 2002, further defined the sediment quality guidelines (SQGs) for 28 chemicals or contaminants of concern in freshwater. Each contaminant is applied to a concentration-response model (Figure 5) to consider SQG approaches. The probability of a biological effect is relatively low until the threshold-effect (TE) guideline is reached. At the high end of the distribution, above the probable-effect (PE) biological effects almost always occur (Figure 5) (MacDonald, et al., 2000; Wenning & Ingersoll, 2002). The SETAC workshop and Scrimshaw et al., (2007) reported the use of SQGs should be in a weight of evidence (WOE) approach as only one, initial line of evidence (LOE).

The main issues for sediment have been deconstructed by Owens et al., (2008). Economic, environmental and societal elements must all be involved in the sustainable management of fluvial systems. SQGs are set for contaminants within the environment but are required for the protection of water, thus protecting societal interests.

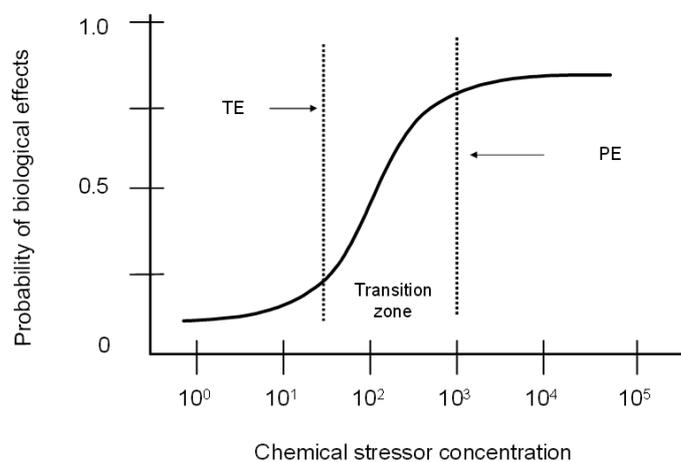


Figure 5: A generalised concentration-response model describing the TE and PE guidelines of increasing probabilities of adverse biological effects for a single sediment-bound contaminant (Wenning & Ingersoll, 2002).

Table 1: Reasons for implementing fluvial sediment management strategies, adapted from Owens (2007).

Sediment Management
To maintain urban drainage and sewage systems
To maintain shipping transportation by 'maintenance dredging' of river channels, estuaries, ports, harbours, etc.
To maintain the life-span of reservoirs and for operation reasons
To ensure the efficient flow of water in watercourse and reduce flooding
To maintain geomorphological features, sometimes for aesthetic or recreational needs (such as gravel bars, beaches, etc.)
To maintain or improve water quality

2.2 Sediment transport

Information on the sediment characteristics and transport processes in fluvial environments are important. A better understanding of sediment can give additional guidance to potentially improve management procedures (Table 1) of fluvial environments for all stakeholders.

There are several definitions of sediment, a useful and agreed upon definition in Owens (2008, p.2) reads: "*sediment is suspended or deposited solids, of mineral as well as organic material, acting as a main component of a matrix which has been or is susceptible to being transported by water.*" Moreover, from a drainage basin management perspective sediment is vulnerable to the objective perception of an individual or stakeholder due to sediment behaviour, such as a natural substrate, a siltation nuisance or a potential contaminant.

Suspended load usually has a lower critical erosion velocity than bedload as it is composed of finer material, which is more easily transported (Bishop & Prosser, 2001). Suspended load accounts for approximately 90% of total sediment flux in rivers, globally (Walling & Fang, 2003). The majority of the suspended load is sediment of grain size <2 mm (sand-sized or less), with much of this being <63 µm (i.e. silt- and clay-size material) (Owens, et al., 2005). During high energy storm events larger particles are able to be transported in suspension (Taylor, et al., 2007). Considerable focus in literature is on the fraction of fine-grained particles (<63 µm) because it is the most chemically active component of sediment. Many contaminants and nutrients are transported and stored in association with these fine-grained particles (Horowitz, 2008; MacDonald, et al., 2000; Owens, et al., 2005).

Table 2: Sediment sources and the associated contaminants in a drainage basin (Taylor et al., 2007).

Material	Source
Sediment (organic and inorganic)	Erosion from rural, agricultural and forested land, channel banks, urban road dust and construction, STW solids, atmospheric deposition, inputs from tidal areas and coastal zone (during flood and ebb tidal cycle)
Metals (Ag, Cd, Cu, Co, Cr, Hg, Ni, Pb, Sb, Sn, Zn, As)	Geology, mining, industry, acid rock drainage, sewage treatment, urban runoff
Nutrients (P, N)	Agricultural and urban runoff, wastewater and sewage treatment
Organic Compounds (Pesticides, herbicides, hydrocarbons, PCBs, PAHs, dioxins)	Agricultural, industry, sewage, landfill, urban runoff, combustion
Radionuclides (¹³⁷ Cs, ¹²⁹ I, ²³⁹ Pu, ²³⁰ Th, ⁹⁹ Tc)	Nuclear power industry, military, geology, agricultural (secondary source).

2.3 Sediment particle size

Suspended sediment (SS) associated contaminants include heavy metals, nutrients, micro-organics and radionuclides and can all be studied using information of the particle size (Table 2) (Walling, et al., 2000). Particulate size and characteristics are fundamental controls on the sediment settling velocity, entrainment, transportation and deposition through fluvial systems (Owens, 2007; Walling, et al., 2000). The specific surface area of a particulate controls the surface chemistry and its ability to absorb sediment-associated contaminants (Walling, et al., 2000). Gibbs (1977) proposed that the major factor governing the concentration of contaminants is the grain size; surface area of the particulate.

Fine-grained particles (< 63 µm) are usually transported and deposited as composite particles or 'flocs.' Flocculation of particles occurs as an aggregation process of naturally cohesive sediments. The flocculated sediment has different characteristics and properties, consequently they exhibit different hydrodynamic behaviour to the constituent particles (Droppo, 2001). Organic compounds flocculate with inorganic fine sediment that modify settling rates, as noted by Petticrew & Arocena (2003). Flocs constitute four main components: mineral properties, organic material, water and air (Figure 6) (Owens, 2007). Flocculation requires consideration especially as it complicates the transportation and deposition of SS. Additionally, the grain size fraction below 63 µm of sediment is the most chemically active component that acts as a vector for the transport of the materials listed in Table 2 (Horowitz, 2008; Owens, et al., 2005).

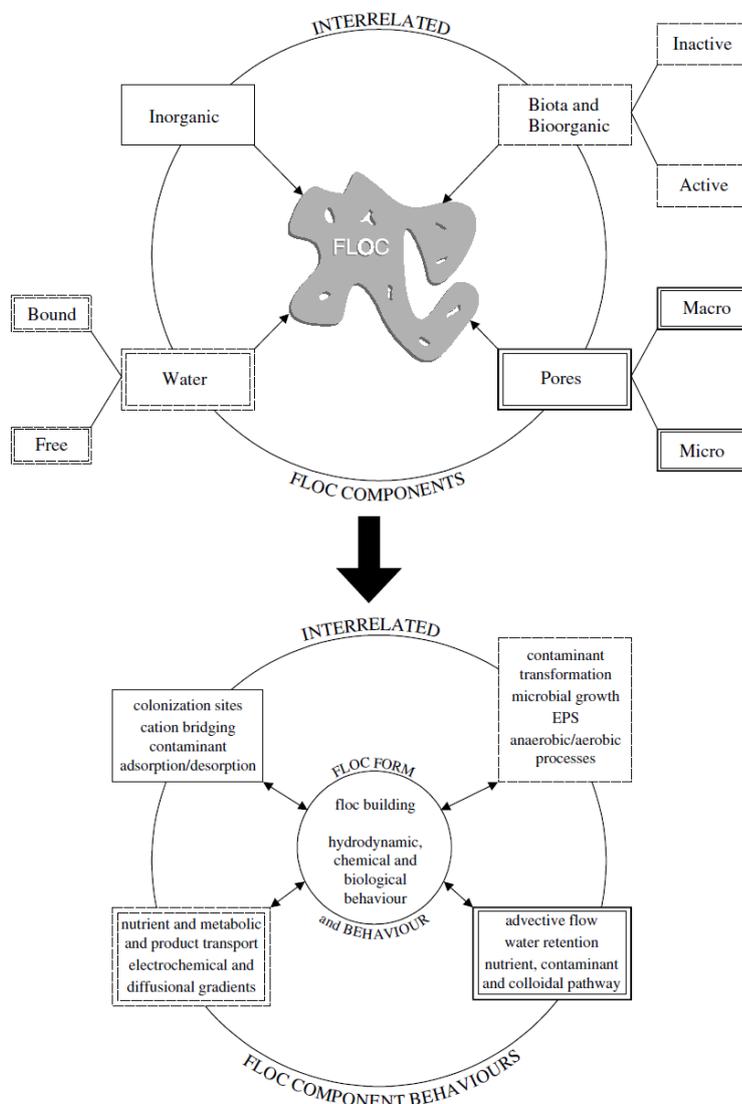


Figure 6: Conceptual model of the interrelationship between form and behaviour of a sediment floc. The model shows the links between individual components and their behaviour on overall floc form and behaviour (Droppo, 2001).

2.4 Organic matter in suspended sediment

The fine-grained fraction of organic matter (OM) is a primary energy source for fluvial systems as it provides a habitat and a feeding source for invertebrates, whilst OM of diameter >10 cm has been proven to reduce the erodibility of sediments in forested streams (Eggert, et al., 2012; Slack & Feltz, 1968). OM supply varies both spatially and temporally through a fluvial system. The River Continuum Concept suggests that the river 'metabolism' changes from heterotrophy to autotrophy, depending on the availability of OM and light. Therefore, the energy inputs and its forms progressively change downstream in conjunction with the biological communities (Vannote, et al., 1980). OM and chemical analysis of biota have a longer time dimension than the chemical quality of the water because the biota can be affected by numerous events that may have occurred before sample collection (UN Environment Programme, 1996). OM supply to rivers also varies seasonally, causing an issue for the fluvial ecosystem's health when heavy litter fall occurs during low stream flow. Additionally, a large amount of OM supply may cause deoxygenation of the water (Hynes, 1975; Slack & Feltz, 1968).

2.5 Sediment importance and associated functions

Owens (2008, p.13) summarised sediment functions and importance:

- as part of the global denudation cycle;
- for global biogeochemical (including carbon) cycling;
- for transferring nutrients and contaminants from terrestrial to freshwater to marine and coastal systems;
- for being (i.e. sediment itself) and creating (e.g. beaches, channel islands, saltmarshes) aquatic habitats and landforms;
- by helping to maintain a high level of biodiversity within aquatic systems through the creation of diverse sedimentary environments;
- for providing an important natural resource (e.g. aggregates, fertile soil on floodplains);
- for the functioning of coastal ecosystems and the evolution of deltas and other coastal landforms.

These functions are also referred to as ecosystem services that are regarded to be the benefits that society gets from the natural environment (Food and Environment Research Agency, 2012). The ecosystem service approach promoted by the Millennium Ecosystem Assessment (2005) has become essential for understanding the links between biodiversity and land use management; four categories are recognised supporting (food and energy materials), regulating (carbon, water flow and quality, human health), provisioning (biodiversity, soil formation, nutrient recycling) and cultural (science & education, tourism & recreation and history) (Gilvear, et al., 2013). An important inter-relationship between sediment amount and composition and the aquatic biota in the fluvial environment has likewise been suggested (Burcher & Benfield, 2006; Cotton, et al., 2006; Owens, 2007).

2.6 Anthropogenic influence on fluvial systems

There is a wealth of studies demonstrating the impacts of change in land use on soil erosion as well as the associated variations in the drainage basins' sediment yield (Walling, 2006). Land use changes are a consequence of human development. Post-1700, significant amounts of natural ecosystems have been converted into agricultural ecosystems (Mannion, 2002). These alterations are mainly driven by the continuing population increase and the related results of permanent settlements, increased trade, food security (e.g. political pre-eminence), innovations in food production and power over nature (Mannion, 1999). Anthropogenic influence is a function of the 'power over nature' topic as humans are characterised as the "*dominant organism whose influence determines the nature of the rest of the system*" Simmons (1993, p.23).

Anthropogenic activities have a diverse and multi-impact influence on fluvial drainage basins. Enforced stakeholders (governmental institutions) of the natural resource (freshwater) create physical management procedures (infrastructure such as; bridges, dams, floodplain reclamation and levee's) for socio-economic reasons, whilst non-government organisations (NGOs) (United Nations, 2013; World Health Organization, 2013; UNICEF, 2013) attempt to increase global access to safe drinking water. Both the governments' and NGOs' actions, whether they are perceived as positive or negative, affect fluvial systems' geomorphology, water quality and associated sediment transport. The quality of water and sediment can be viewed as separate factors or as interlinked components of a drainage basin and the associated ecosystem.

2.7 Sediment sampling challenges

The sampling strategy is determined by the aims and objectives of an investigation. The study must then be further classified into three levels of monitoring: pilot survey, baseline survey or a temporal trend survey, as summarised by Parker et al., 2007. Sediment sampling requires the collection of representative samples. This collection has inherent challenges for researchers that include the requirement of SS in large quantities for analysis and the natural variability (spatial and temporal) (Morrisey, et al., 1992; Owens, 2007; Phillips & Walling, 1995; Taylor, et al., 2007).

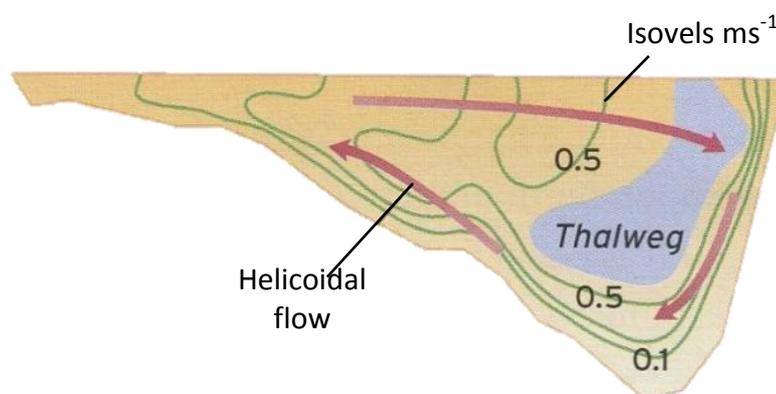


Figure 7: Patterns of velocity distribution within river channels shown using isovels - lines of equal velocity (ms^{-1}). The thalweg and helicoidal flow that complicate instrument placement within the river channel are indicated (Gilvear, 2008).

Spatial variability of SS occurs at the sampling site. Consideration of instrument placement in the river channel cross-section is crucial as SS transport in the channel is not uniform. Channel margins may cause localised disturbance of the river flow so studies like Phillip & Walling (1995) note to locate in-situ sampling near to the channel thalweg (Figure 7 and 9). Helicoidal flow of water near the meander bend of a river channel could also influence SS transport in the water (Gilvear, 2008).

The research objectives and sampling location must include consideration for possible effects of tributaries, point sources and diffuse sources in addition to the study's objectives. The order number of the river within the drainage basin must also be considered. To examine SS associated nutrients and contaminants, samples should not be taken in the upper reaches of a drainage basin (Parker, et al., 2007). To account for the variable distribution of SS within water bodies, pilot studies can identify the spatial characteristics of SS within water bodies and help to make an informed decision about the research site (Morrissey, et al., 1992).

Temporal variability can additionally affect the SSC. Short-term events, such as floods, create peak transport conditions of SS (to $> 10 \text{ gL}^{-1}$), although these events occur infrequently (Owens, 2007). Sampling after high-energy events may cause under-representation of SSC due to a flushing of the fine sediment from the gravel beds (Parker, et al., 2007). Seasonal variations and their impact on flow conditions are also well-studied (Cotton, et al., 2006; Owens, et al., 2005). Long-term events such as seasonal variations are controlled mainly by climatological conditions. Decadal or near-annual episodic events, like El Niño, deliver extraordinary amounts of sediment through fluvial systems as noted by Aalto et al. (2003).

Adequate sampling strategy reviews and clear study objectives have the ability to obtain representative samples and mitigate the majority of spatial and temporal variables caused by the nature of sampling the natural world.



Figure 8: High flow conditions when SS is essentially 'flushed' from the gravel bed (Parker, et al., 2007). Jamieson River, Kamloops, British Columbia, Canada. Credit: Authors own.

2.8 Sediment sampling techniques

The sampling technique for SS collection is dependent on the aims, objectives and funding of a study. Wildhaber et al. (2012) evaluated in-situ SS sampling techniques. The methods also include time-integrated sampling, where the periods of sample collection are dictated by the research objectives (Figure 9) (Phillips, et al., 2000). There are many options to collect SS, for example manual (grab) sampling usually collects bulk samples and is mostly deployed during high flow conditions. Automatic samplers, such as the Wallingford Integrated System for Environmental monitoring in Rivers (WISER) are used for a range of river environments, reduce the manual labour and extensive time required for river monitoring programmes. WISER also has the ability to match the sampling frequency with the flow conditions (Evans, et al., 1997). The Isco portable sampler is a single function automatic sampling technique that collects grab samples of water from a river channel at a pre-determined sampling frequency. This is an accurate and representative automatic sampler when compared to manual grab sampling (Herkenham & Chang, 2013). All sampling techniques have intrinsic limitations but in order to recognise and quantify them, studies are necessary to improve future sampling decisions.



