Tertiary Wastewater Filtration: An Analysis of Crushed Recycled Glass as an Alternative to Sand Filter Media

by

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A Thesis submitted to the Faculty of Graduate Studies of
The University of Manitoba
in partial fulfilment of the requirements of the degree of

MASTER OF SCIENCE

Department of Biosystems Engineering

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Winnipeg

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ABSTRACT

This study evaluated the performance of sand (ES: 0.17 mm, UC: 3.13) and crushed recycled glass (ES: 3.59 mm, UC: 4.18) in pilot-scale subsurface filters treating municipal wastewater. The system provided on-site treatment for secondary lagoon effluents from the Village of Dunnottar, on the southwest shore of Lake Winnipeg, MB. Filters operated at a 24-hour HRT, with active treatment seasons from May to September. System performance was assessed over a two-year period (2017 - 2018), with regards to total suspended solids (TSS), chemical oxygen demand (COD), nitrogen (N) and phosphorus (P). TSS were removed equally well in sand and glass filters, with removal efficiencies above 90%. Sand and glass filter effluents contained mostly dissolved COD, except within the first month of operation, during which both media failed to achieve local BOD guidelines. Dissolved COD removal was 29% less in glass, due to smaller media surface area. NH₄+-N reductions exceeded 90% in both filters. Effluent N guidelines were always met by sand filters, whereas glass exceeded the limit 33% of the time in 2018. Both sand and glass failed to reduce P below 1 mg/L. Sand filters seemed to predominantly remove P through mineral precipitation with Ca and Mg compounds. This contributed to concretion (hardening) of top sand filter layers. P removal was significantly less in glass, due to decreased Ca availability within the media. Obtained results indicate that crushed recycled glass can be used as filter media in tertiary wastewater treatment systems, especially to remove TSS, COD and NH₄+–N.

Keywords: tertiary wastewater treatment, wastewater filtration, filter media, sand, crushed recycled glass, contaminant removal.

ACKNOWLEDGEMENTS

First and foremost, I would like to thank God, for the gift of life and for all the opportunities He has given me. I am very grateful to my advisor, Dr. Cicek, for his patience, help, insight and support during these two years. His guidance throughout the Masters was essential and taught me valuable lessons. I would also like to extend my appreciation to Dr. Yuan and Dr. Grosshans, members of my committee, for their support and advice.

I would like to express my gratitude to Joe Ackerman, who helped me so much in the lab and also in the field. Thank you for always being open to brainstorm and to answer my questions. I am thankful to Daniel Flores, Quintin Litke and Luis Barquero, for helping me with field work and lab analyses. Without you, field days would not have been as fun.

I extend my gratefulness to Dale Bourns, for his technical assistance with the column experiment; Warren Blunt, for his help with the Bradford Assay; Victor Wei and Tom Ward, for their assistance with FIA and ICP analyses; Mark Cooper, for performing XRD analysis on my samples and for his valuable insight on the subject; and Tanner Devlin, Alessandro di Biase, Maciej Kowalski and Xugang Zhang, for running my FIA samples in 2018.

My parents, Ingo and Gudula Salzmann, and my sister, Anke Salzmann, were another important part of this journey. Thank you for always supporting and encouraging me. This achievement is also yours. I am also grateful to all my friends, in both Canada and Brazil, for filling these two years with great moments and adventures.

Last, but certainly not least, I would like to express my gratitude to those who provided the financial support for this program: Dr. Cicek, The Faculty of Graduate Studies (UMGF, IGSES) and Hon. Kelvin Goertzen (MGS). Without your incentive nothing of this would have been possible, thank you!

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LIST OF ABBREVIATIONS

Al – Aluminum
BOD – Biochemical Oxygen Demand
C – Carbon
Ca – Calcium
CBOD – Carbonaceous Biochemical Oxygen Demand
CO ₂ – Carbon Dioxide
COD – Chemical Oxygen Demand
Cu – Copper
DI water – Deionized Water
EDL – Electrical Double Layer
EDS – Energy Dispersive Spectroscopy
EW – Emptying Well
Fe – Iron
FIA – Flow Injection Analysis
H – Hydrogen
HCl – Hydrochloric Acid
HNO ₃ – Nitric Acid
ICP-MS – Inductively Coupled Plasma Mass Spectrometry
k – Permeability Coefficient (cm/s)
K – Potassium

Mg – Magnesium

N – Nitrogen

Na – Sodium

NaOH – Sodium Hydroxide

NH₃ – Ammonia

NH₃-N - Un-ionized Ammonia Nitrogen

NH₄⁺ – Ammonium

NH₄⁺–N – Ammonium Nitrogen

 NO_2^- – Nitrite

NO₂-N – Nitrite Nitrogen

NO₃⁻ – Nitrate

NO₃-N – Nitrate Nitrogen

O – Oxygen

P – Phosphorus

S – Sulfur

SEM – Scanning Electron Microscope

TKN – Total Kjeldahl Nitrogen

TN – Total Nitrogen

TP – Total Phosphorus

TSS - Total Suspended Solids

VSS – Volatile Suspended Solids

XRD – X-Ray Diffraction

Y – Microbial Biomass Yield Coefficient

CHAPTER 1 – INTRODUCTION

1.1 Municipal wastewater: chemical characterization and impacts on receiving water bodies

Municipal wastewater refers to the sanitary sewage produced by households, businesses, industries and institutions. It may also include storm runoff, infiltration and inflow water. Domestic wastewater characteristics are dependent on cultural, economic, climatic and geographic aspects (Hopcroft, 2014). The composition, by weight, consists of approximately 99.9% water and 0.1% solids (Di Bonito, 2008).

The solid fraction comprises organic and inorganic compounds, which can exist in suspended, colloidal or dissolved physical states. Organic substances represent about 50% of the solids in domestic wastewaters (Muralikrishna & Manickam, 2017); proteins, carbohydrates and fats are most abundant (Wentzel et al., 2003). Inorganic components, also referred to as mineral substances, comprise grit, silt, chloride, and mineral and metallic salts (Hopcroft, 2014). Nitrogen (N) and phosphorus (P) exist as both organic and inorganic compounds in municipal wastewater, with ammonia (NH₃) and phosphates being the most common forms (Wentzel et al., 2003).

The combination of organic and inorganic constituents defines the strength of the wastewater, i.e. its potential of polluting the receiving water body. This parameter is related to the concentration of volatile solids in the effluent: the higher the organic content, the greater the strength (Muralikrishna & Manickam, 2017). Wastewater strength is usually quantified in terms of oxygen depletion, through biochemical (BOD) or chemical (COD) oxygen demand.

BOD estimates the oxygen consumption by microorganisms during aerobic oxidation of organic matter (Van Haandel & Van der Lubbe, 2012). It usually takes 20 days for complete biological oxidation to occur; however, most of the total BOD is consumed within 5 days. For this reason, the standard method measures the biological oxygen consumption after a 5-day incubation period at 20°C (BOD_{5,20}) (Wentzel et al., 2003). COD – advantageous due to its shorter experimental duration (2 hours) – estimates the oxygen demand resulting from the chemical oxidation of organic matter.

Oxygen depletion in aquatic ecosystems is only one of the impacts of municipal wastewater discharges. These effluents can also introduce pathogens, chemicals, metals and nutrients into the water. The latter are especially problematic, as N and P are macronutrients that can lead to eutrophication and excessive algae growth (Chambers et al., 1997; Holeton et al., 2011; Hopcroft, 2014). Phosphorus, in particular, is the limiting nutrient in most freshwater bodies (Chambers et al., 2001; Correll, 1999); therefore, P loadings in municipal wastewater effluents have to be strictly regulated (Wentzel et al., 2003). Sanitation systems, wastewater treatment processes and effluent discharge guidelines play key roles in minimizing deleterious impacts of municipal wastewater discharges to surface waters (Holeton et al., 2011).

1.2 Provincial water and wastewater quality guidelines

In the province of Manitoba, water and wastewater quality are regulated by two main laws: The Environment Act (Government of Manitoba, 1987) and The Water Protection Act (Government of Manitoba, 2005).

The Environment Act defines the environmental protection and management system in Manitoba, serving as a base for provincial planning and policy mechanisms. It provides directives for the environmental assessment and licensing process of developments; prohibits the unauthorized release of pollutants that may cause adverse effects on human health and/or the environment; deliberates on environmental protection orders and emergency actions to protect the environment; outlines orders to pay costs based on the 'polluter pays' principle; among others.

The Water Protection Act is intended to promote protection of water resources and aquatic systems within the province. The Manitoba Water Quality Standards, Objectives and Guidelines Regulation (Government of Manitoba, 2011), one of the bylaws under the Water Protection Act, is especially important in the water and wastewater quality context. It addresses water quality management (preservation, conservation and rehabilitation) through technology-based and water quality-based strategies. Following the pollution prevention approach, standard concentrations for different classes of effluent discharges are established by this regulation.

Table 1 indicates maximum allowable concentrations for total suspended solids (TSS), BOD, total phosphorus (TP), total nitrogen (TN), un-ionized ammonia nitrogen (NH₃–N) and *Escherichia coli* (*E. coli*) in industrial and municipal wastewater effluents. Total NH₃ limits are defined as site-specific by the Manitoba Water Quality Standards, Objectives and Guidelines Regulation. Thus, the NH₃–N limit in Table 1 refers to the federal standard, established by The Wastewater Systems Effluent Regulations (Government of Canada, 2012), under the Fisheries Act (Government of Canada, 1985).

Table 1 – Water quality standards for industrial and municipal wastewater effluents discharged to a water body

Parameter	Maximum allowable limit	
TSS (mg/L) ¹	25	
BOD (mg/L) ¹	25	
TP (mg/L) ¹	1.00	
TN (mg/L) ¹	15	
NH_3 – $N (mg/L)^2$	1.25	
E. coli (organisms/100 mL) ¹	200	
Sources		
¹ Provincial guideline (Covernment of Manitoha, 2011)		

¹Provincial guideline (Government of Manitoba, 2011).