Figure 9: Time-integrated SS sampler in-situ in the thalweg of a river channel following Phillips et al., (2000). Fishtrap Creek, Kamloops, British Columbia, Canada. Credit: Authors own.

2.9 Continuous flow centrifugation

A continuous flow centrifugation (CFC) has the ability to sample relatively large quantities of water (> 100 L) and separate the SS for a broad range of sediment quality analysis (Ongley & Blatchford, 1982; Rees, et al., 1991). CFC works within a system that includes a submersible pump to extract water from the water body. The pump operates at a constant rate driving the water and associated SS into the CFC, which then separates the sediment by centrifugal forces and pushes the particles to the bowl wall. Processed water is flushed from the bowl as effluent, more detail of a CFC system is provided in section 3.1, *Methodology*.

The collection of a bulk sample of sediment permits characteristics (geo-chemical, physical, mineralogical, toxicological and radio-nuclei) of the SS to be analysed (Horowitz, 1995; Parker, et al., 2007). Many of these characteristics only occur in low concentrations so that the vast amount of water sampled by the CFC allows for the collection of contaminants in concentrations that are useable for further analysis. Sediment samples analysed for metal element are collected using the re-suspension technique, described by Lambert & Walling (1988) and cross-referenced with samples collected by the US M-512 centrifuge. The sediment collected using these two sampling techniques has shown comparable results for metal concentration analysis (Clark, 2013).

Large samples of sediment also reduce the possibility of contamination during sampling and processing and potentially enable replicate analyses to assess analytical precision (Parker, et al., 2007). Other processes for SS separation like filtration are generally not feasible for applications of large volumes of water due to the time required for processing the sample water. Studies such as Comba & Kaiser (1990) in the St. Lawrence River, used both filtration and centrifugation in river conditions with extremely low SSC (0.9 mg l^{-1}). Centrifugation resulted in greater particulate retention, therefore sediment collection using CFC was more useful for analysis. In addition, Buffle et al., (1992) observed that CFC is not affected by problems like membrane clogging and surface coagulation that result in poor separation during filtration. CFC processing is possible on river channel banks, unlike filtration, this eases the issue of transport and storage of large volumes of water and minimises time for sample alteration (Parker, et al., 2007).

Using CFC has inherent limitations for sediment collection. A CFC is a relatively heavy and cumbersome instrument. This can limit deployment possibilities at certain channel bank locations. Sampling times required for the collection of sufficient amounts of suspended sediment can be in the order of hours, especially when sediment concentrations are as low as in the Comba & Kaiser (1990) study.

Along with the practicalities of deploying a CFC in the field (Figure 10), the representativeness of the sample can be another limitation. During long sampling periods, changes in sediment separation characteristics within the centrifuge bowl may occur (Rossé, et al., 2006).

Sediment retention in a centrifuge is limited to particles of diameter larger than 370 nm (Rees, et al., 1991). This selectiveness by the centrifugation process is accompanied by a potential modification of the natural state of the sediment either by aggregation or disaggregation, and alters the effective particle size of the sediment (Rees, et al., 1991; Rostad, et al., 1998). Rees et al., (1991) and Rossé et al., (2006) mention the centrifuge-collected sediment may be contaminated by components within the instrument that affect the representativeness of the sediment sample. Effectively cleaning the instrument and using stainless-steel centrifuge bowls can reduce such contamination of samples.

Various manufacturers produce CFC systems, but while they all operate on the same principle of centrifugation, their design and reported efficiency (calculation in *Methodology 3.1*) rates differ. This variety of instruments available has led to research on efficiency valuations of CFC: 65-85 per cent using the Alfa Laval (Ongley & Blatchford, 1982); 74 per cent (Moody & Meade, 1994); 70-90 per cent using the Alfa Laval (Ongley & Thomas, 1989); 72-94 per cent using the Sharples (Leenheer, et al., 1989); 84-90 per cent using the Westphalia (Burrus, et al., 1989) and 92-99 per cent using both the Westphalia and Alfa Laval centrifuges (Horowitz, et al., 1989). The revolutions per minute (rpm) that a centrifuge is rotated at also differ according to the manufacturer: Sharples AS-12 \approx 16,000 rpm, Westphalia \approx 9,800 rpm (Table 7, *Appendix*), that may restrict the comparison of CFC due to the centrifuge instrument used.

Centrifuge instruments are still deployed in the field despite the lack of recent evaluation. In this study CFC evaluated using sediment and the constituent particle characteristics that are crucial for the majority of sediment quality analysis.



Figure 10: CFC deployment on a gravel beach.

Quesnel River, Cariboo Region, British Columbia, Canada. Credit: Authors own.

3. Methodology

3.1 Experimental set-up

The CFC experiment was set up as a controlled laboratory study. Sediment characteristics analysed to assess the efficiency of the CFC were SSC, OM and PS. To address the project objectives, two components were controlled:

- (1) sediment type, specifically laboratory kaolinite, re-suspended river sediment ($< 500 \mu\text{m}$) and agricultural soil ($< 125 \mu\text{m}$)
- (2) sediment concentration ($5\text{-}15 \text{ mg l}^{-1}$, $15 - 40 \text{ mg l}^{-1}$) inputs.

The experiment was controlled to limit the impact of environmental conditions. The groundwater used throughout the experiment was aerated and pumped on-site at the Quesnel River Research Centre (QRRC) and characterised by a conductivity $\approx 180 \mu\text{S}$ and a $\text{pH} \approx 7.15$. The reservoir water was replaced by fresh groundwater before each experimental run. Water was drawn up into the CFC using a submersible pump located within the reservoir by a 'food-grade' non-toxic PVC tubing and out through PVC rubber hosing at an average flow rate of $93.6 \pm 0.52 \text{ l min}^{-1}$ at a rate of 3000 rpm. A 'run' of one type of sediment, at one concentration was repeated three times to complete a 'set' of runs. Each set of runs was averaged out for data analysis.

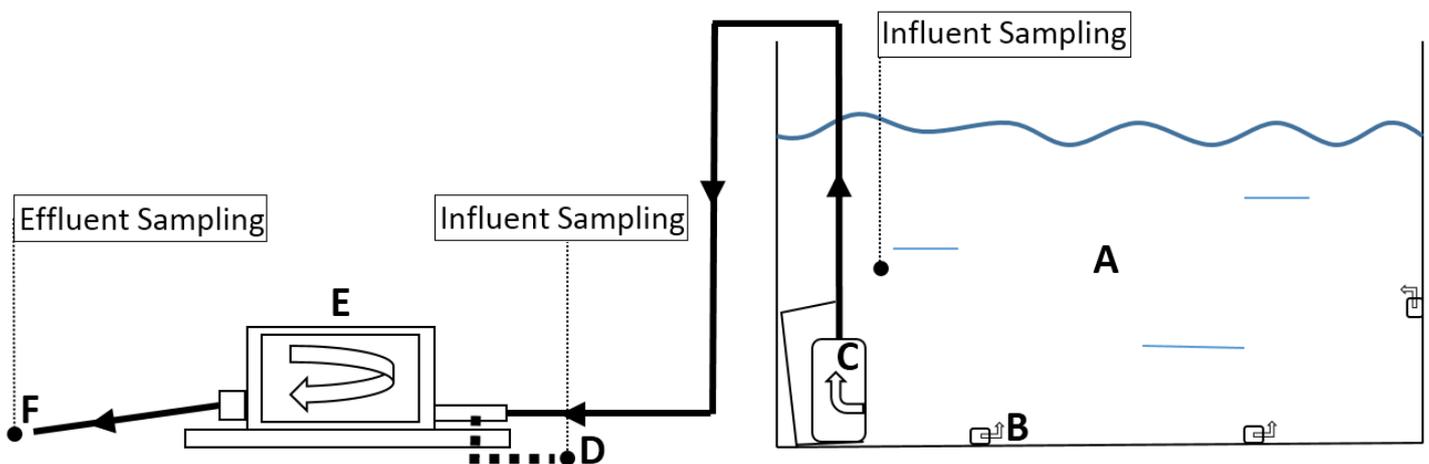


Figure 11: Representation of the experimental set-up. A: reservoir; B: aquarium bubblers; C: submersible pump; D: CFC bypass outlet, only opened to take influent sample at t ; E: US M512 centrifuge system; F: Outlet PVC hose at an angle (approximately 15°). A, D and F represent sample collection points at $t = 2, 4, 6, 8$ and 10 min.

The centrifuge was operated in a closed system as depicted in Figure 11. The reservoir was filled prior to the experiment and aquarium bubblers were located on the reservoir floor and walls to maintain circulation. Sediment was initially processed in an ultrasonic bath before being introduced into the reservoir (Cole-Palmer 8890 ultrasonic bath). The sediment was dispersed into the reservoir and re-suspended using a paddle in between sample collection. CFC influent and effluent samples were collected at $t = 2, 4, 6, 8, 10$ minutes (min). The operating system adapted for the experiment was chosen to provide comparable data between sediment and concentration types.

To minimise the potential contamination within the experimental set-up, a rigorous set of rinsing procedures were undertaken on each instrument in the set-up, and a 'clean' run of the system using only tap water was completed to flush the system between runs.

The US M-512 manual centrifuge system and submersible pump were powered using a current-converter housing and Canadian standard 230V mains outlet. The water flow was controlled using the pump on the '*automatic*' setting. To avoid contamination, a sample of influent water was taken from within the reservoir (sampling point A) and used to determine the consistency of the reservoir SSC and the sample entering the centrifuge SSC (sampling point D).



Figure 12: US M-512 centrifuge system in controlled experimental set-up at the Quesnel River Research Centre, British Columbia, Canada.

3.2 Sediment concentrations and organic matter

The suspended sediment concentration (SSC) and organic matter (OM) were calculated as described below. This process was done on-site at the QRRC.

Influent and effluent samples were collected in polyethylene bottles (Nalgene®) and a representative sub-sample of 100 ml was directly transferred for particle size analysis (*Particle size analysis*, section 3.3). The remaining samples were vacuum-filtered through filters (Whatman™ glass microfiber filters with > 0.6-0.8 µm particle retention) that were pre-ashed (Thermolyne furnace at 550°C for one hour), sat on a filter manifold, and subsequently placed in numbered pre-weighed tin trays to minimise contamination. Samples were processed using the loss on ignition (LOI) method (Ball, 1964). The sediment laden filters were dried (VWR muffle oven, 60°C for 24 hours) before a dry weight was taken (Sartorius™ four digit analytical balance). The filters were then ashed (furnace-dried at 550°C for one hour) and weighed again.

Equations for OM content and the centrifuge efficiency:

Equation 1

$$OM\ Content\ (\%) = \frac{M\ ashed - M\ dried}{M\ dried} * 100 \quad M = \text{mass (grams)}$$

Equation 2

$$Efficiency\ of\ Retention\ (\%) = \frac{SSC\ before - SSC\ after}{SSC\ before} * 100 \quad SSC = \text{suspended sediment concentration (mg l}^{-1}\text{)}$$

3.3 Particle size analysis

The sub-samples collected from the influent and effluent water were kept in solution, stored in a cool, dry place out of direct sunlight and transferred to the University of Northern British Columbia (UNBC) prior to analysis. A Sequoia Scientific, Inc. (Bellevue, WA, USA) laser *in situ* scatter and transmissometry (LISST-100) particle size analyser instrument was used for particle size analysis (PSA) of samples. The LISST has a working particle range of 2.5 to 500 µm, in 32 logarithmically spaced classes. The instrument uses the principle of laser diffraction to measure the particle size instantaneously. Pedocchi & García (2006) stated that the LISST utilises the Lorenz - Mie theory model for scattering that allows the effect of the refractive index and the measurement of the scattered energy at large angles to be taken into account. Therefore, the LISST gives a better description of the laser scattering pattern than the one obtained by Fraunhofer diffraction theory.

The LISST sensing reservoir is designed to be non-intrusive and is set back to allow the optical chamber that fits into the reservoir to be inserted. The LISST was operated in a laboratory setting connected to a computer using the LISST standard operating procedure software (version 5).

The LISST instrument was placed on a lab bench with operating stands near to a sink to facilitate loading samples, flushing and cleaning. Deionized water (DI water) was used for dilution and flushing of the LISST instrument prior to and between each sample, thus reducing the potential risk of contamination. Samples were diluted by a factor of two using DI water and manually mixed before being processed in the LISST. Dilution of samples is necessary so that the sample can be accurately measured by the LISST, as concentration is a controlling factor of LISST limitation. PSA using the LISST was repeated three times for each 'run' sample collected at $t = 2, 4, 6, 8, 10$ min for a period of one minute. The particle size distributions reported in this research are the mean calculations of repeated PSA consecutive measurements of the same sample for ≈ 30 seconds in the LISST. The LISST standard operating procedures were followed (Sequoia, n.d.).

The main aim of the PSA procedure was to produce quality results. Objectives included:

- (1) keeping the environmental conditions consistent, rinse the measuring cell and optical windows using DI water prior to and between runs,
- (2) visually inspecting the optical windows for signs of dirt, if any, and using pre-moistened lens wipes for clean-up,
- (3) collecting a background scattering measurement to verify that the LISST is functioning correctly and that the optics are calibrated to factory settings,
- (4) pouring sample into the LISST reservoir without generating bubbles (Gartner, et al., 2001).

Limitations due to sample preparation, handling and sampling in the LISST were considered to be minimal by the procedure listed above.

Equations for PSA distribution standard error bars:

Equation 3

$$\%RSD = \frac{s}{\bar{u}} * 100$$

s = standard deviation

\bar{u} = mean

%RSD = relative standard deviation

3.4 Statistical analysis

There are three hypotheses related to the sediment characteristics to be statistically analysed in this project as displayed in Figure 3 (*Research Objectives, 1.6*).

- The CFC has an efficient collection rate of SS
- OM values of collected SS are representative of original sample
- PS of collected SS is representative of original sample

3.3.1 Suspended sediment concentrations and organic matter

Statistical analysis was conducted using Minitab® 16 (2010). The SSC data included normally (parametric) and non-normally (nonparametric) distributed data assessed by the Anderson-Darling Test (*Appendix, Table 8*). A Box-Cox Transformation was used on the entire data set so that the transformation did not erase potential differences between the normal and non-normal data. Subsequently, a paired *t* test was used to analyse differences between the influent and effluent SSC data (*Results and Discussion, Table 3*).

Linear regressions were used to evaluate the effect of centrifugation on the SSC of the sample water through time to test specific research questions. All null hypotheses and *p*-values were assessed at an α level of 0.05. The ggplot2 package (Wickham, 2009) for the statistical software R (R.3.0.1, 2013) was used to produce Figure 12 whilst all other plots were produced using Excel (Microsoft, 2010).

3.3.2 Particle size

LISST data was processed using R (R.3.0.1, 2013). Replicates of each PSA water sub-sample were measured using the LISST three times and an average for these PS distributions was calculated in R. All mean PS distribution data was nonparametric as indicated using an Anderson-Darling Test in Minitab (2010). The PS distribution differences between influent and effluent samples was analysed using a Wilcoxon Signed Rank Test (Burt & Barber, 1996). Again, the *p*-values were assessed at an α level of 0.05.

4. Results and Discussion

4.1 Suspended sediment concentrations

The first hypothesis of this project was that continuous flow centrifugation (CFC) has an efficient collection rate of suspended sediment (SS). The lowest rate of efficiency recorded was by Ongley & Blatchford (1982) using the Alfa Laval, at 65-85 per cent, and the most efficient centrifugation systems were studied by Horowitz et al. (1989) at 92-99 per cent using both the Westphalia and Alfa Laval centrifuges. CFC has become a well-studied technique although it is unknown how transferrable these reported efficiency results are to other centrifuge or sediment research.

In contrast with the findings of CFC efficiency reported previously, efficiency results for sediment retention using the US M-512 centrifuge are markedly lower (Figure 12). There was a statistically significant difference of SSC in the samples prior to and after centrifugation ($p < 0.05$). Furthermore, the occurrence of SS in the effluent water indicates that the centrifuge is not collecting the total mass of sediment in the water.

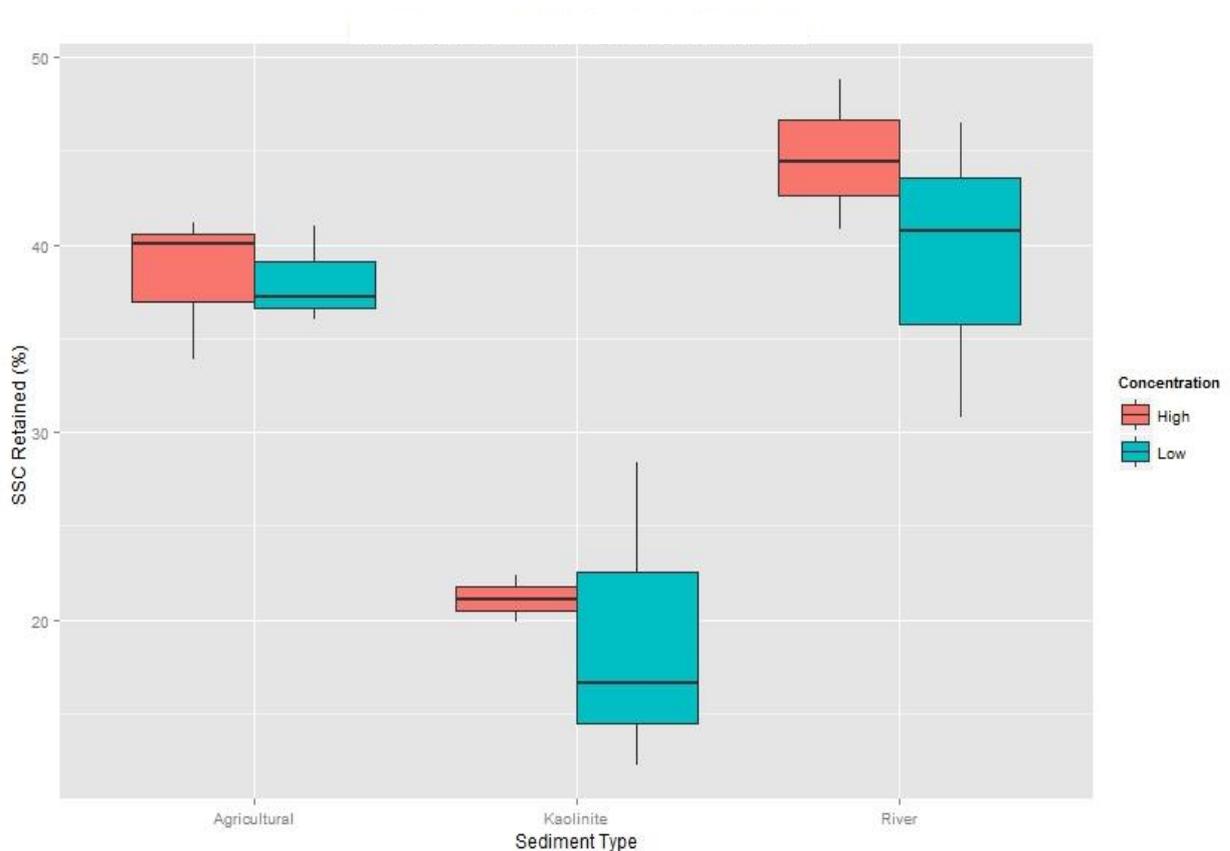


Figure 13: Percentage of SSC retained in the US M-512 centrifuge system bowl.

Figures 12 and 13 display the ineffective ability of the US M-512 centrifuge to collect the total SS in the water. Previous studies of CFC have not reported a complete retention of SS either. The US M-512 centrifuge was most effective at retaining the river sediment (*high concentration mean = 44 per cent, low concentration mean = 41 per cent*). Agricultural sediment was also retained by the centrifuge with similar efficiency (*high concentration mean = 40 per cent, low concentration mean = 37 per cent*), although the kaolinite recovery was even lower (*high concentration mean = 21 per cent, low concentration mean = 17 per cent*).

The mean retention values for all sediment types identified above indicate that the CFC instrument was more effective when sediment concentration was high ($> 15 \text{ mg l}^{-1}$). Although, when analysing retention efficiency Ongley & Blachford (1982) omitted data less than 5 mg l^{-1} due to inherent error in the subsequent sample processing that involved filtration. Furthermore, the river sediment high concentration values do not reach this project threshold (15 mg l^{-1}), due to challenges involving re-suspending river sediment in the reservoir. The low concentration river sediment is still significantly lower than the high concentration of river sediment; two-sample *t*-Test, $t = 10.14$, $df = 23$, $p < 0.001$. Due to the overlapping quartile ranges of retention efficiency for the high and low concentrations (Figure 12) and discounting the river sediment in accordance to Ongley & Blachford (1982), it cannot be determined whether the US M-512 centrifuge was more effective at higher concentrations.

The reported efficiency results for river and agricultural sediment have similar retention values, although, kaolinite sediment is an evident outlier from these (Figure 12). The characteristics of kaolin clay are well known, as it is the most abundant mineral in soils and sediments (Miranda-Trevino & Coles, 2003). Furthermore, the rationale for including kaolinite as control sediment in this study was due to a similar PS distribution to that of some soils and sediments (Coles, 1998; Miranda-Trevino & Coles, 2003). Kaolinite did not prove to be a representative control sediment for the SSC analysis. However, the PS distribution characteristics were valuable for *Particle size analysis*, Section 4.3. Kaolin clay is generally referred to as cohesive sediment that would theoretically promote aggregation when under centrifugation (Lin & Penumadu, 2010) this was not supported by the retention efficiency values. Therefore, the SS retention results indicate that laboratory kaolinite does not exhibit similar hydrodynamic behaviour to soils and sediments.

The SS retention results may be due to various factors, these include the structural design of the US M-512 centrifuge system that differs to those in previous studies and may not be as designed as well to effectively promote particulate retention. Moreover, the flow rate induced by the submersible pump was too great for the centrifuge. Reported flow rates used by studies, 4 lmin^{-1} (Ongley & Blatchford, 1982) and 6 lmin^{-1} (Rossé, et al., 2006) are considerably lower than the flow rate used in the US M-512 centrifuge set-up ($93.6 \pm 0.52 \text{ lmin}^{-1}$). The centrifuge system may have become more efficient through time of operation due to sediment in the bowl causing increased surface area for the promotion of sediment aggregation.

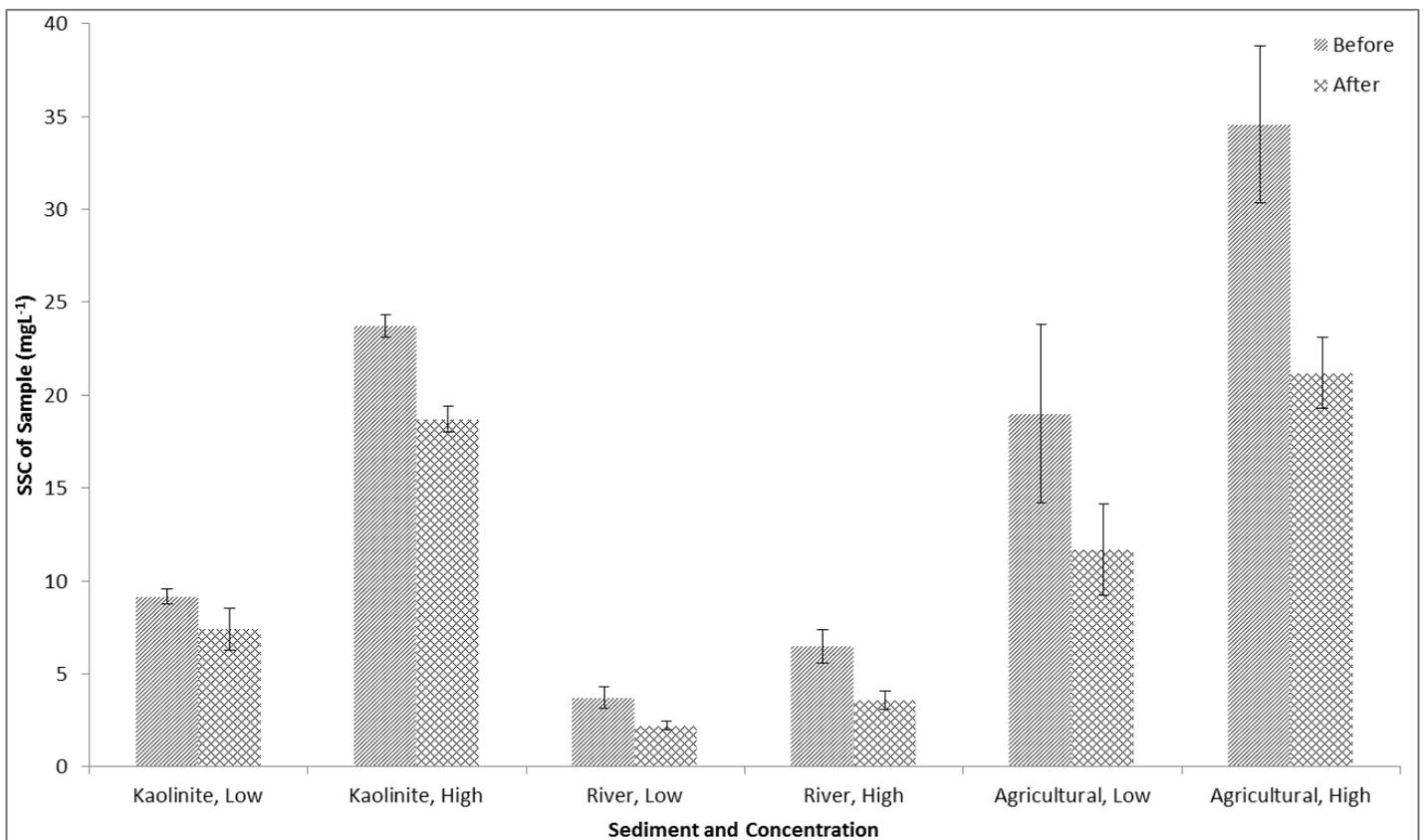


Figure 14: SSC (mgL^{-1}) concentration of sample water before (D of Figure 9) and after (F of Figure 9) CFC with standard deviation error bars.

The short sampling time frame of this laboratory experiment (10 min), compared to field investigations (sampling periods $<240 \text{ min}$ (Rossé, et al., 2006)) could have contributed to the poor sediment retention. SSC values of the effluent samples collected at $t = 2, 4, 6, 8, 10 \text{ min}$ were plotted against time (Figure 15). The high and low concentration runs were separated for graphical clarity.

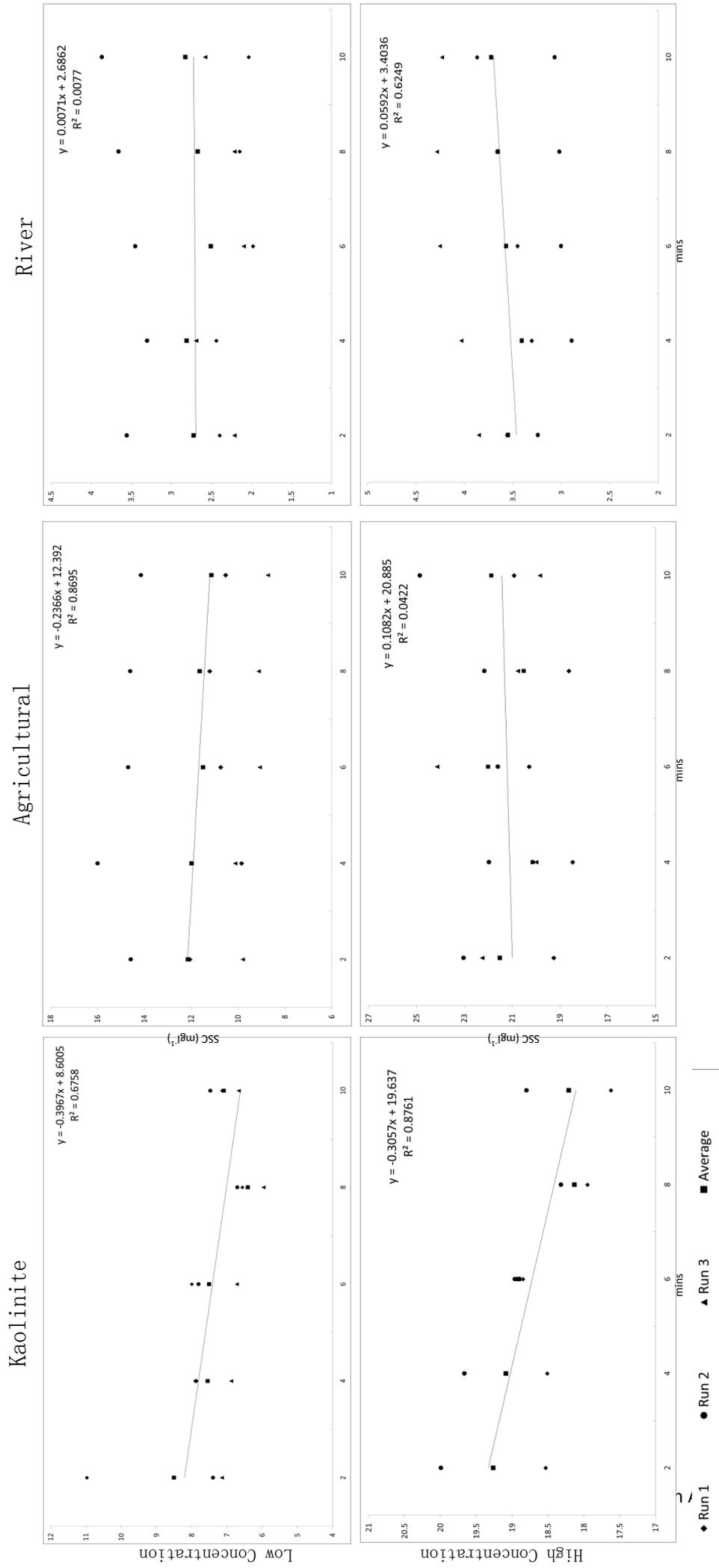


Figure 15: SSC (mg l⁻¹) plotted against time (min) through experiment including average values calculated, linear regression trend lines of averages and R² values.

River sediment concentrations did prove difficult to keep in suspension in the reservoir, consequently the high concentration results did not vary as greatly from the low concentration as the other sediment types (Figure 14 and 15). High and low concentration experiments using kaolinite experience a general decrease of SSC in the effluent water implying that the retention efficiency of the CFC was increasing throughout the experiment (low concentration average; $t_2 = 8.51 \text{ mg l}^{-1}$, $t_{10} = 7.1 \text{ mg l}^{-1}$, average Kaolinite high concentration; $t_2 = 19.26 \text{ mg l}^{-1}$, $t_{10} = 18.21 \text{ mg l}^{-1}$). In contrast, agricultural sediment mean SSC of high and low concentration trends do not correlate from t_2 to t_{10} (high concentration average; $t_2 = 21.51 \text{ mg l}^{-1}$, $t_{10} = 21.87$, low concentration average; $t_2 = 12.15 \text{ mg l}^{-1}$, $t_{10} = 11.13 \text{ mg l}^{-1}$). SSC measured through time for river sediment does not indicate any significant change in retention of sediment in the centrifuge bowl either (high concentration average; $t_2 = 3.55 \text{ mg l}^{-1}$, $t_{10} = 3.72 \text{ mg l}^{-1}$, low concentration average; $t_2 = 2.72 \text{ mg l}^{-1}$, $t_{10} = 2.83 \text{ mg l}^{-1}$). No general trend is evident for sediment retention within the centrifuge through time. It can be deduced that the efficiency rates recorded for the US M-512 centrifuge are not a result of the sampling time frame.

4.2 Organic matter

A poor retention efficiency of SS by the US M-512 centrifuge does not determine whether the instrument collects a representative sample. Consequently, the OM content of the samples was prepared using LOI as described in the *Methodology*, section 3.2. The effluent OM content was not significantly less than the influent OM content of most sediment and concentration types ($p > 0.05$), with the exception of high concentration agricultural sediment (Table 3). The percentage of OM content in the influent and effluent water is shown for each experiment (Figure 16). The OM content varies between sediment types as the sources of sediment OM characteristics are inherently different, a decision made for this study mentioned in the *Experimental Considerations*, section 5. The effect of CFC on the OM content of the sediments was not consistent.

Figure 16 displays very little variation of OM content of kaolinite. This was an expected result as it is a clay mineral (Miranda-Trevino & Coles, 2003). The high concentration agricultural sediment experiences a small decrease of OM due to centrifugation in each experiment ($p = 0.037$) (Table 3).

Low Concentration

High Concentration

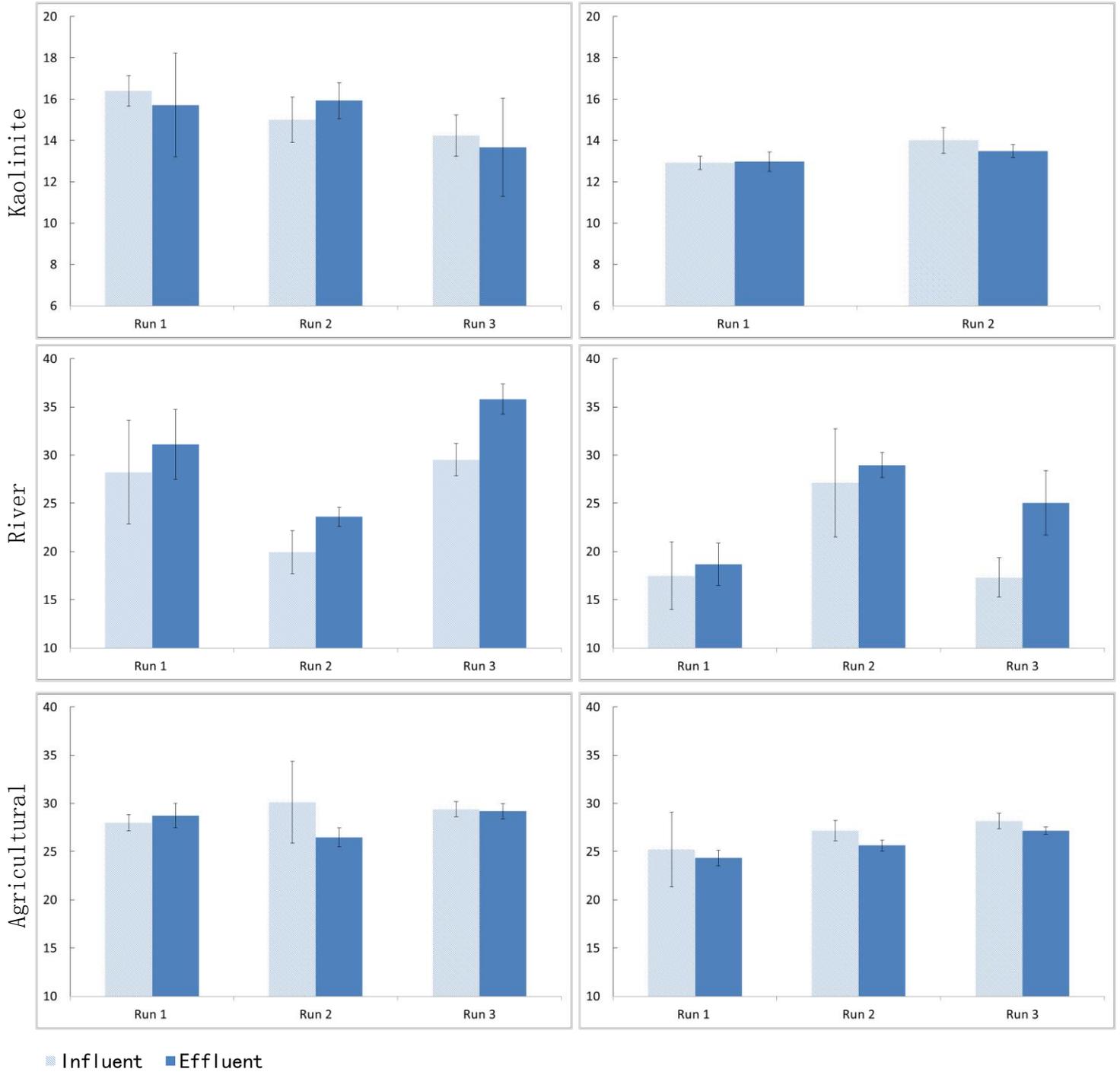


Figure 16: Mean OM content (%) and standard deviation error of influent and effluent water for each run of the sediment types.