The phosphorus standard has recently become stricter: since January 1st, 2016, all sewage treatment plants or wastewater treatment lagoons owned or operated by the Manitoba Government are required to maintain effluent concentrations at a maximum of 1.0 mg TP/L (Government of Manitoba, 2011). Phosphorus concentrations for compliance check are determined based on frequency of discharge. For developments that discharge continuously, TP is obtained from a 30-day rolling average. Developments that discharge intermittently, with a discharge period of 30 days or more, also follow the aforementioned calculation. Those that operate at intermittent discharges lasting less than 30 days, quantify TP as an average per discharge period.

²Federal guideline (Government of Canada, 2012).

1.3 Municipal wastewater: collection systems and treatment process

According to the 2006 Municipal Water and Wastewater Survey (MWWS), the mean Canadian wastewater production corresponds to 668 L/capita/day, with Manitoba's average being 31% less than that (Environment Canada, 2010). The 2009 MWWS determined that the percent population served by sewers equals 87.1% in Canada and 88.6% in Manitoba (Environment Canada, 2011). The same survey indicated that private septic systems and sewage holding tanks account for about 13% of the Canadian population that is provided with some sort of residential wastewater service, whereas in Manitoba this statistic reached 11.4%.

Municipal wastewater discharge represents one of the principal causes of water quality decrease in Canadian surface waters (Government of Canada, 2017). In order to minimize adverse effects on receiving water bodies, domestic effluents undergo treatment before discharge. This process involves preliminary, primary, secondary and tertiary treatment stages, in which contaminant removal is achieved through physical, mechanical, biological and chemical mechanisms.

Preliminary treatment removes coarser and larger solids from the wastewater. This process is commonly employed to minimize operation and maintenance issues in succeeding treatment stages, therefore maximizing their performance (Mateo-Sagasta et al., 2015). Preliminary treatment units may include screening and skimming devices; grit chambers; shredders and grinders; flow measurement and equalization devices; septage receiving stations; among others (Chiban et al., 2013).

Primary treatment occurs in sedimentation/clarification tanks, where solids removal is achieved through two mechanisms: 1) Gravity settling; and, 2) Flotation

(Sutherland, 2008). Larger solids settle vertically, forming primary sludge; buoyant materials (e.g. plastic, oil and fat) float to the surface, generating a scum layer (Cheremisinoff, 2002). Most of the solids removed during this phase are organic, including some N and P compounds. Chemical precipitation and flocculation techniques can be applied to enhance the quality of primary effluents. These contain approximately 35, 50 and 65% of the initial oil/grease, TSS and BOD levels, respectively (Chiban et al., 2013; Hopcroft, 2014).

Secondary wastewater treatment consists of biological mechanisms that remove colloidal and dissolved organic matter from the primary effluent (Van Haandel & Van der Lubbe, 2012). These systems are based on fixed film or suspended growth mechanisms (Cheremisinoff, 2002). Common secondary treatment methods include activated sludge systems, stabilization ponds, trickling filters, oxidation ditches, rotating biological contractors, and constructed wetlands. In comparison to the initial concentrations, secondary treatment provides an additional removal of BOD (50%) and TSS (35 - 45%), achieving a total dissolved solids (TDS) reduction of 85% or more (Hopcroft, 2014). Nutrient removal rates are rarely significant at this stage.

Tertiary (or advanced) treatment represents the final wastewater treatment step. This stage is not always required, being only necessary when secondary effluents do not meet discharge guidelines or reuse quality standards (Sutherland, 2008). Contaminants removed during the advanced treatment include organic matter, residual suspended solids, pathogens, and nutrients (P and N substances) (Van Haandel & Van der Lubbe, 2012). Tertiary treatment methods include disinfection (for instance chlorine,

ozone and ultraviolet light), activated carbon adsorption, membrane technologies, filtration, infiltration, among others.

Table 2 indicates wastewater treatment levels in Canada and Manitoba, according to the 2006 and 2009 MWWS surveys. At the national level, 78.7% of the population receives secondary or tertiary treatment, with secondary mechanical processes as conventional treatment. In smaller municipalities (up to 5,000 residents), main treatment methods consist of secondary systems using waste stabilization ponds or lagoons (Environment Canada, 2010). As for Manitoba, secondary mechanical systems treat the majority of the wastewater produced in the province. Less than 5% of the population does not have access to secondary or higher wastewater treatment levels.

Table 2 – National and provincial wastewater treatment levels by national and provincial population (%)

Treatment	Canada ¹	Manitoba ²
None or preliminary only	3.2	0.5
Primary	18.1	3.9
Secondary (waste stabilization ponds/lagoons)	6.8	10
Secondary (mechanical)	54.5	69
Tertiary	17.4	16.7
Sources		
¹ Environment Canada (2011).		
² Environment Canada (2010).		

1.4 Filtration as a wastewater treatment method: an emphasis on tertiary granular filters

Wastewater treatment commonly includes liquid filtration – a phase separation mechanism where a porous medium is used to isolate the solid fraction of an incoming fluid (Cheremisinoff, 2002; Crittenden et al., 2012; Sparks & Chase, 2016). The effluent obtained after that process is denominated filtrate. Filter design and operation affect the composition of the solids retained by the system, which may comprise both suspended and dissolved compounds.

Filtration techniques are implemented to achieve one or two of the following aspects: 1) Product recovery of solid or liquid fraction (Sutherland, 2008); and, 2) Fluid purification prior to discharge or reuse (Sparks & Chase, 2016). The latter represents the main application of filter units in wastewater treatment processes. Within this context, this thesis focuses on granular filters as polishing systems for municipal wastewater lagoon effluents.

1.4.1 Operation principles of tertiary granular filter systems

Granular filtration is frequently applied to achieve removal of residual suspended solids from domestic wastewater effluents (Hamoda et al., 2004; Gill et al., 2011; Verma et al., 2017). These treatments are effective for influent TSS concentrations between 100 and 200 mg/L, achieving filtrate TSS levels ≤ 10 mg/L (Cheremisinoff, 2002). Granular filters can be gravity or pressure-fed, operating in two different phases: filtration and backwash (Cheremisinoff, 2002; Drinan & Spellman, 2013).

In the former, wastewater is added to the surface of a porous medium, through which it vertically percolates until the underdrain collection system is reached. Backwashing, which is periodically performed, consists of pumping clean water through the filter in the reverse direction, to minimize clogging and extend the lifetime of the filter media (Amirtharajah, 1985). Backwash frequencies depend on both filter media properties and influent solids loading.

Filter media can be grouped into two main classes: surface-type and depth-media. The first refers to substrates that retain contaminant particles on their surface. The second describes a medium that allows solids to penetrate inside the pore structure of the grain (Cheremisinoff, 2002). In granular filters both behaviours are observed. Common physicochemical contaminant removal mechanisms include adherence, interception, inertial deposition, diffusion, among others (Crittenden et al., 2012). Biofilm activity also exists, enabling the removal of not only particulate but also dissolved organic compounds (Lessard & Bihan, 2003).

Sand is the most traditional granular filter medium (Hu & Gagnon, 2006; Horan & Lowe, 2007; Sutherland, 2008; Gherairi et al., 2015); however, materials such as gravel, glass, anthracite, garnet, polonite, and dolomite have also been employed. With regards to layer configuration, granular filters can assume mono, dual or multi-media settings. The most typical media in these setups are sand; anthracite and sand; and, anthracite, sand and garnet, respectively (Hamoda et al., 2004). Dual and multi-media layouts can also be obtained by using different size gradations of the same filter medium (Sparks & Chase, 2016).

Filter media size properties are usually described by effective size (ES) and uniformity coefficient (UC) of grains. These parameters are obtained from standard sieve analyses. The ES, also known as the 10^{th} percentile (d_{10}), represents the particle size (mm) for which 90% of the grains are larger by weight. The UC is determined by the ratio of the 60^{th} percentile grain diameter to the ES: d_{60}/d_{10} . This value indicates media stratification: the greater the UC, the larger the range of particle sizes in the sample (Crittenden et al., 2012).

The performance of granular filters depends on multiple aspects: grain properties – ES, UC, porosity, density and shape (Cheremisinoff, 2002; Soyer et al., 2010; Verma et al., 2017); filter media depth (Bourke et al., 1995; Verma et al., 2017); flow velocity and head loss (Cheremisinoff, 2002; Hamoda et al., 2004); backwashing frequencies (Bourke et al., 1995; Crittenden et al., 2012); hydraulic loading rate and retention time (Hamoda et al., 2004; Verma et al., 2017); influent wastewater quality, in terms of solids, nutrients, etc. (Bourke et al., 1995; Cheremisinoff, 2002; Hamoda et al., 2004; Verma et al., 2017); and, filter clogging (Crittenden et al., 2012).

1.4.2 Physicochemical mechanisms in tertiary granular filtration

The removal of wastewater contaminants in granular filters is affected by the combined action of transport, attachment and detachment phenomena.

1.4.2.1 Transport mechanisms

Transport mechanisms approximate solids to the filter media surface or to existing particle deposits. These processes generally assume that flow within the

medium is laminar (Ives, 1970). In this scenario, suspended solids are carried along wastewater streamlines that flow around filter grains, while moving through the bed (Sutherland, 2008; Sparks & Chase, 2016). Transport processes include straining, interception, inertial impaction, sedimentation, diffusion, and hydrodynamic action (Jegatheesan & Vigneswaran, 2005; Sutherland, 2008; Crittenden et al., 2012; Sparks & Chase, 2016).

- Straining (sieving): Takes place on the filter's surface or along its depth (Ives, 1970; Sutherland, 2008). In the first case, solids that do not fit through the media voids are retained on the upstream filter face (Cheremisinoff, 2002). This can yield to the formation of filter mats, which can favour clogging (Crittenden et al., 2012). Depth straining occurs when particles get trapped while moving through media pores, due to size or shape constrictions (Sutherland, 2008).
- ❖ Interception: Occurs when the distance between medium grains and contaminants travelling along fluid streamlines is equal to or less than the particle's radius (Jegatheesan & Vigneswaran, 2005). These conditions lead to particle-surface contact, with this mechanism depending on both particle and grain diameters (Ives, 1970). As highlighted by Ives (1970), interception and straining are similar, with the difference that the former also applies to very small particles.

- ❖ Inertial impaction: Contaminant-media collisions that happen when particles have enough inertia to drift away from fluid streamlines, which are bending around grains (Sparks & Chase, 2016). Larger particles or those travelling at high velocities are more likely to experience inertial interception (Sutherland, 2008).
- Sedimentation (gravitational settling): Large particles with high densities are more prone to deviate from the streamlines and suffer sedimentation (Sparks & Chase, 2016). In contrast to wastewater flow, these particles move at a constant velocity (Jegatheesan & Vigneswaran, 2005), which stimulates settling due to gravitational forces.
- Diffusion: Particles that have diameters smaller than 1 μm are influenced by Brownian motion (Ives, 1970) a random movement caused by the thermal agitation of wastewater molecules. This mechanism deflects contaminants from the fluid streamlines (Sutherland, 2008; Crittenden et al., 2012), increasing the probability of inter-particle and particle-surface encounters (Cheremisinoff, 2002).
- Hydrodynamic action: Random movement of particles across wastewater streamlines, due to the shear flow within filter pores (Ives, 1970; Jegatheesan & Vigneswaran, 2005). Hydrodynamic forces maximize the chances of occurrence of contaminant-surface collisions.

1.4.2.2 Attachment mechanisms

The particle-surface contact facilitated by transport mechanisms represents only part of the contaminant removal process in granular filters. Attachment forces are responsible for binding solids to the filter media or to particle aggregates, therefore removing them from suspension. These mechanisms are divided into two main groups:

1) Long-range forces; and, 2) Short-range forces.

- Long-range forces: Can affect particles located at a distance up to 100 nm from the filter medium (Jegatheesan & Vigneswaran, 2005). Van der Waals forces and electrical double-layer interactions (EDL) are the main types. The former promotes intermolecular attraction between contaminants and filter surface (Cheremisinoff, 2002), with a particle-media distance range < 50 nm (Ives, 1970). The EDL forces depend on the electrical charges of filter grains and contaminants. In granular systems, filter grains hold a surface charge, which consists of adsorbed ions and/or dissociated surface groups (Jegatheesan & Vigneswaran, 2005). This electric layer interacts with the ions contained in the fluid, generating attractive or repulsive forces.</p>
- ❖ Short-range forces: Take place when the separation between particles and grain surface is ≤ 5 nm (Jegatheesan & Vigneswaran, 2005). This group includes born repulsion forces and hydration forces. Born repulsion occurs when similarly charged ions are electrostatically pushed away from each other; these forces are intense at short interatomic distances (Jegatheesan &

Vigneswaran, 2005). Hydration happens when water molecules on both particle and media surfaces are connected through hydrogen bonds (Ives, 1970).

1.4.2.3 Detachment mechanisms

Detachment mechanisms can occur during backwashing or due to hydrodynamic flow forces within the filter (Ives, 1970; Jegatheesan & Vigneswaran, 2005). These phenomena can induce previously adhered solids to separate from the filter media or from existing particle deposits. Detached contaminants are put back in suspension, undergoing further transport and attachment processes.

1.4.3 Biological mechanisms in tertiary granular filtration

In granular filtration, biological contaminant removal relies mostly on fixed-film processes, which are characterized by biofilm growth on filter media surfaces (Gray, 2004; Von Sperling, 2007). Biofilm first develops when bacteria are adsorbed onto the surface of a solid medium. If there is enough moisture and nutritional substrate, i.e. sources of macro (C, H, O, N, P, S) and microelements (K, Ca, Mg, Na, Fe), adhesion occurs (Chan, 2003). Bacteria then start to grow and reproduce, forming a matrix of extracellular polysaccharides (Morgenroth, 2008a).

Although bacteria are dominant in fixed-film reactors, other microorganisms can be found, including fungi, protozoa, algae, larvae and worms (Hedaoo et al., 2012; Lessard & Bihan, 2003). Depending on how much the biofilm expands, both aerobic and anaerobic or anoxic conditions may exist. The former prevail in granular filtration,

occupying the thickest part of the film. Anaerobic or anoxic layers can be formed beneath the aerobic fraction, occurring in close proximity to the support medium (Gray, 2004). In these zones, denitrification, fermentation and methanization can take place (Lessard & Bihan, 2003).

Biofiltration of municipal wastewater removes mainly dissolved organic contaminants, as microbial nutrition is mostly based on absorption processes (Chan, 2003). Transport phenomena within the filter system enable the approximation of colloidal and dissolved solids to the biofilm. Some particles adhere to the surface of the film; oxygen and other dissolved organic contaminants are transported through the biofilm via diffusion (Von Sperling, 2007). The latter are primarily metabolized by heterotrophic bacteria, with the purpose of: 1) Energy production (catabolism); and, 2) Synthesis of new protoplasm (anabolism) (Wentzel et al., 2003).

Catabolic reactions oxidize part of the organic content absorbed by the biofilm, producing carbon dioxide (CO₂), water and energy (Chan, 2003). Some of this energy is consumed during anabolic pathways, in which organic contaminants are modified into new bacterial cell mass (Comeau, 2008). This process also requires inorganic compounds such as ammonium (NH₄+) and P (Wentzel et al., 2003), resulting in the expansion of the biofilm within the filter.

Microbial biomass yields (*Y*), in terms of volatile suspended solids (VSS), are estimated based on the mass of electron donor utilized. Electron donor refers to the substrate that is oxidized during catabolic reactions (Comeau, 2008). In the case of heterotrophic bacteria, organic matter (in COD units) serves as the main electron donor. If nitrifying microorganisms are present, electrons are provided by NH₄⁺ or nitrite (NO₂⁻).

For aerobic heterotrophs, Y varies between 0.39 and 0.49 g VSS/g COD (Comeau, 2008; Wentzel et al., 2003), with a theoretical yield of 0.42 g VSS/g COD (Wentzel et al., 2003). For nitrifiers, Y coefficients are 0.34 g VSS/g NH₄+–N and 0.08 g VSS/g NO₂-N (Comeau, 2008).

In vegetated filters, biological contaminant removal is achieved by both biofilm activity and plant uptake. Nitrogen and phosphorus, which exist in large quantities in secondary wastewater effluents, are primary plant nutrients (Mitra, 2017). Phosphorus accounts for approximately 0.2% of a plant's dry weight (Schachtman et al., 1998), while N content varies between 1 - 6% (Mitra, 2017). These nutrients are primarily consumed by plants in the form of inorganic phosphates, nitrate (NO₃⁻) and NH₄⁺. In order to effectively remove contaminants from the system, plants have to be harvested from filter surface after treatment season is complete (Arias et al., 2001).

1.4.4 Phosphorus removal in tertiary granular filtration

Secondary wastewater effluent contains mostly inorganic P, in the reactive form of orthophosphates (Hopcroft, 2014). The majority of these compounds are dissolved in the water. Phosphate removal is achieved through plant uptake, biological assimilation, and physicochemical processes (Bubba et al., 2003; Prochaska & Zouboulis, 2006). The latter prevail in filtration (Aulenbach & Meisheng, 1988; Tofflemire & Chen, 1977), where adsorption and chemical precipitation are main mechanisms.

Adsorption is characterized by the adhesion of atoms or molecules to the filter media surface (Lüth, 2015). The P-sorption capacities of materials are described by Langmuir and Freundlich isotherms. The former considers that the media surface is uniform (Arias et al., 2001), consisting of sites that have similar P-sorption affinities (Bubba et al., 2003). Therefore, at maximum coverage, a monolayer is formed (Chung et al., 2015; Samadi et al., 2015). This model assumes that chemical precipitation phenomena are absent. The Freundlich equation, on the other hand, is suitable for describing P-sorption when precipitation is also present. This model is based on the assumption of surface heterogeneity (Samadi et al., 2015), resulting in sites with different P-sorption affinities and in the formation of multilayers (Bubba et al., 2003; Chung et al., 2015). Adsorption isotherm tests are usually performed over a period of 2-5 days (Tofflemire & Chen, 1977), as equilibrium can be reached relatively fast. Aulenbach & Meisheng (1988), for example, obtained saturation of the P-sorption capacity of natural sand treated with secondary wastewater after 22 hours.

While adsorption might be the initial P removal mechanism within filter systems, long-term reductions are mostly achieved via chemical precipitation (Arias et al., 2001; Aulenbach & Meisheng, 1988; Tofflemire & Chen, 1977). This process, also known as slow mineralization, is intimately related to pH, wastewater composition and metallic ion content of the filter media (Erickson et al., 2006; Gill et al., 2009; Jenkins et al., 1971). Under acidic conditions, phosphates precipitate as aluminum (Al) and iron (Fe) compounds (Arias et al., 2001). At pH levels > 6, phosphate removal is achieved through the combined action of two mechanisms: 1) Adsorption onto Al and Fe oxides; and, 2) Mineral precipitation with calcium (Ca) and magnesium (Mg) compounds

(Erickson et al., 2006; Gill et al., 2009; Jenkins et al., 1971). The alkaline nature of municipal sewage favours the precipitation of calcium phosphates during filtration (Arias et al., 2001; Peng et al., 2018), especially in sand beds, which are rich in Ca. Besides, wastewater contains Ca⁺² and Mg⁺² ions that can be consumed during phosphate precipitation processes. Phosphorus removal may also be affected by the presence of calcium carbonate (CaCO₃, calcite) in the media, due to co-precipitation and adsorption of calcium phosphates onto the calcite surface (Aulenbach & Meisheng, 1988; Prochaska & Zouboulis, 2006; Xu et al., 2014).