Table 3: Paired *t*-Test *p* value results for the OM content (%) effluent is 'less than' the influent water samples.

Sediment	Concentration	<i>p</i> - value
Kaolinite	Low	0.435
	High	0.183
River	Low	1.00
	High	0.99
Agricultural	Low	0.098
	High	0.037

In contrast to the slight reduction of OM in agricultural sediment, the river sediment had an increase of OM for all runs (Figure 16) and is reinforced by the rejection of the statistical test that the effluent OM is less than the influent (Table 3). To examine the increase further and remove potential bias with the standard error bars in Figure 16, the river sediment OM content was tested using a paired *t*-Test with the hypothesis that the effluent OM was greater than the influent water. The results, low ($p < 0.001$) and high ($p = 0.010$) concentrations both supported the hypothesis that OM content increased after centrifugation. Therefore CFC has not retained a representative sediment sample in terms of OM for river sediment.

OM is a major component of fine grained sediment (Figure 5) (Droppo, 2001) transported by rivers as described in section 2.3, *Literature Review*. It can be sourced from degraded vegetation debris and soil organic matter (SOM) (Eggert, et al., 2012; Hynes, 1975; Slack & Feltz, 1968). These upstream sources are transported and flocculate with mineral particles (Hedges, et al., 1986). The downstream transport of flocculated material with various components (Droppo, 2001) in river systems does not lend itself to representative sample collection using CFC. Ongley & Blatchford (1982) observed that their centrifuge, the Westfalia Alfa-Laval MAB102, had higher retention efficiency results for mineral-rich sediment than organic-rich sediment. It can be implied that it is the process of CFC that preferentially retains mineral rich sediment rather than the US M-512 centrifuge itself.

Figure 16 displays the OM content values for the repeated experiments that were undertaken in sequence. Additionally, Figure 15 plots the SSC values of the individual experiments in sequence. Both figures do not suggest that sediment loads increase after each experiment. In contrast, Horowitz et al. (1989) experienced an issue with CFC system components, observing an inability to clean the centrifuge completely between sediment collection as a limitation of the instrument. Centrifuge efficiency could be artificially lowered for OM-rich water during filtration by “fibril matting” that causes retention of suspended organic material that would normally pass through the membrane filter (Comba & Kaiser, 1990).

4.3 Particle size analysis

It has been detailed that the centrifugation process can cause fragmentation of larger particles that result in a fining of the effective particle size (Rossé, et al., 2006; Rostad, et al., 1998). Due to this fragmentation of sediment and the importance of PS in sediment study as mentioned in the section 2.3, *Sediment particle size, Literature Review*, particle size analysis (PSA) was conducted to evaluate the effects of CFC on sample sediment. The general response of the sediments to the centrifuge technique in this study was an increase in the fine particle fraction (< 63 µm) as highlighted in Figures 17, 18 and 19 and Table 5. PSA indicated that the centrifuge was disaggregating naturally occurring flocs into their constituent primary particulates (Droppo, 2001; Rees, et al., 1991). The hypothesis that the PS of the collected SS is representative of the original sample is not conclusively supported by the results.

The LISST instrument does not output an exact mean diameter value as the instrument divides particles into 32 logarithmical size classes (Gartner, et al., 2001). The modal particle size classes for the particle size distributions are listed in Table 4. Most of the sediment and concentrations types experience a decrease in the modal PS class after centrifugation, except for low concentration river sediment and high concentration agricultural sediment, when the modal class remained the same (Table 4). The volume of particles in the finer-fraction (< 63 µm) of the influent and effluent PS distributions are listed in Table 5. A comparison of the particle volume below 63 µm of the CFC influent and effluent (Table 5) shows that there is an increase in the fine-fraction particle volume for all sediment and concentration types after centrifugation. The increase is coherent with conclusions from preceding centrifugation effects on particle size research (Horowitz, et al., 1989; Rees, et al., 1991; Rossé, et al., 2006; Rostad, et al., 1998).

The influent and effluent PS distributions were statistically different for the majority of the experiments except for high concentrations of kaolinite and river sediment (Table 6). This further supported the results that demonstrated centrifugation had an effect on the sediment and supports the conclusions of centrifugation impacts on particle size. In contrast with the statistical differences, the amount of increase in particle volume below 63 μm is less consistent (Table 5). The increase in volume is minor, for example the particle volume for high concentration of kaolinite and agricultural sediment only increased by 2 per cent and 4 per cent, respectively. The largest fine-particle size volume increase is 11 per cent, for low concentration kaolinite sediment.

Table 4: Particle size (PS) class with the largest volume of particles (%).

Sediment	Concentration	Influent modal PS class (μm)	Effluent modal PS class (μm)
Kaolinite	Low	32.5	27.6
	High	27.6	23.4
River	Low	32.5	32.5
	High	74.5	53.5
Agricultural	Low	32.5	23.4
	High	38.4	38.4

Figures 17, 18 and 19 display the particle size distribution as a function of volume particles. The shaded boxes on the figures highlight the most chemically active fraction of suspended particles that acts as a vector for the transport of many contaminants and nutrients (*Literature Review, Section 2*).

The kaolinite high and low concentrations PS have similar distribution characteristics to one another (Figure 17 (a) and (b)). The influent and effluent PS had a greater difference for the low concentration ($p < 0.001$) than the high concentration ($p = 0.063$) but both concentration types experience a mean increase of particles below 63 μm (Table 5).

The effluent high concentration river PS does not vary significantly from the influent ($p = 0.071$) and the PS distribution displayed in Figure 18 (a) has a bimodal distribution. This distribution is not similar to the low concentration river sediment and does not demonstrate the same distribution trend as any other experiments. The simple explanation for this variation would be the extra effort that was made to keep the river sediment in suspension in the reservoir. This limitation is further mentioned in section 5, *Experimental Considerations*. There is a 5 per cent increase in the $< 63 \mu\text{m}$ fraction for high concentration river sediment although the error bars on Figure 18 (a) are ≈ 2 per cent for each PS class above 32.5 μm .

The centrifuge instrument caused the low concentration river sediment influent and effluent particle size distribution to vary significantly ($p = 0.009$). Figure 18 (b) shows that the mean PS volume between 63 μm and 237 μm decreased after centrifugation whilst the mean PS volume between 23.4 μm and 45.3 μm increased. This could possibly indicate that centrifugation was causing particle fragmentation (Rossé, et al., 2006) or that the centrifuge was preferentially retaining the larger particle fraction.

Table 5: Volume of particles below 63 μm as graphically displayed in the particle size distribution figures.

Sediment	Concentration	Influent volume (%) < 63 μm	Effluent volume (%) < 63 μm	Volume (%) change
Kaolinite	Low	86	97	11
	High	79	81	2
River	Low	68	74	6
	High	46	51	5
Agricultural	Low	81	85	4
	High	78	88	10

High concentration (Figure 19 (a)) and low concentration (Figure 19 (b)) form complimentary particle size distributions to one another, whilst both concentration types experienced a significant difference between the influent and effluent ($p < 0.001$). The centrifuge effluent particle volume is less than the influent above 53.5 μm and up to 144 μm . The particle volume in the fine-fraction increased by 10 per cent and 4 per cent for the high and low concentrations, respectively (Table 5). The fining trend of PS for the agricultural sediment can be observed clearly in Figure 19.

Table 6: Wilcoxon Signed Rank Test for the nonparametric particle size distribution data. The null hypothesis, influent and effluent PS distributions are 'not equal' was tested.

Sediment	Concentration	p - value
Kaolinite	Low	<0.001
	High	0.063
River	Low	0.009
	High	0.071
Agricultural	Low	<0.001
	High	<0.001

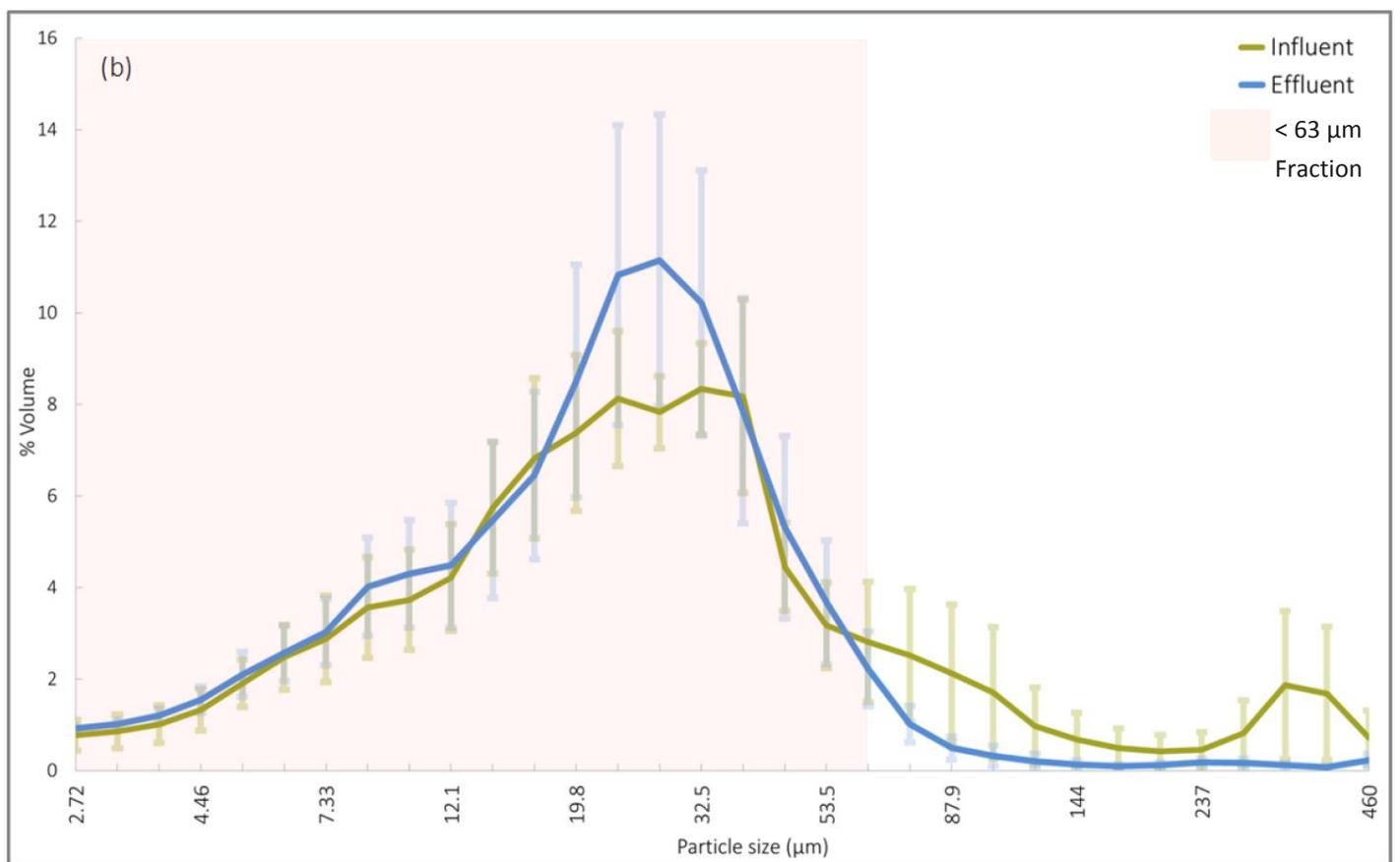
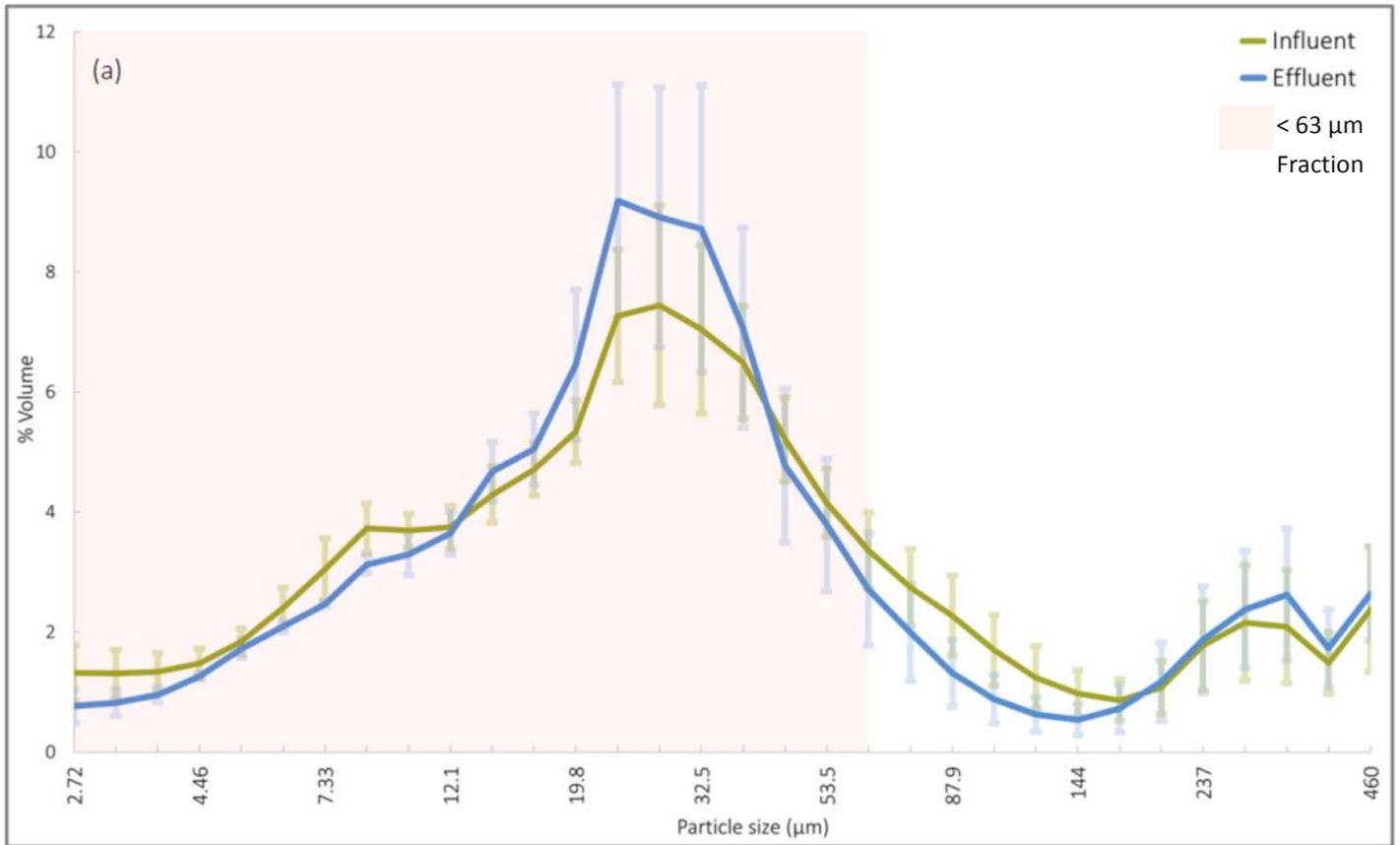


Figure 17: Particle size distribution for (a) high concentrations and (b) low concentration of kaolinite sediment.

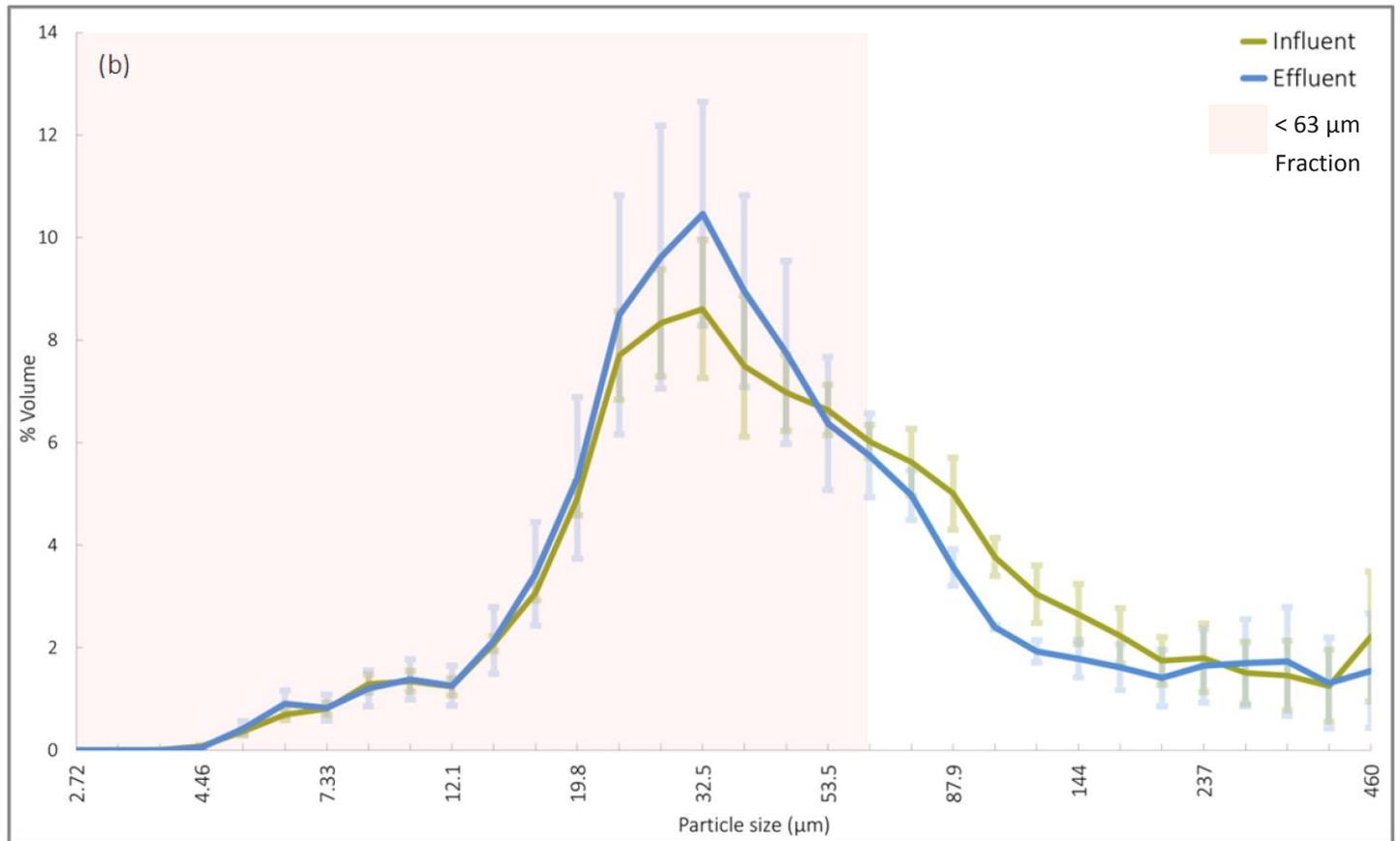
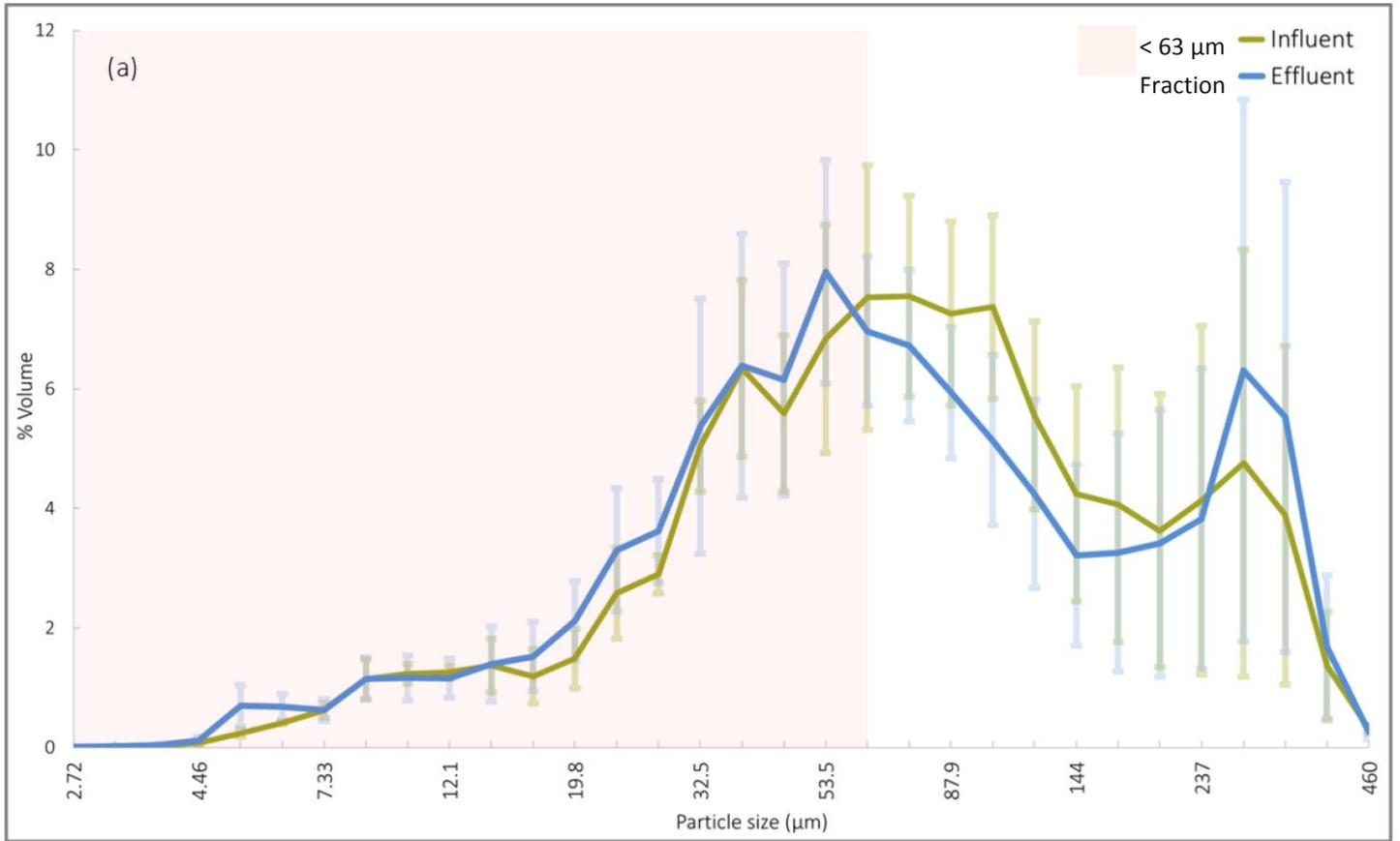


Figure 18: Particle size distribution for (a) high concentrations and (b) low concentration of river sediment.

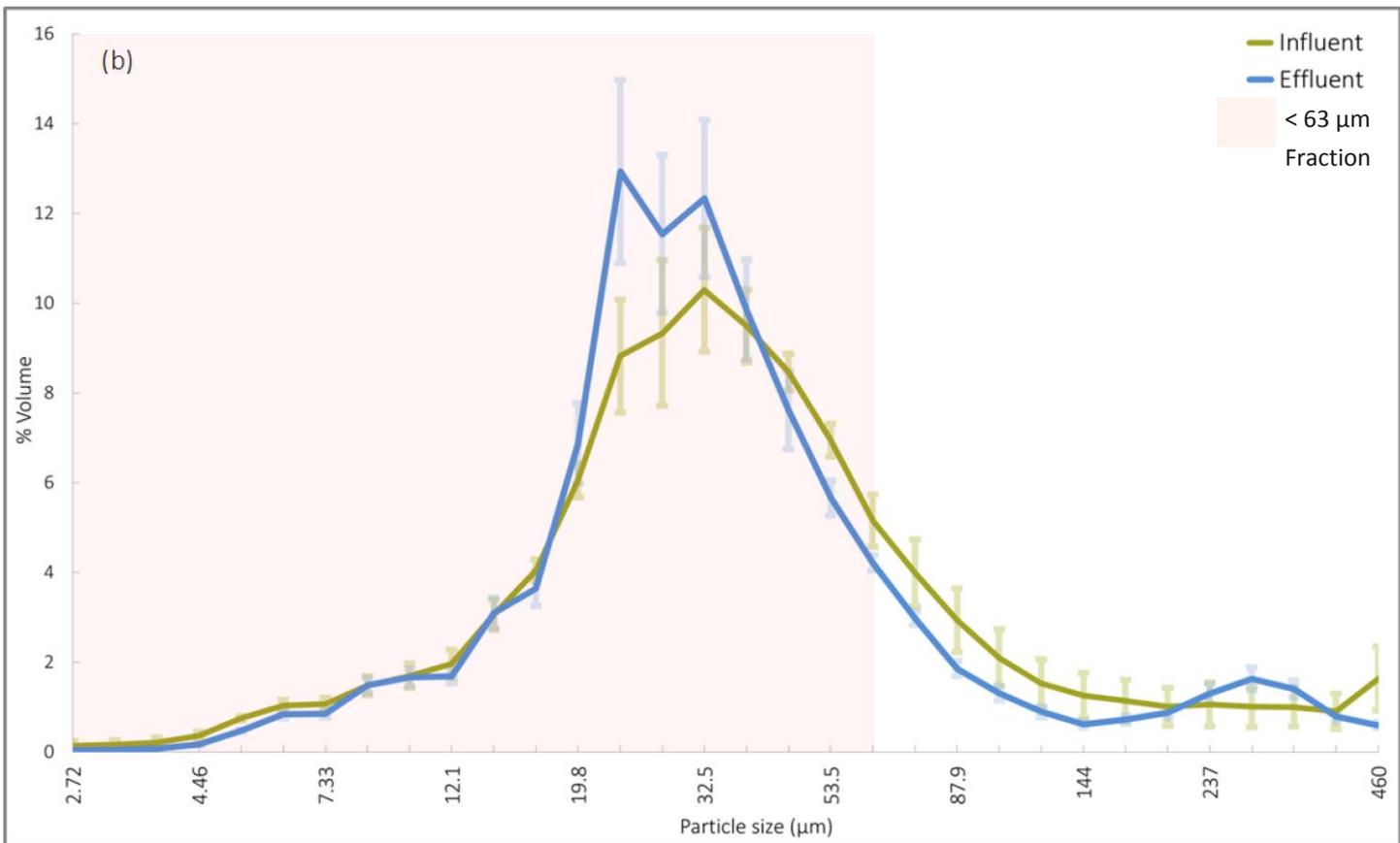
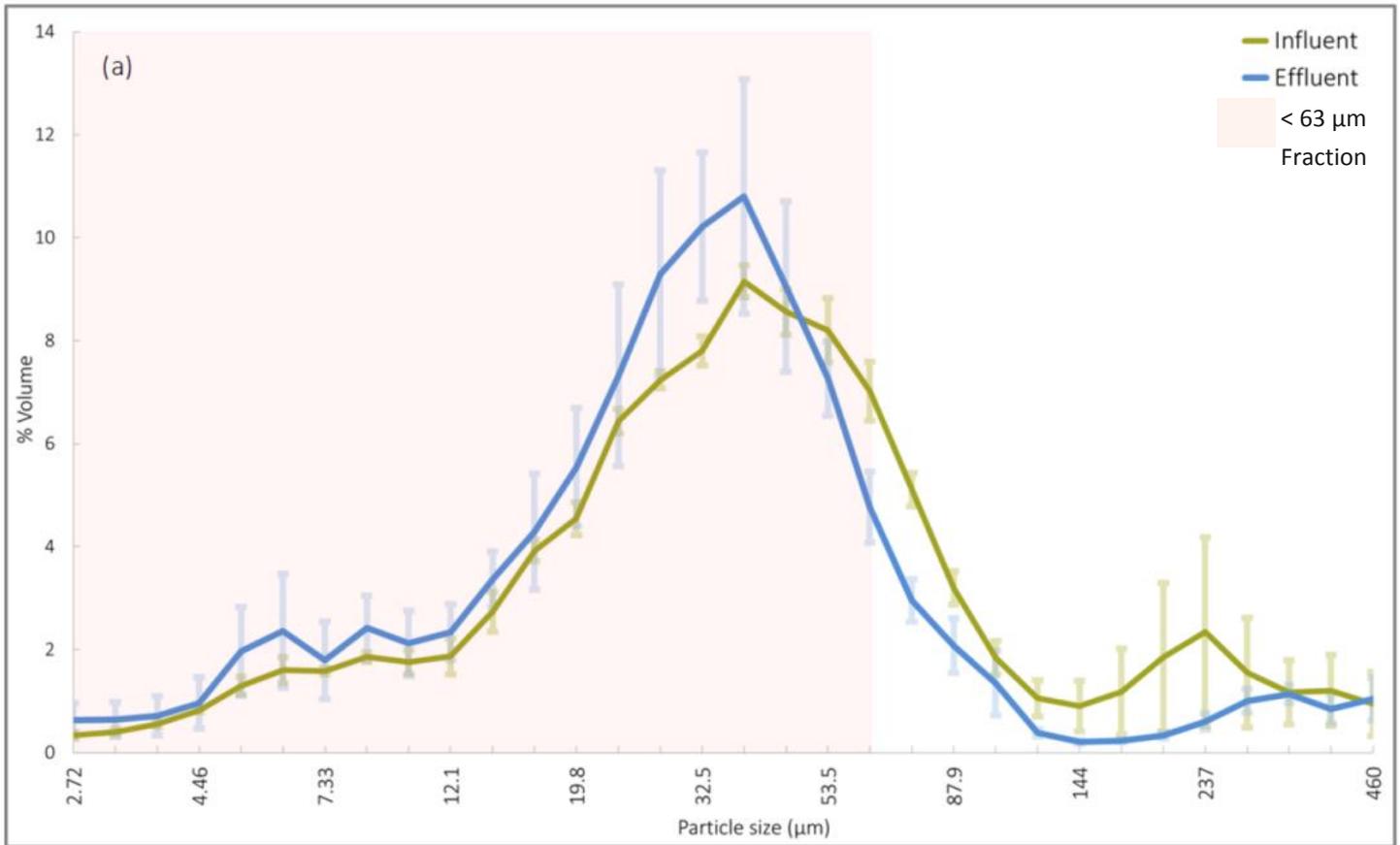


Figure 19: Particle size distribution for (a) high concentrations and (b) low concentration of agricultural sediment.

5. Experimental Considerations

Throughout this study possible experiment bias has been considered where appropriate. This section is intended to provide an evaluation of the bias within the experimental design and mentions adaptive strategies taken in unforeseeable circumstances.

The experiment was artificially structured: three sediment types at known concentrations were pumped through the centrifuge to ensure a controlled and repeatable methodology. Moody & Meade (1994) have already evaluated the effect of pumping sample water to a centrifuge. Additionally the sampling location 'D' (Figure 10, *Methodology*) reduced any adverse effects caused by the pumping action. Therefore, the experiment was focused on evaluating CFC, not on the pumping of water.

Despite the natural variability of suspended sediment characteristics and quantities in nature, the sediment types used in this project were identified specifically to vary from each other and still represent the natural environment.

After CFC, sediment is removed from the centrifuge bowl as a water-sediment mixture. This removal process from the bowl and the subsequent analysis has associated analytical errors (Moody & Meade, 1994). Filtration of centrifuge collected sediment can incur problems, such as membrane clogging and surface coagulation. This method may result in an unrepresentative separation of the water and sediment mixture during filtration (Buffle, et al., 1992). Forstner (2004) has documented the appropriate methods that must be used for sediment sample preparation with regard to the study aims and objectives.

The limited reservoir dimensions (total capacity \approx 1600 l) and controlled experimental inputs (sediment and concentration) enabled a relatively short-term investigation of CFC when compared to centrifuge deployment in field-research, as noted in section 2.9, *Literature review*. Within the laboratory setting, sampling at two minute intervals for 10 minutes was the most effective and achievable time frame for sample collection, sample bottle change and additional re-suspension efforts within the reservoir. This method provides little information about CFC's ability to collect sediment during longer sampling periods that are sometimes necessary (Comba & Kaiser, 1990). It was observed in this study that there was no general change of CFC behaviour through this experiment (Figure 15). Moreover, Rossé et al., (2006) studied possible temporal trends of contamination of collected sediment from centrifuge components indicating that researchers recognise CFC is not a consistent technique, although their results demonstrated no clear trend.

During the experiment procedure it became apparent that the fluvial sediment proved more difficult to maintain in suspension in the reservoir. Additional effort was made by the researchers to sustain more turbulent conditions within the reservoir to keep the river sediment in suspension for the 10 minute sampling periods.