1.5 Crushed recycled glass as an alternative wastewater filter media

Different driving forces have led to the investigation of crushed recycled glass as an alternative filter media to sand in wastewater treatment. The most common motivations for studies carried out in this field include: large availability of waste glass and lack of alternative markets for this commodity (Horan & Lowe, 2007; Hu & Gagnon, 2006); decrease in the amount of glass that is landfilled (Gill et al., 2011; Horan & Lowe, 2007); achievement of national glass recycling targets (Horan & Lowe, 2007); reduced exploitation of natural sand resources (Gherairi et al., 2015); minimized energy and transportation costs, regarding both sand importation and waste glass exportation (Gill et al., 2011); reduced carbon dioxide emissions (Horan & Lowe, 2007); possibility of pulverizing glass into different particle sizes (Horan & Lowe, 2007; Hu & Gagnon, 2006); lower backwash rates, as a result of reduced clogging (Gill et al., 2011; Horan & Lowe, 2007); lower implementation, operation and maintenance costs (Elliott, 2001a; Elliott, 2001b; Gill et al., 2011; Horan & Lowe, 2007; Hu & Gagnon, 2006); and favourable

publicity/increased competitiveness due to the use of a sustainable product (Gill et al., 2011; Horan & Lowe, 2007; Hu & Gagnon, 2006). In summary, environmental sustainability is generally indicated as the determining factor supporting the use of recycled glass as filter media.

1.5.1 Challenges of waste glass recycling

Glass containers are widely used in food and beverage packaging, accounting for approximately 98% of the total waste glass production (Arvanitoyannis, 2008). For glass to be recycled, it has to be cleaned, separated by colour and crushed to cullet. Cullet is then mixed with virgin materials and melted at 1,425 - 1,535°C (Arvanitoyannis, 2008). The addition of cullet reduces not only the amount of raw materials (Mohajerani et al., 2017), but also the energy required during the process: the heat consumption for cullet meltdown is 33 - 50% lower than that of virgin glass (Jain et al., 2012).

Although glass can be continually recycled without loss of quality (Sobolev et al., 2007), recycling rates are relatively low (Arvanitoyannis, 2008; Horan & Lowe, 2007; Jain et al., 2012; Ling et al., 2013; Mohajerani et al., 2017). In the short term, recycling can be financially and energetically more demanding than using sand to produce virgin glass (Meyer et al. 2001). As glass is easily breakable, fragments are often mixed with other recyclable materials in disposal bins or collection vehicles. This poses obstacles to glass separation and retrieval at recycling facilities, increasing equipment, maintenance and processing costs (Arvanitoyannis, 2008; Jain et al., 2012; Meyer et al. 2001). Additionally, it leads to the incorporation of glass contaminants into recycled products such as paper, plastics, and metal, reducing their economic values.

Source separation systems and deposit-return programs minimize the complications of waste glass recycling (Arvanitoyannis, 2008). In the former, consumers separate waste glass according to colour. This configuration is common in Europe. In Switzerland, for instance, glass recycling rates exceed 90% (Mohajerani et al., 2017). Separation is not the only obstacle for waste glass recycling. Logistics also play an important role in the process, especially when recycling processing plants and factories are not locally available. In some cases, shipping costs equal or exceed the value paid for the material, making the process unfeasible (Ling et al., 2013; Meyer et al., 2001).

As a result of the aforementioned factors, recycled glass is a low-value commodity, with a restricted marked (Elliott, 2001a; Horan & Lowe, 2007). This forced some municipalities to discontinue glass collection for recycling purposes and to landfill instead (Arvanitoyannis, 2008). Within this context, alternative markets for waste glass have been sought, especially in which cullet quality is not a determining factor.

These generally include the use of waste glass as an additive in cement (Ling et al., 2013; Mohajerani et al., 2017; Sobolev et al., 2007); aggregate material for concrete and asphalt (Gaitanelis et al., 2018; Jain et al., 2012; Ling et al., 2013; Mohajerani et al., 2017; Rutledge & Gagnon, 2002; Sobolev et al., 2007); base and sub-base filler (Mohajerani et al., 2017; Sobolev et al., 2007); drainage material (Rutledge & Gagnon, 2002); and, constituent of insulating materials (Jain et al., 2012; Rutledge & Gagnon, 2002), ceramics (Gaitanelis et al., 2018) and bricks (Jain et al., 2012; Mohajerani et al., 2017).

1.5.2 Canadian statistics for glass waste production and recycling

In Canada, glass constitutes 3% of the solid waste generated at the household level (Statistics Canada, 2005). Based on this average, residential sources in Manitoba produced 9.3 kt of waste glass in 2016 (Statistics Canada, 2018a). In the same year, 8.4 kt of glass were diverted from disposal sites for recycling purposes, from both residential and non-residential provincial waste sources (Statistics Canada, 2018b). This value represents less than 4% by weight of the total amount of materials diverted that year.

In Manitoba, deposit-return policies are not enforced and waste glass is considered a hard-to-recycle product (Government of Manitoba, 2014). The volume of waste that is transformed into new glass containers is minimal (Morawski et al., 2016), as it involves shipping to out-of-province recycling facilities in Moose Jaw, SK and Shakopee, MN. Most of the collected glass is crushed and reused as sidewalk and road base aggregate (City of Winnipeg Water and Waste Department, 2018; Morawski et al., 2016; Reindl, 2003), occasionally serving as fill in water and sewer trench systems (Multi-Material Stewardship Manitoba, 2018). The Brady Road Resource Management Facility, for instance, constructs roadways within the landfill using crushed recycled glass and gravel as base and top layers, respectively (Hood, 2006). Rocky Road Recycling Ltd. is one example of a local facility that transforms waste glass into granules for reuse in concrete and asphalt production (Stantec, 2011).

1.5.3 Previous studies on the use of recycled glass as wastewater filter media

Although sand has been the leading medium in wastewater filter systems, alternative media have been evaluated, including crushed recycled glass (Elliott, 2001a; Elliott, 2001b; Gherairi et al., 2015; Gill et al., 2011; Horan & Lowe, 2007; Hu & Gagnon, 2006). Elliott (2001a, 2001b) and Hu & Gagnon (2006) assessed the performance of glass in intermittent recirculating biofilters (RBFs) treating septic tank effluent. Gherairi et al. (2015) compared BOD and COD removal in sand and glass filters receiving synthetic household sewage. Gill et al. (2011) and Horan & Lowe (2007) analyzed the efficiency of crushed recycled glass and sand in domestic wastewater polishing filters.

Three aspects of the aforementioned studies stand out. First, the use of three grades of recycled glass – fine (0.2 - 1.0 mm), medium (0.5 - 1.45 mm) and coarse (1.5 - 2.5 mm) – by Horan & Lowe (2007). These gradations were evaluated at pilot-scale filters (reactor volume: 0.03 m³), with the medium-grade glass further analysed at full-scale dimensions (0.8 m³). Second, the effluent sampling methodology followed by Gill et al. (2011). Samples were collected at multiple gradients of the filter media, so that removal performances across different layers could be assessed. Third, the adoption of similar ES and UC values for sand and glass media by Gherairi et al. (2015) and Hu & Gagnon (2006). This is important when comparing two different materials, as removal performances depend on physical characteristics of filter media.

Glass filters have been found successful in reducing TSS. Elliott (2001b) and Hu & Gagnon (2006) verified TSS reductions of 98% and 79% after glass filtration, respectively. In both studies, most of the TSS was removed in septic (60%) (Elliott, 2001b) and recirculation tanks (69%) (Hu & Gagnon, 2006). At the pilot scale, Horan &

Lowe (2007) observed that the fine glass produced the best TSS effluent quality (15 mg/L), treating a volume of 15 m³ before backwash was required. This volume was relatively small in comparison to sand (35 m³), medium glass (55 m³) and coarse glass (48 m³). In terms of total solids removed, medium glass performed best (0.86 kg), while sand retained about 0.35 kg. In full-scale filters, 75% TSS removal was verified for influent concentrations \leq 70 mg/L, in both sand and medium glass. The authors concluded that, for TSS guidelines to be met, flow velocity and influent solids loading should be less than 15 m/h and 0.25 kg solids/m³/h, respectively. At an optimised backwash rate (0.45 m³/m²/min), the glass filter treated 10% more flow than the sand, indicating that blinding was less frequent.

As for oxygen demand, Gill et al. (2011) and Hu & Gagnon (2006) both proved that sand and glass performed equally well in terms of organic material removal. Elliott (2001b) and Hu & Gagnon (2006) observed BOD removal efficiencies around 96% in glass filters, while Gill et al. (2011) obtained COD reduction percentages of 73% in the same medium. Gherairi et al. (2015) also achieved high COD and BOD removal after glass filtration, reaching maximums of 91.9% and 92.5%, respectively. Gill et al. (2011) noted that 52% of the total COD reduction occurred within the first 300 mm of the sand filter, comprised of gravel (first 100 mm) and sand (200 mm). In the glass filter, this proportion was only 30%, indicating that COD removal within this medium occurred along its depth.

In terms of nitrogen removal in glass filters, both Elliott (2001b) and Hu & Gagnon (2006) observed ammonium nitrogen (NH₄+–N) reductions above 90%. No significant difference between the performance of sand and glass at the 95% confidence level was

identified (Hu & Gagnon, 2006). The latter noted that TN removal (75%) occurred mostly within the recirculation tank, rather than through filtration. Gill et al. (2011) obtained TN removal rates of 16% and 28% in sand and glass effluents, respectively. The authors attributed the higher efficiency of the glass to the fact that less COD (30% against 50% in the sand) was removed within the first 300 mm of the bed. This allowed greater amounts of organic matter to reach deeper filter layers rich in nitrates, providing substrate for heterotrophic denitrifying bacteria.

Phosphorus removal was measured by Hu & Gagnon (2006) and Gill et al. (2011). Hu & Gagnon (2006) failed to achieve satisfactory TP removal in both filters, leading to high effluent concentrations of 8.6 mg/L (sand) and 11.6 mg/L (glass). The authors believe that media adsorption capacities were low or exhausted by the time the experiment was conducted, as the RBF had been running for 9 months prior to that. This resulted in insignificant P removal in sand and glass filters. Gill et al. (2011) included a limestone sand layer (100 mm) into each filter bed, which retained 50% of the TP removed. The removal efficiency of the sand filter was 28% higher, which was expected: Freundlich and Langmuir isotherms indicated low affinity for phosphate in the glass media. Sand and glass adsorption data were more successfully modelled with the Freundlich isotherm, indicating that both adsorption and precipitation mechanisms removed phosphate within the filters. As in Hu & Gagnon (2006), sand and glass TP effluent concentrations were not below legal limits.

Pathogenic microorganism removal was investigated by Hu & Gagnon (2006) and Gill et al. (2011). The former observed *E. coli* log removals of 1.1 and 3.3 in sand and glass effluents, respectively; the latter verified 3 log reduction rates in both media.

Gill et al. (2011) noted that more than 65% of the coliform log removal took place within the first 200 cm of the beds. In both studies the final effluent quality obtained after sand or glass filtration did not meet *E. coli* discharge requirements.

With respect to physical properties, glass showed to have higher durability than sand (Elliott, 2001b). This was determined based on acid solubility and magnesium sulphate soundness tests, which indicated that weight losses in the sand were 29 and 17 times higher, respectively. Gill et al. (2011) performed Scanning Electron Microscope (SEM) analysis on glass and granite sand with respective effective sizes of 0.23 mm and 0.15 mm. Results revealed that both surface roughness (µm) and area (m²/g) were higher in the latter, by factors of 9 and 4, respectively. This supports the hypothesis that contaminant removal efficiencies are greater in sand media, as these are usually characterized by smaller grain size.

Horan & Lowe (2007) highlighted the need for cost-benefit analyses regarding the use of glass in wastewater filtration. This information is extremely relevant, especially for wastewater companies, as it provides details on the commercial viability of such filters. Until now, Elliott (2001a, 2001b) was the only author who reported on the financial information of these systems. This included construction costs (\$177,500.00), annual operation & maintenance expenses (\$8,650.00) and electric power consumption (\$1,730.29 over a period of 458 days). The author demonstrated that using waste glass instead of sand provides significant savings, as the former costs \$12.50 less per tonne. It was also indicated that transportation plays a major role, reaching up to 50% of the media price.

1.6 Research objectives

As shown previously, information regarding the efficiency of recycled glass as wastewater filter medium is relatively scarce, with the literature being limited to a handful of papers. Side-by-side, pilot-scale studies of sand and glass subsurface filters treating municipal lagoon effluents are not available. This hinders the implementation of such systems. The lack of technical data motivated this research, as well as the possibility of contributing to environmental sustainability by employing a locally recycled material as filter media.

1.6.1 General objective

To assess and compare the performance of crushed recycled glass and river sand as media in subsurface gravity filters treating secondary wastewater lagoon effluents from the Village of Dunnottar, MB.

1.6.2 Specific objectives

- Determination of filter media physical properties (ES, UC, void space, porosity, permeability, and bulk & particle densities);
- Monitoring of metal content (Ca, Mg, K, Na) and physicochemical parameters of influent and filtered effluents (pH, temperature, electrical conductivity and alkalinity);

- ❖ Quantification of filter removal performance in terms of TSS, VSS, COD, nitrogen (NH₄+–N, NO₂⁻ and NO₃⁻) and phosphorus compounds (TP and orthophosphate);
- ❖ Evaluation of the contaminant retention within filter cells through mass balances of TSS, VSS, total and dissolved COD, N content, TP and orthophosphate; and
- Investigation of the formation and chemical composition of concretious sand, which results in filter and pipe clogging.

CHAPTER 2 – MATERIALS AND METHODS

2.1 Study site

This study was carried out at the Village of Dunnottar Wastewater Treatment Facility (50°26'39.3"N, 97°01'04.0"W), on the southwest shore of Lake Winnipeg (Figure 1). The establishment serves the municipality of Dunnottar, with a permanent population of 763 (Statistics Canada, 2017) that increases up to 3,500 during summer months (Dillon Consulting, 2013). This seasonal population peak yields a maximum organic loading rate of 39 kg BOD/ha/day (Dillon Consulting, 2013).

Dunnottar's municipal wastewater treatment is achieved through a three-cell facultative lagoon system (Figure 1), with a combined capacity of 60,000 m³ and an active treatment season from May to October (Dillon Consulting, 2013). The facility comprises one primary and two secondary cells that operate sequentially. Septic trucks collect sewage from 1,173 holding tanks on a weekly basis (Village of Dunnottar, 2018), disposing it into the primary lagoon. Primary and secondary cells are connected by valves that remain open until three weeks prior to discharge, which occurs once a year for both secondary lagoons, between September 16th and October 31st (Village of Dunnottar, 2005). Treated effluents are released into Tegula Creek after compliance check (Stantec Consulting, 2016), eventually flowing into Lake Winnipeg.

In 2013, the establishment was upgraded with a full-scale tertiary passive filter operated by Dillon Consulting Ltd, which treats secondary overflow lagoon effluents during spring and summer. The filter was installed to: 1) Increase the hydraulic capacity of the facility; and, 2) Reduce P loadings by half, to 82 kg P/year (Dillon Consulting,

2013). The system consists of two vertical filter cells with a total volume of 3,000 m³ (Dillon Consulting, 2013), followed by an ultraviolet disinfection component. Filter beds are filled with natural media (rocks, sand and gravel) and covered with organic soil. The passive filter does not interfere with secondary lagoon discharges, operating on continuous discharge between June 16th - September 15th, using the same drainage route of the lagoons (Dillon Consulting, 2013).

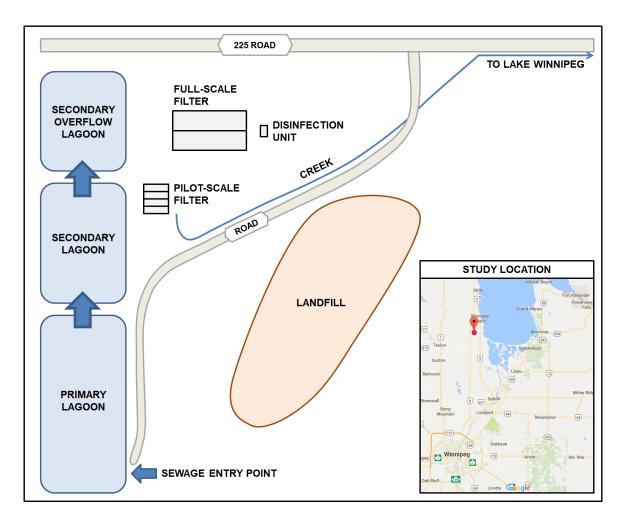


Figure 1 – Scheme of the Village of Dunnottar Wastewater Treatment Facility and map of the study location. **Source of scheme:** The author. **Source of map:** Google Maps (2018).

2.2 Pilot-scale filter

Prior to the construction of the full-scale filter, a pilot system was used for field evaluation within Dunnottar's Wastewater Treatment Facility (Figure 1). The subsurface gravity filter contained four filter cells, providing tertiary treatment for secondary wastewater lagoon effluents. Experimentation started in 2009 and consisted of testing different filter media and flow directions (Dillon Consulting, 2013).

The system was rebuilt in 2016, currently comprising two filter cells with river sand (1W and 4W) and two with crushed recycled glass (2W and 3W). 1W is the closest bed to the primary lagoon, whereas 4W is the nearest filter to the overflow lagoon. Each cell is 10 m long, 2 m wide and 1 m deep (bed volume of 20 m³). Cells are separated by plywood and individually lined with polyethylene plastic, containing limestone cobble on the bottom and filter media on top of that (Figure 2). Wood chips cover the upper part of the beds, so that it is possible to walk on the surface without damaging the system.

The pilot-scale filtration mechanism can be summarized in six steps (Figure 2):

1) Wastewater is pumped from the secondary lagoon through a transfer pipe, splitting water into the four filter beds; 2) Two parallel perforated PVC pipes deliver the water to the surface of each cell; 3) Wastewater percolates vertically through the bed, reaching a collection pipe at the bottom; 4) Treated effluent is collected in individual wells (1W, 2W, 3W and 4W) at the end of each filter; 5) Holding tanks are connected by a main pipe to a final emptying well (EW), which releases treated effluent into the overflow lagoon; and, 6) An emergency flow switch located in EW turns off the main pump – that feeds wastewater to the cells – when EW's water level is too high and there is risk of flooding.

The wastewater pump (Burcam 400500, Burke Water Systems Manufacturing Inc.) that sits in the secondary lagoon works at a head of 1.5 m, yielding a flow rate of 17,000 L/h (Burcam, 2018). A timer controls the pump, leaving it on for 5 min and off for 99 min, resulting in a filter hydraulic residence time (HRT) of 24 hours. Wastewater is delivered to filters for 70 min/d, producing a daily flow rate of 4,958 L to each filter cell. As a result, collection wells at the end of each filter bed, which have an approximate volume of 100 L, are filled and emptied multiple times a day.

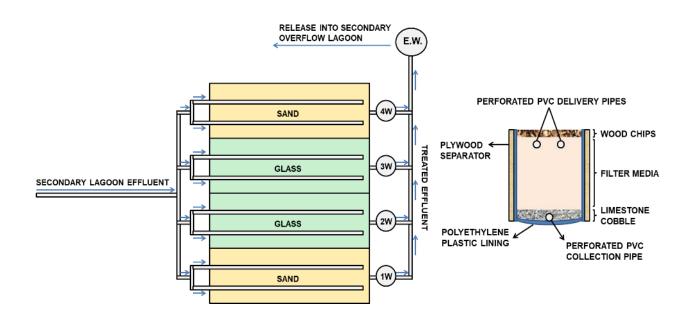


Figure 2 – Top view of the pilot-filter configuration (left) and cross-sectional view of an individual filter bed (right). **Source:** The author.