6. Synopsis

Continuous flow centrifugation (CFC) is used in water studies for the bulk collection of suspended sediment (Ongley & Blatchford, 1982; Rossé, et al., 2006). Researchers, including those at the Quesnel River Research Centre (QRRC), continue to implement CFC systems to collect SS for the study of various sediment characteristics, although various studies have demonstrated issues associated with CFC (Moody & Meade, 1994). There is no current research for the US M-512 Manual centrifuge system, used at the QRRC, by independent parties. There is also a lack of recent research with a specific focus on the effects of centrifugation on sediment characteristics that are intrinsic for many sediment quality analysis procedures.

The retention efficiency results of the US M-512 centrifuge are markedly lower than previous studies of centrifugation as listed in section 2.9, *Continuous flow centrifugation, Literature Review*. The results of the efficiency study were 20 to 50 per cent lower than previous research studies. The US M-512 centrifuge efficiency was ≈ 42 per cent for agricultural and river sediment retention. The control sediment, Kaolinite, did not produce a comparable efficiency result (≈ 19 per cent). Kaolinite was included as a control sediment for OM and PS analysis as it is a clay mineral (Miranda-Trevino & Coles, 2003) and the particle size distribution is similar to that of some soils and sediments (Coles, 1998; Lin & Penumadu, 2010). The poor retention efficiency of the US M-512 centrifuge system promoted further study into previous centrifuge research. The induced flow rate by the submersible pump and the revolutions per minute (rpm) set-up for the US M-512 centrifuge varied greatly to other systems. The pump flow rate was much greater, $\approx 90 \text{ lmin}^{-1}$, and the rpm was one third of comparable CFC instruments (Table 8). The centrifuge system set-up will be considered and discussed at the QRRC by the researchers who have the opportunity to improve the instruments collection of suspended sediment as a result of this study.

The effect of centrifugation on the OM content did not demonstrate a general trend for the three sediment types (Figure 16). The kaolinite sediment did not have a statistical decrease after centrifugation (Table 3) proving its usefulness as a control sediment. The OM content of the agricultural sediment did decrease significantly in the low concentration experiments but not in the high concentration, although the standard error bars for each experiment do not establish confidence in these results. The effluent OM content for the river sediment was significantly greater than the effluent for both high ($p = 0.010$) and low ($p < 0.001$) concentrations. This result supported research by Ongley & Blachford (1982) that demonstrated poor retention of organic-rich river sediment by centrifugation.

PSA indicated that the mean particle volume below $63 \mu\text{m}$ of the centrifuge effluent increased for all of the experiments (Figures 17, 18 and 19 and Table 5). The fining of the SS in the CFC effluent supports the research of centrifugation by Rees et al. (1991), Horowitz et al. (1989) and Rossé et al. (2006).

7. Conclusion

In conclusion, the SSC and OM data suggests that the US M-512 centrifuge system does not collect particulate matter as efficiently as the studies using different manufacturer's systems report. The high flow rate induced through the M-512 centrifuge may negatively impact the instrument's ability to separate sediment by forcing the sample water through the instrument too forcefully. Additionally, the relatively low rpm of the M-512 does not create the same centrifugal force as other centrifuge systems do.

The preferential retention of mineral particles by centrifugation for certain sediment types clearly indicates that an understanding of the sediment characteristics of the sediment sampled is necessary to avoid distorted conclusions for OM of centrifuged SS. A combination of sampling techniques, for example implementing a re-suspension bucket technique, to collect a reference sample of sediment that has not undergone centrifugation can mitigate any false conclusions.

Furthermore, the PS data does not conclusively indicate a statistically significant difference of particle distributions in the influent and effluent sample water. A reduction in the larger fraction of the particle distribution is witnessed, confirming other researchers' results that state centrifugation causes fragmentation of larger particles is evident in this project. However, the results presented here suggest that the US M-512 centrifuge did not have a strong impact on the particle size distribution of sample water.

A suggestion for future study would be to adapt the US M-512 centrifuge set-up to include a slower pump flow rate. In addition, this study indicates it is often a necessity for the assessment of instruments in current use for research regardless of well-documented use, as the results of previous literature were not directly transferrable to the US M-512 centrifuge.

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9. Appendix

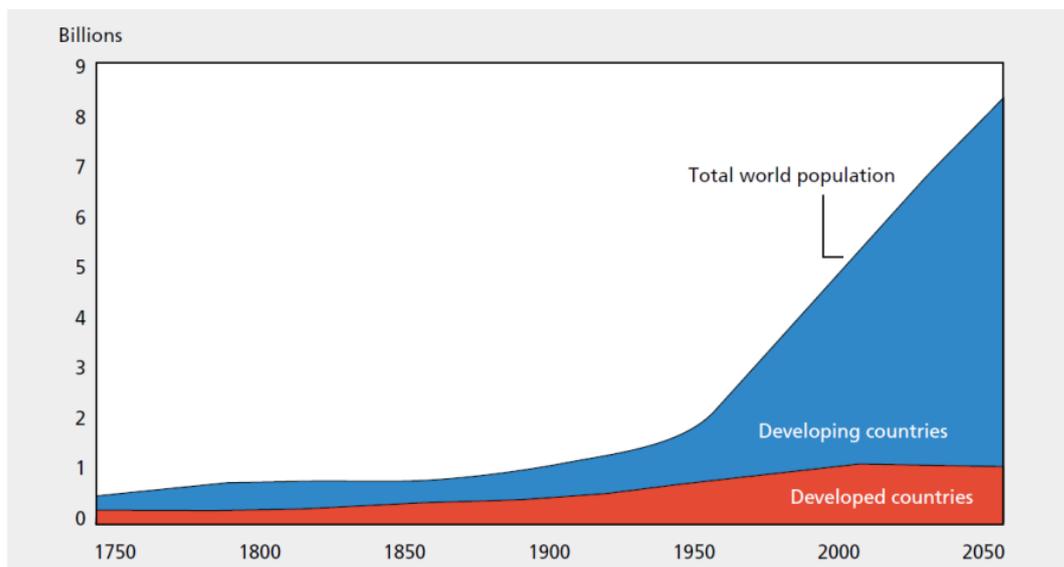


Figure 20: World population trend since 1750 and expected population dynamics until 2050 (Worldbank, n.d.).

Table 7: Contributing factors to an eventual downturn of anthropogenic influenced contamination of fluvial environments. Adapted from Selden & Song (1994).

Socio-economic factors	
1	Positive income elasticities for environmental quality
2	Changes in composition of production and consumption
3	Increasing levels of education and environmental awareness
4	More open political and connected governmental (integrated) systems

Table 8: CFC instruments and their reported rpm values in literature.

Researchers	Instrument	Reported rpm
(Comba & Kaiser, 1990)	Westfalia	9,800
(Moody & Meade, 1994)	Sharples Model AS-12	16,000
(Ongley & Blatchford, 1982)	Westfalia MAB102	n/a
(Rees, et al., 1991)	Sharples Model AS-12	16,000
(Rossé, et al., 2006)	Westfalia KA2-06-075	9,700
(Rostad, et al., 1998)	Sharples AS-12V	16,000

Table 9: Output from paired *t*-Test of SSC, illustrating the Box-Cox Transformation to create comparable, normally distributed data.

Sediment	Concentration	Position	<i>p</i> value prior to Box-Cox process	Converted <i>p</i> value
Kaolinite	Low	Influent	0.915	0.931
		Effluent	0.006	0.05
	High	Influent	0.120	0.142
		Effluent	0.737	0.789
River	Low	Influent	0.215	0.248
		Effluent	0.873	0.966
	High	Influent	0.781	0.515
		Effluent	0.404	0.437
Agricultural	Low	Influent	0.033	0.093
		Effluent	0.039	0.089
	High	Influent	0.192	0.145
		Effluent	0.912	0.959

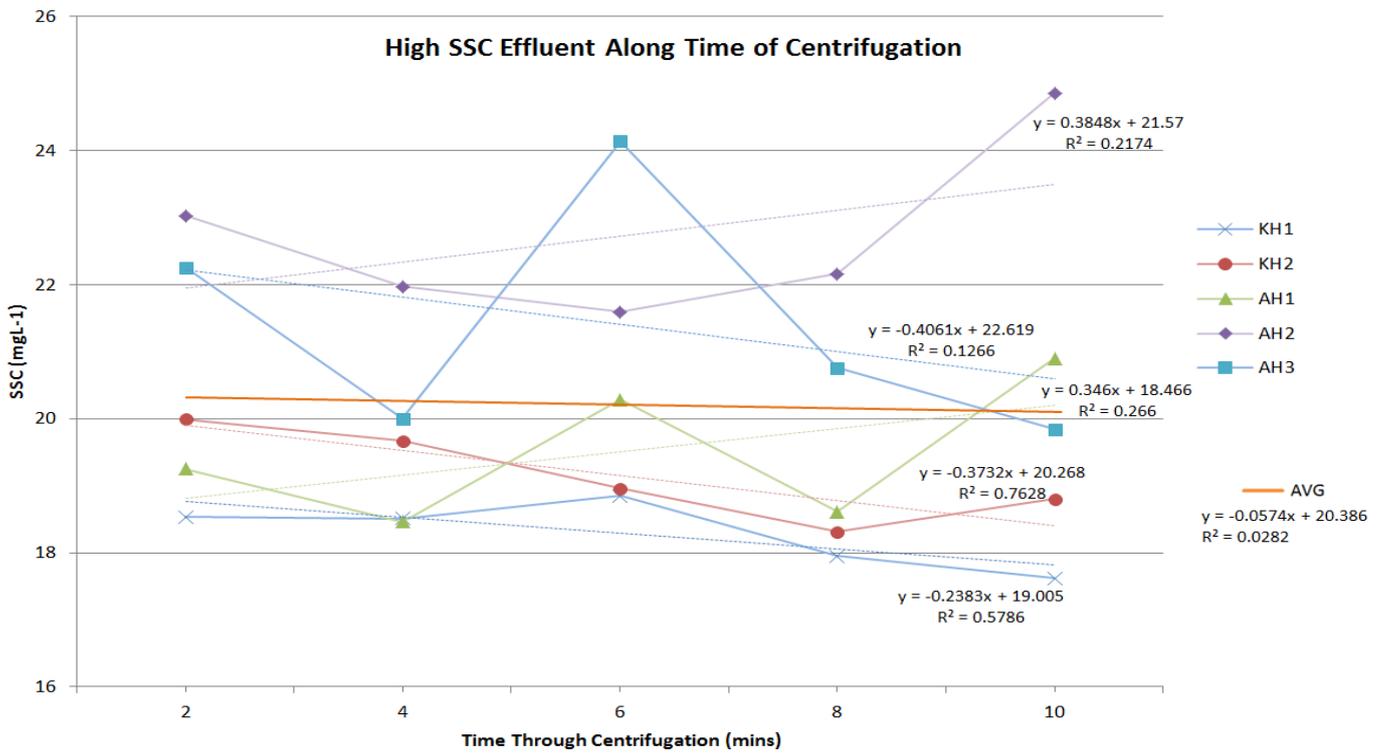


Figure 20: Change of effluent SSC (mgL-1) in high concentrations through sampling time.