2.2.1 Characterization of the filter media

River bottom sand and crushed recycled glass were used as filter media in the pilot-scale system. Sand was supplied by a local sand and gravel vendor and was retrieved within a 30 km radius of the site. Glass was obtained from the Cascades

Recovery+ recycling centre located in Winnipeg, which provides sustainable solutions for discarded materials through waste diversion strategies (Cascades Recovery+, 2018). The company contracted a sand and gravel outfit that sieved the material through a ³/₄" tumbling screen; therefore, some gravel, plastic and finer organic materials might have been combined with the crushed glass.

The particle-size distribution of both filter media was obtained through standard sieve analysis (Day, 1965). Gradation curves were used to estimate ES, UC and d_{60} values, as well as to determine sample fractions < 0.075 mm, between 0.075 - 4.75 mm and > 4.75 mm. The physical characterization of the media was also assessed in terms of bulk and particle density (g/cm³), porosity (%), void ratio and permeability (cm/s). Bulk densities were obtained from the ratio of dry mass to bulk volume (ISO, 2017a), whereas particle densities were determined from water displacement measurements (ISO, 2017b). Bulk and particle densities were used to calculate porosity (ASTM International, 2018), with the latter serving as base for the estimation of the void ratio (ASTM International, 2018). Permeability coefficients (k) were calculated by measuring the volume of water that percolated through media specimens under constant head (Eq. 1), with saturated samples prior to testing.

$$k = \frac{Q \times L}{A \times h}$$
 (Eq. 1)

Where: k = permeability coefficient (cm/s); Q = discharge (cm³/s); L = length of specimen (cm); A = cross-sectional area of specimen (cm²); h = head (cm).

2.2.2 Sample collection

This study was based on two field seasons, the first from May 30th to September 18th, 2017 and the second from May 8th to September 26th, 2018. During these time frames, weekly grab samples were collected from the secondary lagoon and from each well of the pilot filter (1W, 2W, 3W and 4W). Table 3 summarizes the main information regarding sample collection throughout the study.

Table 3 – Sampling details of 2017 and 2018 field seasons

	Season 2017	Season 2018
Filter start-up	May 30 th	May 22 nd
Sampling start date	May 30 th	May 8 th
Sampling end date	September 18 th	September 26 th
Total duration of season (d)	111	127
Number of sampling events	17	21
Pump failure sampling dates (d after filter start-up)	7, 21, 58, 65 and 111	28, 34, 100, 105 and 113

Filter start-up refers to the day on which the pump started to deliver wastewater to the filters; in both years this occurred in late May. In 2018, influent sampling started two weeks prior to filter start-up, so that a better characterization of the shoulder season could be obtained. Sampling events were named according to the time count (d) after filter start-up, with the latter representing day zero.

Pump failures occurred on five sampling dates in both years. On these days, the pump that delivered secondary wastewater to the filters was off. Common reasons for

that included: 1) Power outages, which shut down the timer that controlled the pump and consequently interrupted water delivery to the beds; 2) Malfunction of the drainage pump that released treated effluent from EW into the secondary overflow lagoon, forcing an emergency shutdown of the main pump; and, 3) Low water levels in the secondary lagoon. Field samples collected on pump failure days were more prone to contamination, as the effluent might not have been fresh or as there might have been backflow between the four holding tanks and EW.

Backflow problems were more frequent in 2018 and sometimes even occurred when the main pump was working. A combination of two factors contributed to this:

1) Possible clogging along the pipe connecting EW and holding tanks, which reduced the rate at which EW was filled, favouring backflow; and, 2) Inconsistent performance of the emptying pump: occasionally it did not reach full power, therefore slowing down the drainage of EW and forcing water back to the wells.

Holding tank 4W was affected the most by these phenomena, with the water becoming scummy and containing blue-green algae. To minimize sample contamination due to backflow issues, sampling had to be adapted on five days in 2018. Instead of sampling from inside the wells, tanks were pumped out and fresh effluent was collected. This happened on days 21, 28 and 34 (only 4W was pumped out), as well as on days 43 and 51 (all four wells were pumped out), whereas in 2017 all samples were collected from inside holding tanks.

At the end of the first field season (September, 2017), the upper layer of both sand filters (1W and 4W) was replaced with new sand. These sections consisted mostly of concretious sand blocks that developed during the treatment season. The removal of

these layers was performed to evaluate if concretion would reoccur in the following year. Some of the blocks were taken to the laboratory, so that formation and constitution of concreted sands could be further investigated.

2.2.3 Sample analysis

Influent (secondary lagoon wastewater) and treated effluents (collected from 1W, 2W, 3W and 4W) were analysed for pH, temperature (°C), electrical conductivity (mS/cm), total alkalinity (mg CaCO₃/L), total and volatile suspended solids (mg/L), total and dissolved COD (mg O_2/L), total phosphorus and orthophosphate (mg $PO_4^{-3}-P/L$), ammonium (mg NH₄⁺–N/L), nitrite (mg NO₂⁻–N/L) and nitrate (mg NO₃⁻–N/L), and metal content (mg/L), in terms of Ca, K, Mg and Na. Temperature and pH were measured on site, with a water quality meter (YSI Professional Plus 10, Xylem Inc.). Electrical conductivity was recorded using a benchtop conductivity reader (Accumet XL50, Fisher Scientific Inc.). Total alkalinity, TSS and VSS were determined according to the Standard Methods for the Examination of Water and Wastewater 2320B, 2540D and 2540E, respectively (APHA, 1999). Total and dissolved COD were analyzed using HACH TNT 821 kits (HACH, 2014), and TP was measured following the HACH TNT 843 procedure (HACH, 2017). Nitrogen compounds were analyzed with flow injection analysis – FIA (QuikChem 8500 series 2 FIA System with ASX-410 series Autosampler, Lachat Instruments), whereas metal concentrations were quantified via inductively coupled plasma mass spectrometry - ICP-MS (Varian 725-ES ICP-OES with Varian SPS 3 Autosampler, Agilent Technologies Inc.). Orthophosphate was analyzed with FIA in 2017 and ICP-MS in 2018.

Field flow measurements were performed during the two sampling seasons by timing wastewater delivery to filter beds. This was done so that the proportional flow distribution could be assessed. In 2018, the secondary lagoon water level was monitored on every sampling event, using a wooden measuring stick. Level measurements were always taken at the same location: next to where the main pump was located. In the same year, biomass growing on top of filter beds was harvested, dried for one week (at approximately 35°C) and weighed, so that N and P removal due to plant uptake could be estimated. In 2017, plant biomass was not quantified, just periodically removed from the filter's surface. As for mass balances, these were performed for TSS, VSS, total and dissolved COD, N content, TP and orthophosphate. The contaminant retention within filter cells was calculated according to Eq. 2. This equation assumes that there were no interruptions in wastewater delivery due to pump failures, with treated volumes of 5.5 x 10⁵ L/bed/season and 6.3 x 10⁵ L/bed/season in 2017 and 2018, respectively.

$$M_{\text{retained}} = (C_{\text{lagoon}} - C_{\text{well}}) \times V$$
 (Eq. 2)

Where: $M_{retained}$ = mass retained within the filter bed (g); C_{lagoon} = influent concentration (g/L); C_{well} = effluent concentration (g/L); V = wastewater volume treated by the filter (L).

2.2.4 Statistical analysis: Fisher-Pitman permutations

Fisher-Pitman permutations are used to check if distributions of a numeric response variable in two independent groups are equal against shift alternatives (Neuhäuser & Manly, 2004). This test was used to verify if two separate filter beds, containing equivalent media, differed significantly in terms of contaminant removal. Sand and glass filters were also contrasted against each other. A two-sided $oneway_test$ (R Software v.3.5.2, The R Foundation for Statistical Computing) for an asymptote distribution was performed three times, comparing: 1) Sand wells 1W and 4W; 2) Glass wells 2W and 3W; and, 3) Sand and glass wells (bed duplicate averages). The statistical null (H_0) and alternative hypotheses (H_a) were as follows, with p-values evaluated at the 95% confidence level:

- ✓ H_0 The true difference in means is equal to zero (μ = 0); therefore, the distributions of the two groups are equal against shift alternatives.
- ✓ H_a The true difference in means is not equal to zero ($\mu \neq 0$); therefore, the distributions of the two groups are not equal against shift alternatives.

2.3 Investigation of concretious sand

Several experiments were carried out to investigate composition and formation of concreted sands formed on the surface of sand filter beds. These included physical characterization tests, filter media washes, protein assays, lipid extraction, X-ray diffraction (XRD), dissolution tests and column experiments. Analyzed concreted sands were retrieved from beds 1W and 4W in 2017, after the end of the first field season.

2.3.1 Physical characterization of concretious sand blocks

Some of the concreted sand formations removed from the pilot filter were filed down to smaller blocks (Figure 3a). The physical characterization of the latter followed the methodology described in section 2.2.1. Lateral body filler walls were added to the blocks for permeability testing, to induce vertical water percolation through the brick (Figure 3b). Compressive strength of concreted sand was also tested. For this experiment, upper and lower block surfaces were straightened with body filler (Figure 3c). Duplicates with similar cross-sectional areas and depths, the latter varying between 2 and 5 cm, were compressed with a hydraulic press (Instron 300 DX Satec series, Illinois Tool Works Inc.) (Figure 3d), until rupture was achieved. Test configuration was so that breakage would occur within two minutes after compression start.

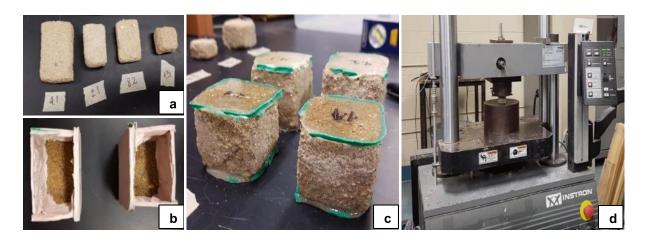


Figure 3 – Filed concretious sand blocks (a); blocks for permeability testing (b); blocks for compressive strength testing (c); experimental set-up of compressions tests (d).

2.3.2 Filter media washes, protein assay, lipid extraction and XRD analysis

Media washes were performed to enable elemental analysis and determination of protein and lipid content of filter materials. Washes were carried out for: 1) Concreted sand (powder); 2) Virgin sand, retrieved from the outlet of beds 1W and 4W; and, 3) Glass, extracted from both outlet (virgin glass) and inlet of cells 2W and 3W. Virgin filter media per se were not available. Sand and glass at bed outlet received little or no wastewater flow and were therefore used to characterize virgin media. Four different wash liquids were employed: deionized water (DI water), 0.2 M hydrochloric acid (HCI), 0.2 M sodium hydroxide (NaOH), and organic solvent (1:1 mixture of chloroform and cyclohexane).

Raw media samples were mixed for 4 min with a mortar and pestle, then sieved through a #200 mesh (0.074 mm). The powder yield obtained for each filter material was recorded (g filter media ground/g powder produced). Washes were performed in triplicates, using approximately 1 g of powder and 10 mL (base, organic solvent) or 15 mL (acid, water) of wash liquid. Containers with powder and wash liquid were inverted for 2 min and left sitting for 1h before being drained through 0.45 µm filters.

Base-wash filtrates were analyzed using a Bradford assay (Bradford, 1976) modified for the estimation of soluble protein in supernatant. Lipid extraction was accomplished with organic solvent washes: filtrates were evaporated and residue weights determined. Crystalline structures of virgin and concreted sands were qualitatively analyzed with X-ray diffraction. ICP-MS analysis was performed on water and acid filtrates for Ca, Cu, K, Mg, Na and P levels. Nitrogen species in water and acid filtrates of virgin and concreted sands were determined using FIA analysis.

Concentrations (mg/L) obtained from the elemental analyses were converted to mass yields (µg/g) (Eq. 3), and molar ratios of Ca:P and Mg:P were calculated.

$$Ratio_{mass} = \frac{(Dil \times ICP_{conc} \times Vol_{wash_liquid})}{Yield_{powder} \times Mass_{powder}}$$
(Eq. 3)

Where: $Ratio_{mass}$ = mass ratio (µg/g filter media); Dil = dilution factor; ICP_{conc} = concentration obtained from the ICP-MS analysis (mg/L); Vol_{wash_liquid} = volume of wash liquid added (mL); $Yield_{powder}$ = powder yield (g filter media ground/g powder produced); $Mass_{powder}$ = mass of powder washed (g).

2.3.3 Block dissolution experiments

This experiment consisted of 8 runs, in which virgin and concreted sands were washed with DI water and citrate buffer at pH 4, 5 and 6 (Gomori, 1955). The objectives of the test were: 1) To evaluate how much acidity was necessary to completely dissolve concretious sand; and, 2) To obtain effluent composition in terms of Ca, Mg and P (ICP-MS analysis). The experimental set-up is shown in Figure 4. Each sand sample was treated with approximately 4.5 L of buffer, which were pumped (Little Giant 505000 5-MSP, Franklin Electric Co. Inc.) drop-wise at a rate of 3.2 mL/min onto the surface of the specimen. The wash liquid percolated through the sand and was collected in a container below. At the end of each wash, effluent pH was recorded, and 50 mL effluent samples were collected, passed through 0.45 µm filters, acidified with 0.25 mL of 70% nitric acid (HNO₃) and stored at 4°C.



Figure 4 – Experimental set-up of the block dissolution test.

Similarly to filter media washes, effluent mass yields (mg/g) and molar ratios were calculated. P yields of virgin and concreted sands were used to estimate P retention in sand filter beds according to two scenarios. Scenario 1 was based on two assumptions: 1) The concretious layer can be approximated by semi-cylinders that form around the two flow delivery pipes (at a radius of 15 cm), over the entire length of the bed (10 m) (Figure 5); and, 2) Virgin sand occupies the remaining volume of the bed. Within this context, concreted sand represented 3.4% of the volume of a single sand filter cell. Scenario 2 considered that the entire bed consisted of concreted sand.

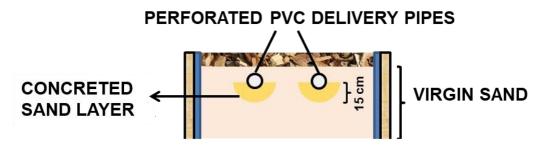


Figure 5 – Side cut of the upper surface of a sand filter cell, illustrating the concretious layer that forms around the flow delivery pipes and extends throughout the entire length of the bed. **Source:** The author.

2.3.4 Column experiments

Column testing was carried out to investigate if concretion would occur in glass filters with similar particle-size distribution than that of sand. The glass used in this experiment was very fine crushed, with an ES of 0.19 mm and a UC of 2.9. Sand ES and UC values were 0.17 mm and 3.13, respectively. The experimental set-up comprised three plexiglass columns (height: 25 cm; inner diameter: 15.5 cm): one control containing virgin sand (#1) and two replicates filled with fine glass (#2 and #3). Secondary wastewater lagoon effluent – brought from the Dunnottar facility – was added drop-wise onto the surface of each column, through gravity flow, which resulted in slightly different flow rates to each column (Table 4).

The area where the wastewater reached the filter medium was covered with gravel, to minimize drop-surface impact and avoid flow bypass. Influent percolated through a 15 cm filter layer before being drained by a valve at the bottom of the column. Treated effluent samples (50 mL) were collected periodically, filtered (0.45 µm), acidified (0.25 mL of 36.5 - 38% HCl) and submitted to FIA analysis. All columns were equipped with a fake bottom — a round perforated plexiglass plate located at approximately 2 cm from the base of the column. The fake bottom was covered with a 15 µm stainless steel mesh, serving as a support for the filter media layer. The experiment had a duration of 24 days, running from August 28th to September 20th, 2018. Figure 6 and Table 4 contain details regarding column experimental configuration.





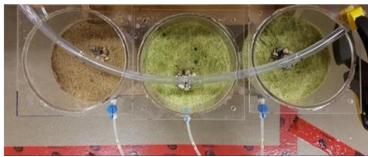


Figure 6 – Experimental layout of the sand and fine glass columns treating secondary wastewater lagoon effluent.

Table 4 – Information regarding experimental set-up of sand and glass columns used to investigate concretion in fine glass filter media

Column		Filter Media		Average flow	Total volume
C C C C C C C C C C C C C C C C C C C	Material	Weight (kg)	Height (cm)	(L/day)	treated (L)
#1	Virgin sand	4.6	14	10	235.44
#2	Fine glass	4.2	16	8	188.64
#3	Fine glass	4.1	16	7	159.12

CHAPTER 3 – RESULTS

Results were divided into two sections. The first refers to pilot-scale filter characterization and performance. The second relates to sand concretion phenomena.

3.1 Pilot-scale filter

3.1.1 Characterization of the filter media

Particle-size distribution and physical properties of sand and crushed glass used in Dunnottar's pilot filter are presented in Table 5 and Table 6, respectively. Sieve analysis of virgin media indicated that glass had an effective size 21 times greater than that of sand. Most of the sand fell between 0.075 and 4.75 mm, while 87% of the glass was above 4.75 mm. Glass media was more stratified, with a UC value exceeding sand by one unit. Bulk and particle densities, as well as void ratio and porosity, were similar in both media. Permeability, on the other hand, was 8 times faster in the virgin glass.

Table 5 – Particle-size distribution of the sand and glass filter media contained in the pilot-scale system

		UC	ES	Fractions (%)				
			(mm)	< 0.075 mm	0.075 - 4.75 mm	> 4.75 mm		
Virgin sand	Avg.	3.13 <i>0.04</i> 2	0.17 0.004	2.78 0.205	97.19 <i>0.162</i>	0.03 0.045		
Virgin glass	Avg. SD	4.18 1.678	3.59 1.666	0.84 <i>0.408</i>	12.52 <i>4.6</i> 36	86.64 5.019		

^{&#}x27;Virgin' indicates that the material was retrieved from bed outlet, close to collection wells.

Table 6 – Physical properties of the sand and glass filter media contained in the pilot-scale system

	Bulk Density		F		Porosity	Void Ratio			
		(g/cm³)	(g/cm ³)	(cm/s)	(%)				
Virgin sand	Avg.	1.82	2.53	0.015	27.91	0.39			
viigiii odiid	SD	-	0.050	0.002	-	-			
Virgin glass	Avg.	1.77	2.43	0.123	26.97	0.37			
	SD	-	0.079	0.046	-	-			
Absence of standard deviation means that replication was not performed.									

3.1.2 Nutrient uptake by plants growing on filter surface

Approximately 11 kg of plant biomass were harvested from filter beds in 2018 (Table 7), including thistles (*Cirsium spp.*), willow shrubs (*Salix spp.*), grass (Poaceae) and dandelions (*Taraxacum spp.*). Willows and thistles were found in greater abundance, with grass occurring close to flow measurement pits. Increased plant growth was observed on beds 1W and 4W, accounting for 54% and 24% of the total biomass collected, respectively. In cell 1W, plant uptake would represent a removal of 12 g P and 350 g N over the season.

Table 7 – Biomass harvest in 2018 and dry weight estimation of phosphorus and nitrogen uptake by plants

	Biomass (kg)	P uptake (g)	N uptake (g)
1W _{sand}	5.8	11.6	348.7
$2W_{\text{glass}}$	1.6	3.1	94.4
$3W_{\text{glass}}$	0.7	1.4	41.1
4W _{sand}	2.6	5.2	156.6
Total	10.7	21.4	640.8

Calculations consider that P and N make up approximately 0.2% (Schachtman et al., 1998) and 6% (Mitra, 2017) of a plant's dry weight, respectively.

3.1.3 Filter performance

This section describes filter performance in terms of physicochemical parameters (pH, EC, temperature and alkalinity), metal content (Ca, Mg, Na and K), suspended solids, chemical oxygen demand, N and P species. Fisher-Pitman permutation *p*-values are presented in Appendix 1.