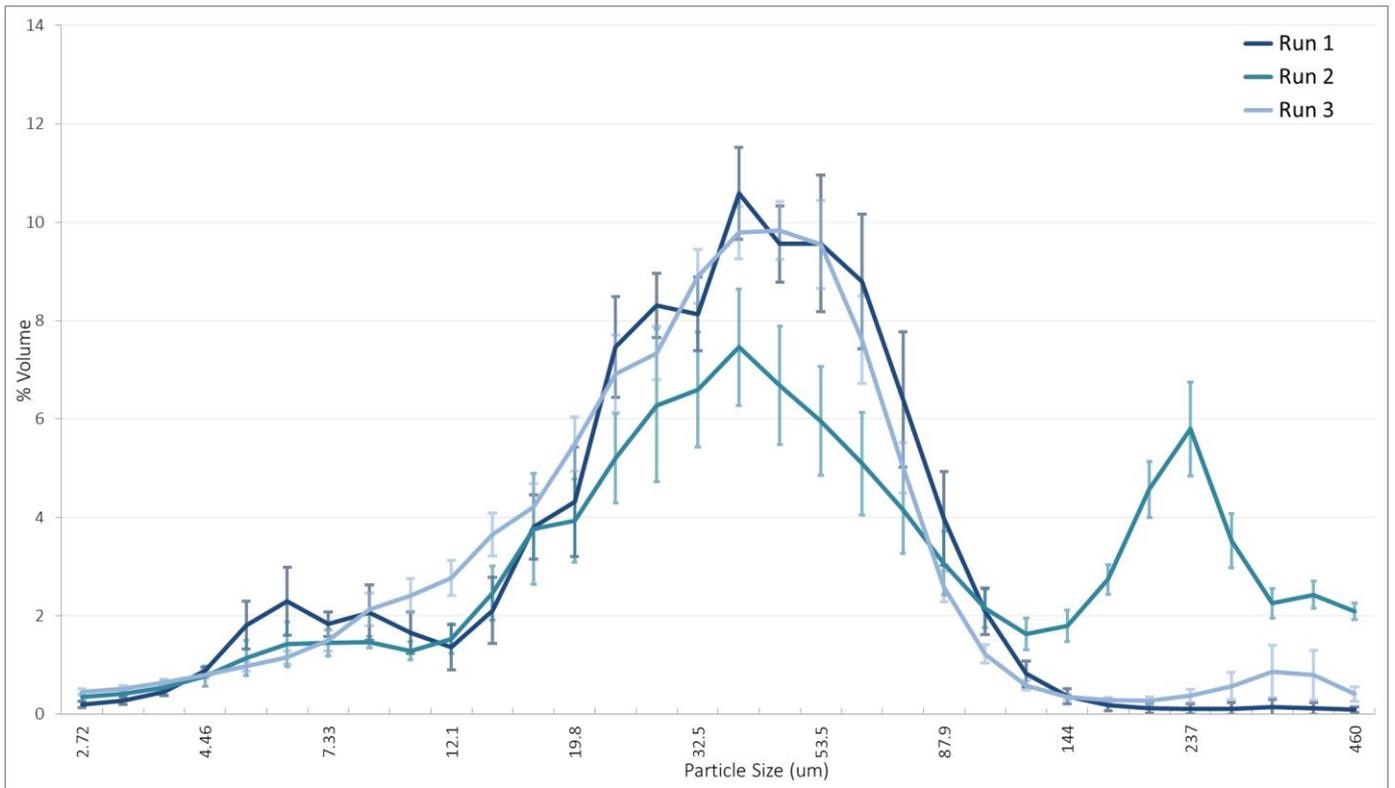


Figure 21: Agricultural high concentration run averages before centrifugation

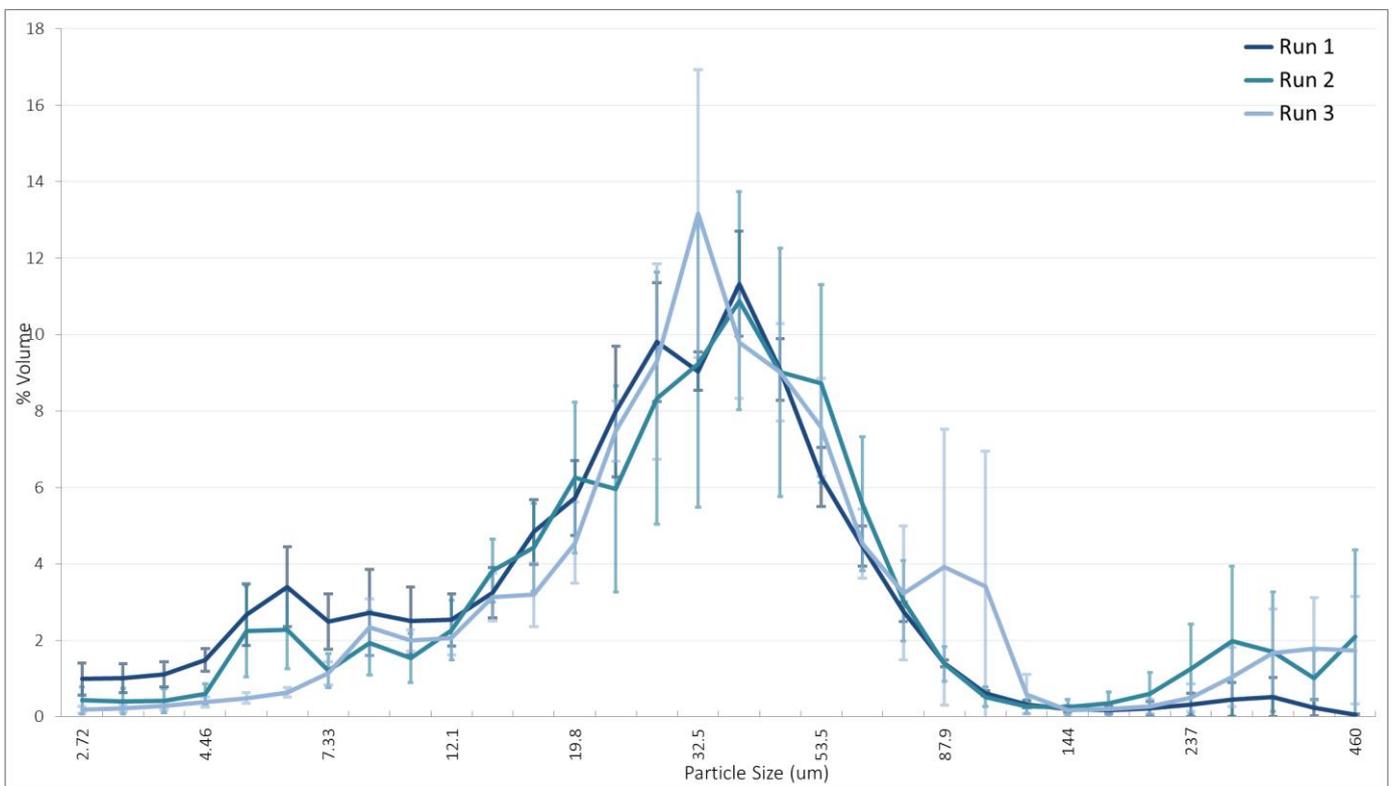


Figure 22: Agricultural high concentration run averages after centrifugation

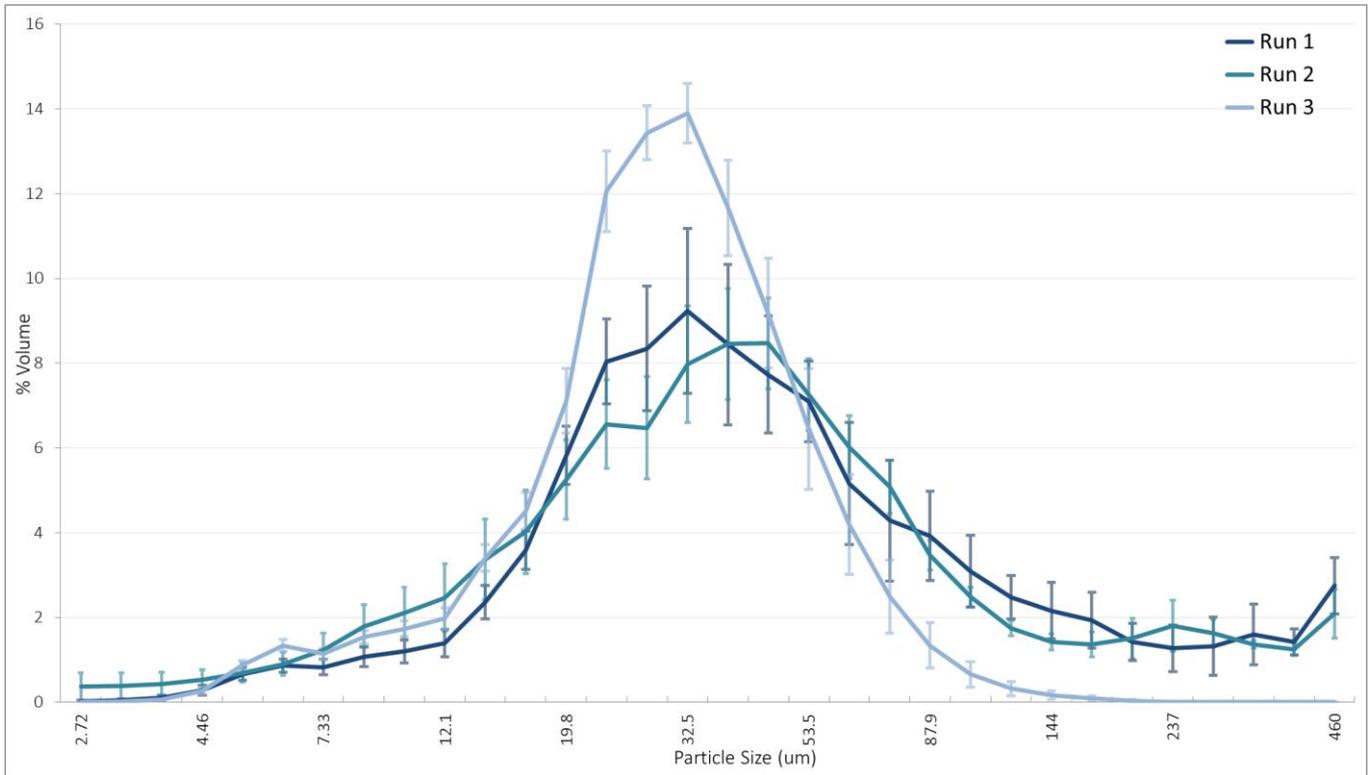


Figure 23: Agricultural low concentration run averages before centrifugation

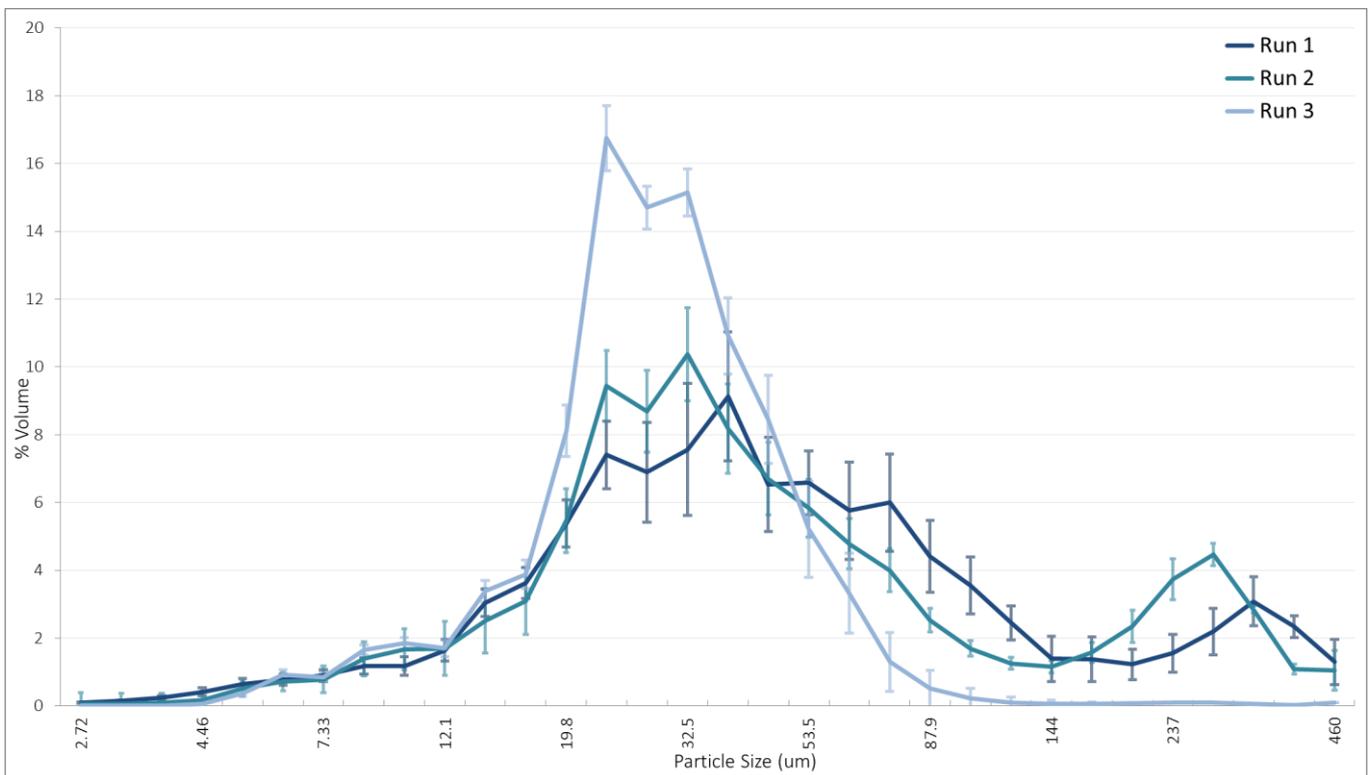


Figure 24: Agricultural low concentration run averages after centrifugation

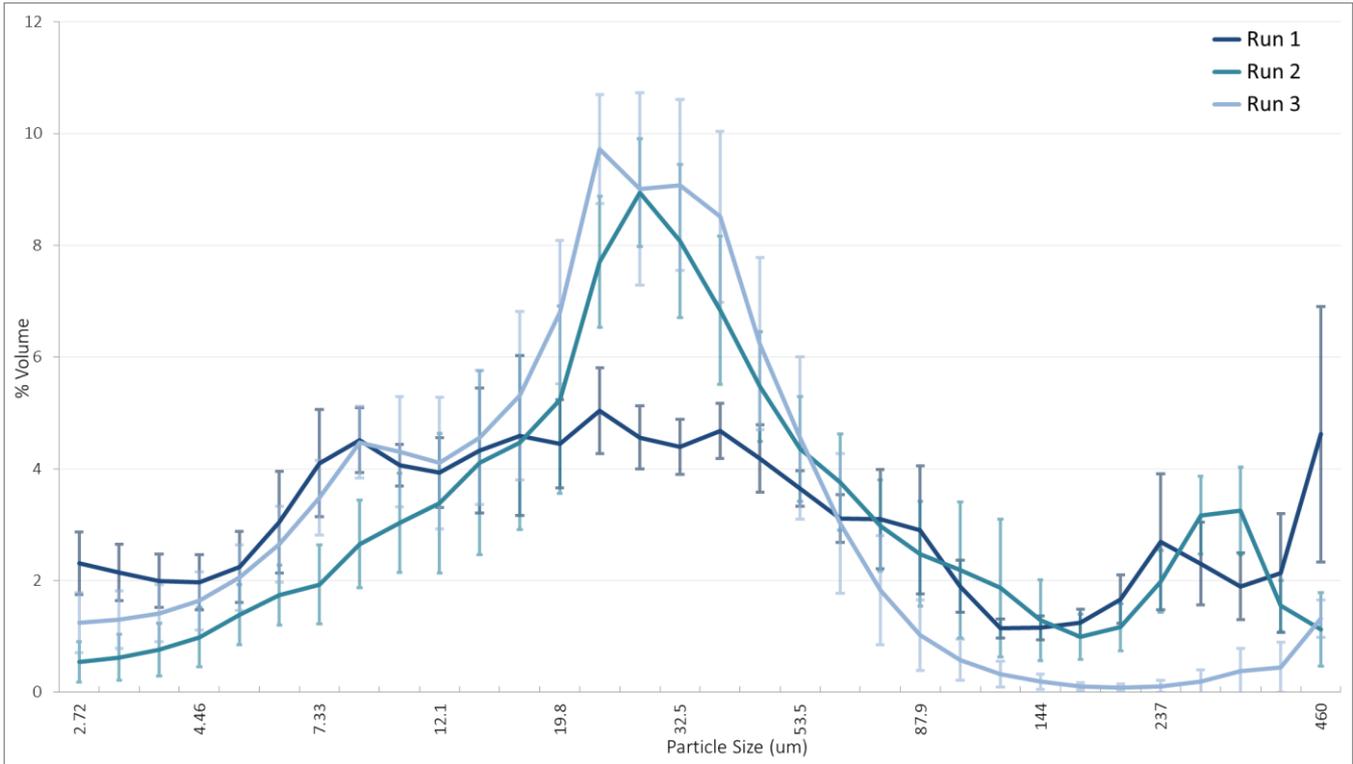


Figure 25: Kaolinite high concentration run averages before centrifugation

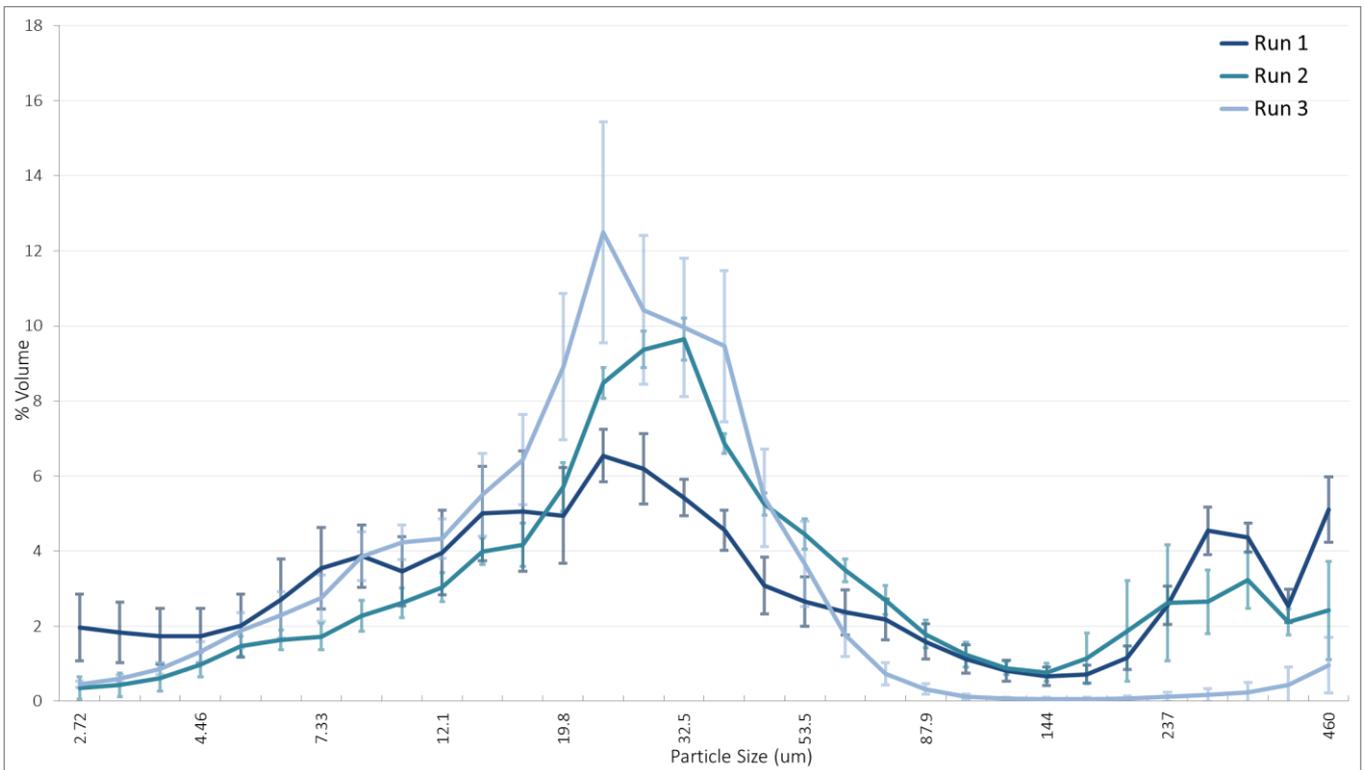


Figure 26: Kaolinite high concentration run averages after centrifugation

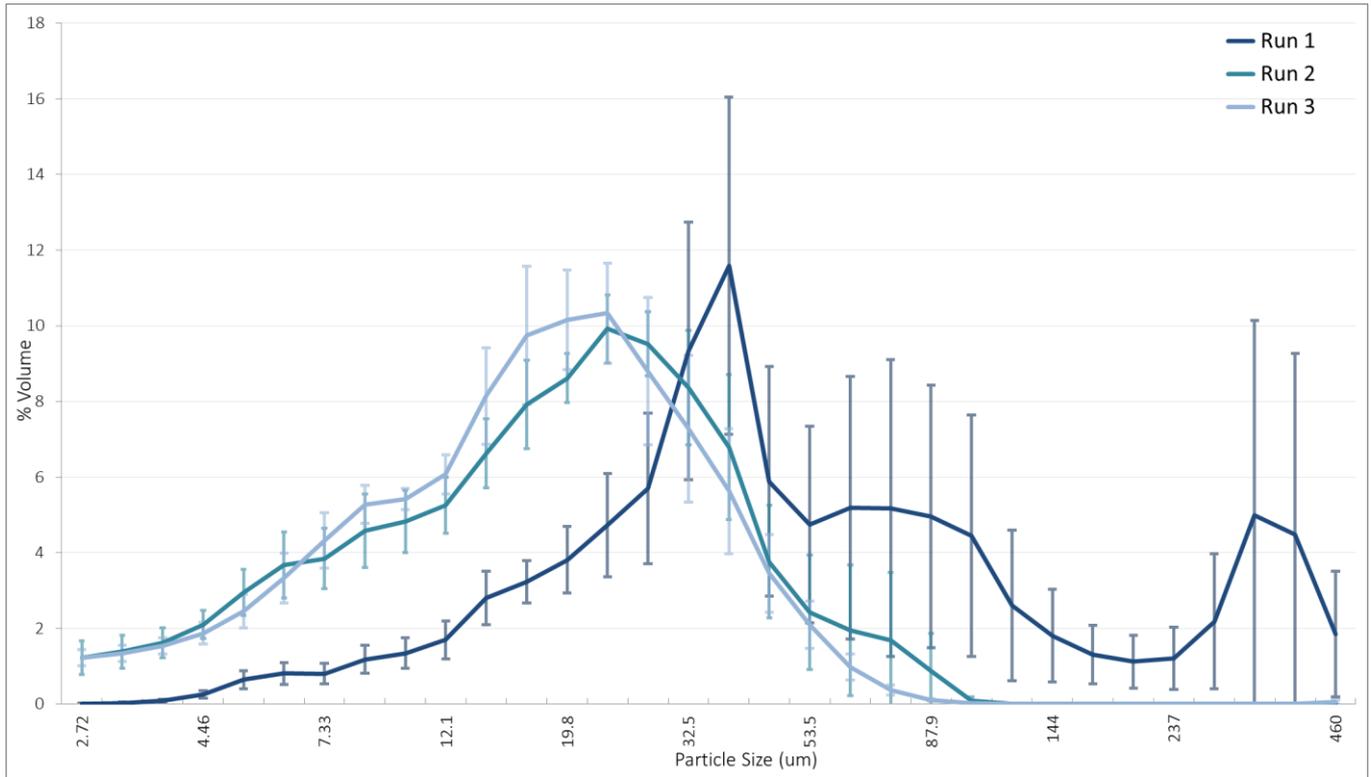


Figure 27: Kaolinite low concentration run averages before centrifugation

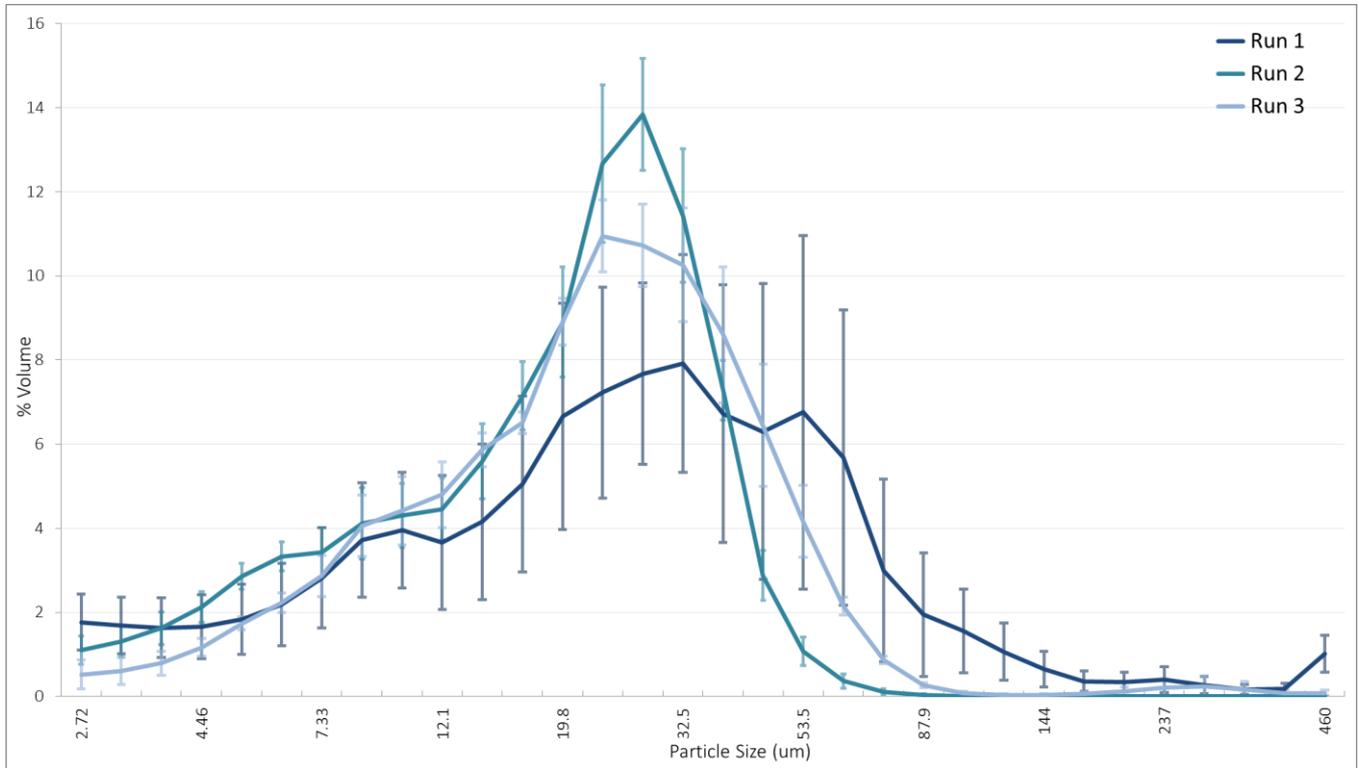


Figure 28: Kaolinite low concentration run averages after centrifugation

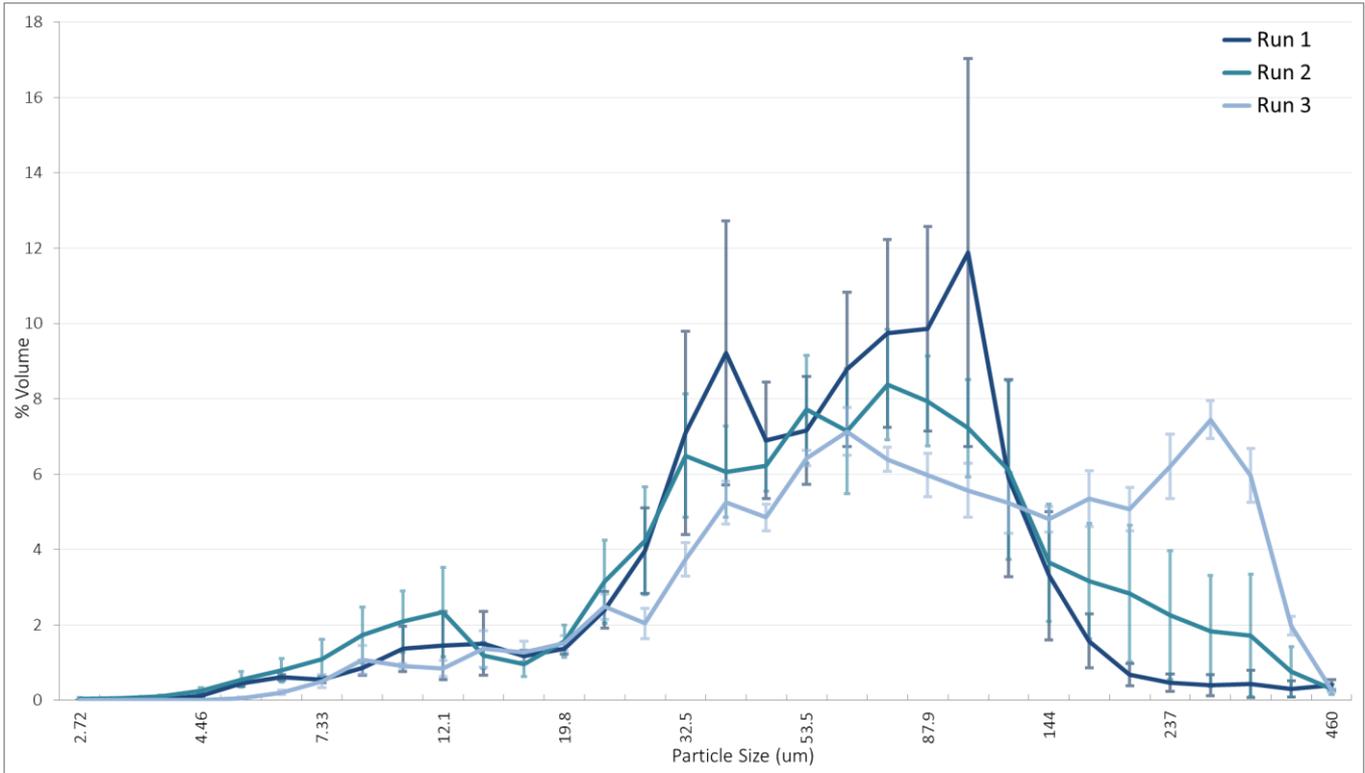


Figure 29: River high concentration averages before centrifugation

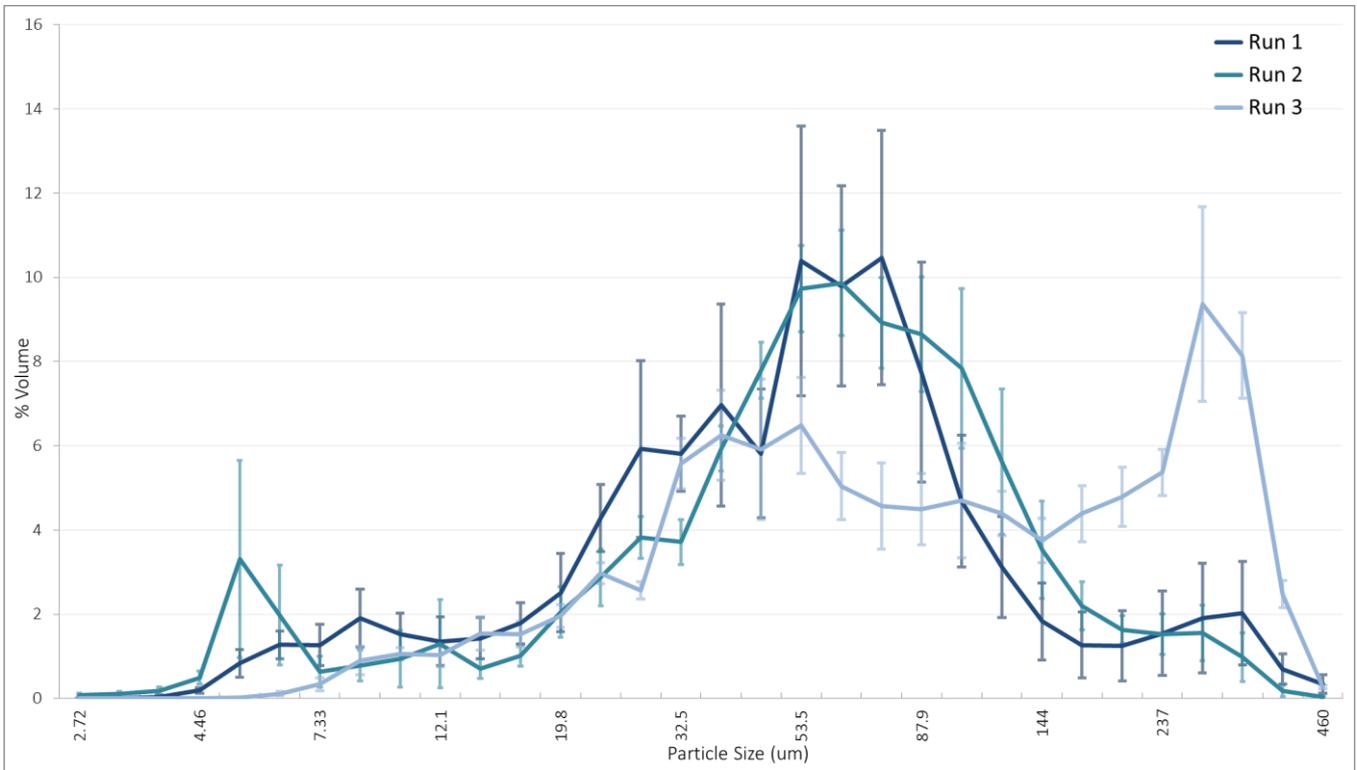


Figure 30: River high concentration averages after centrifugation

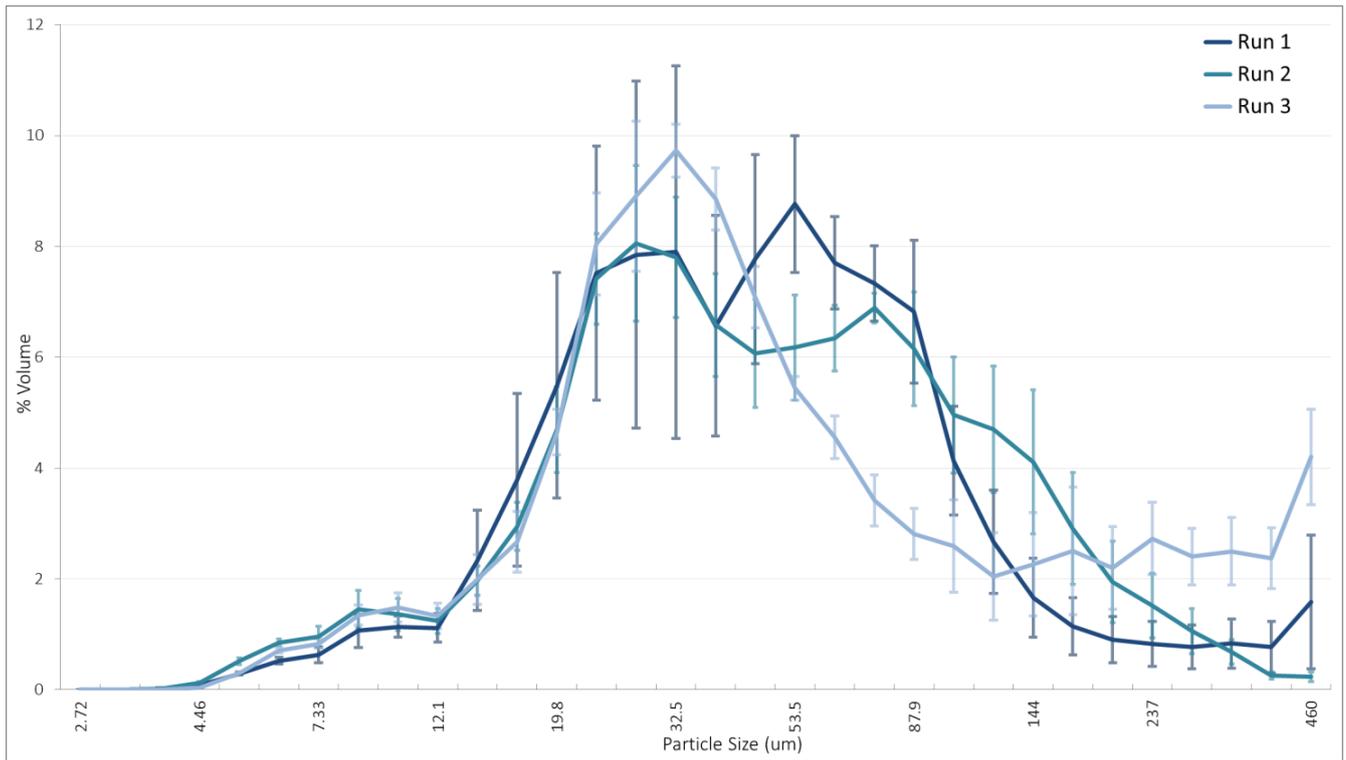


Figure 31: River low concentration run averages before centrifugation

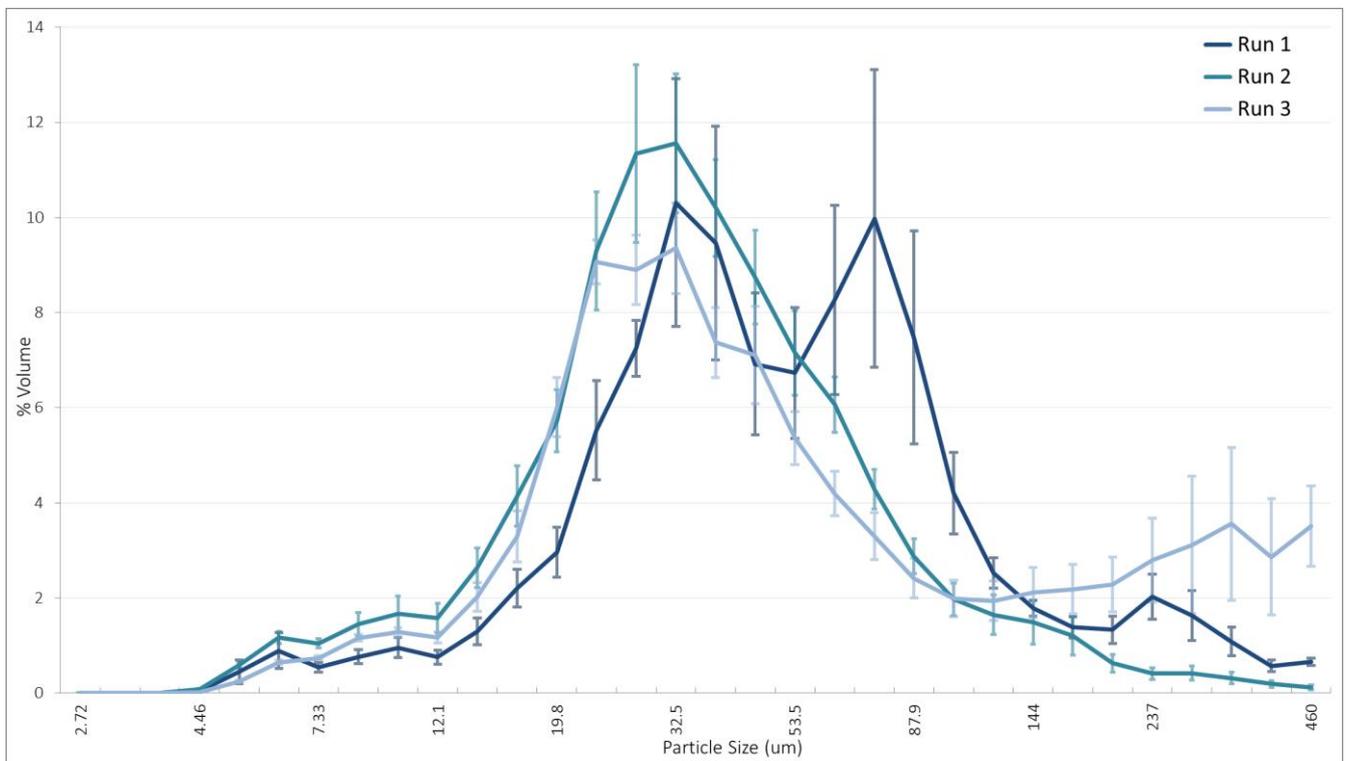


Figure 32: River low concentration run averages after centrifugation