3.1.3.1 Physicochemical parameters

Figure 7 compares influent and effluent pH, EC, alkalinity and temperature throughout both sampling seasons. Influent pH values were as high as 9.6 in 2017, with a mean of 8.3 in the second year. In 2018, effluent pH averages were 7.7 (glass) and 7.4 (sand) – a significant difference according to Fisher-Pitman permutations (Appendix 1). Alkalinity removal occurred occasionally within sand and glass filter beds, reaching up to 23% and 14% in 2018, respectively. Temperature and EC were practically equal in both effluents (*p*-values > 0.05, Appendix 1), not differing much from influent values.

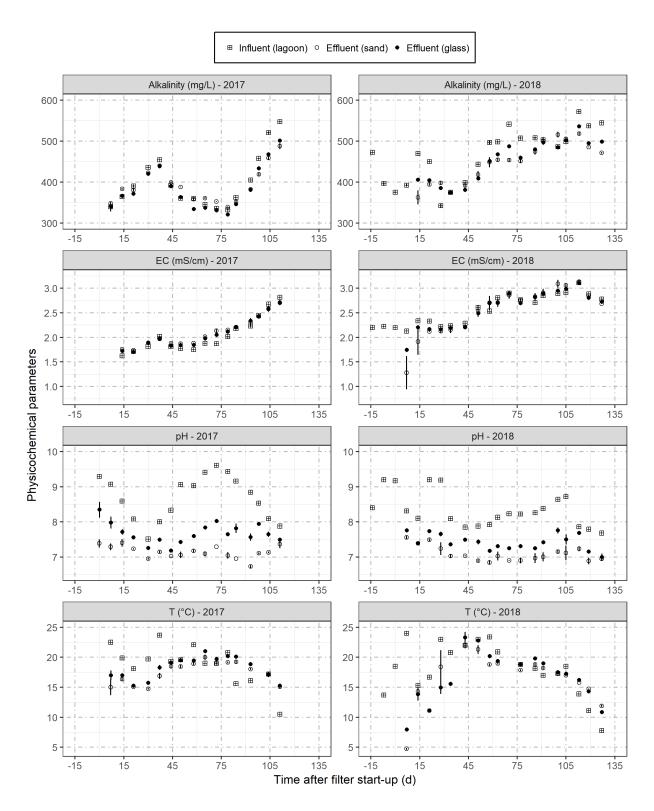


Figure 7 – Alkalinity (mg/L), electrical conductivity (mS/cm), pH and temperature (°C) of influent and treated effluents, the latter represented as means of the sand (1W, 4W) and glass (2W, 3W) filter bed duplicates.

3.1.3.2 Metal content

Figure 8 shows influent and effluent concentrations of Ca, Mg, Na and K in season 2018. Insufficient datapoints are available for the first year, as metal content monitoring was discontinuous and started late in the season. Results indicate that metal levels did not differ substantially between influent and effluent samples in 2018, except for Ca concentrations at 28 and 34 days after filter start-up. Influent Ca concentrations varied between 50 and 100 mg/L, with an average of 86 mg/L.

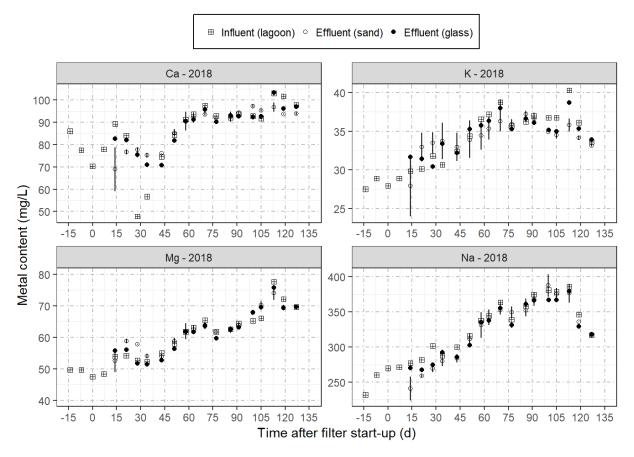


Figure 8 – Season 2018 levels of Ca, K, Mg and Na of influent and treated effluents, the latter represented as means of the sand (1W, 4W) and glass (2W, 3W) filter bed duplicates.

3.1.3.3 Suspended solids

Influent and effluent suspended solids are shown in Figure 9. Incoming TSS reached a maximum of 130 mg/L, with seasonal averages of 33 mg/L (2017) and 47 mg/L (2018). Effluent TSS and VSS levels remained close to zero. Removal efficiencies were above 90% in all beds, with no significant difference between sand and glass (*p*-values > 0.05, Appendix 1). Mass retentions within filter cells were about 40% higher in the second year (Table 8). Influent VSS/TSS was 0.6, whereas effluent ratios varied between 0.5 - 0.9.

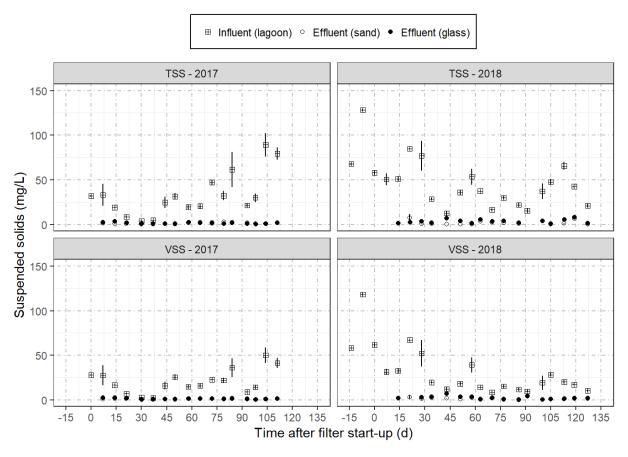


Figure 9 – Total and volatile suspended solids (mg/L) of influent and treated effluents, the latter represented as means of the sand (1W, 4W) and glass (2W, 3W) filter bed duplicates.

Table 8 – Retention of TSS and VSS (kg) within sand and glass filters, expressed for both bed duplicates and individual filter cells

		Removal (%)		Influent	Retention within filter beds (
		Sand	Glass	(kg)	$1W_{\text{sand}} \\$	$2W_{\text{glass}}$	$3W_{\text{glass}}$	$4W_{\text{sand}} \\$	
TSS	2017	96	96	18.1	17.2	17.2	17.3	17.5	
133	2018	94	91	26.0	24.7	23.8	23.8	24.4	
VSS	2017	94	94	11.3	10.6	10.6	10.6	10.7	
V 33	2018	96	92	16.2	15.6	15.0	14.8	15.4	

3.1.3.4 Chemical oxygen demand

Figure 10 portrays influent and effluent concentrations of total and dissolved COD. Lagoon total COD values are unavailable in early 2017 (measurement errors), whereas dissolved COD was recorded just in the second half of the season. Influent total COD was as high as 400 mg/L in 2018, with particulate COD above 100 mg/L within the first month after filter start-up (Figure 11). Effluent particulate COD was detected only until day 34. After that, total COD averages were approximately 45 mg/L and 61 mg/L in sand and glass filter effluents, respectively. Removal was about 17% (total COD) and 29% (dissolved COD) lower in glass filters (Table 9), with sand and glass differing significantly with respect to soluble COD removal (Appendix 1).

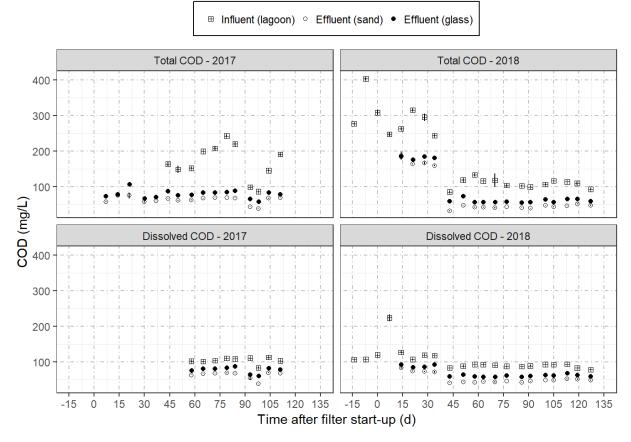


Figure 10 – Total and dissolved COD (mg/L) of influent and treated effluents, the latter represented as means of the sand (1W, 4W) and glass (2W, 3W) filter bed duplicates.

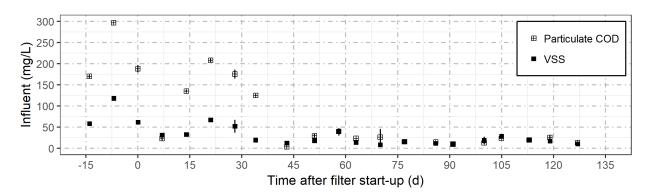


Figure 11 – Particulate COD and VSS concentrations (mg/L) of the secondary wastewater lagoon in season 2018.

Table 9 – Retention of total and dissolved COD (kg) within sand and glass filters, expressed for both bed duplicates and individual filter cells

		Removal (%)		Influent	Retention within filter beds (
		Sand	Glass	(kg)	$1W_{\text{sand}}$	$2W_{\text{glass}}$	$3W_{\text{glass}}$	$4W_{\text{sand}} \\$
COD _{total}	2017	63	53	93.4	59.1	50.6	49.1	57.7
CODIOIAI	2018	55	45	102.5	57.7	47.0	46.0	54.7
CODdissolved	2017	39	26	57.5	21.6	15.3	14.3	23.4
OODaissoivea	2018	48	34	65.5	32.4	22.7	22.3	31.1

3.1.3.5 Nitrogen

Influent and effluent N levels are displayed in Figure 12, in which values below detection level – 0.25 ppm in 2017 and 1 ppm in 2018 – were represented as zeros. Incoming NH₄+–N differed between the two seasons, with average concentrations of 1.7 mg/L (2017) and 7.5 mg/L (2018). Effluent ammonium concentrations followed similar trends in both years, with glass filters removing on average 9% less than sand (Table 10). In 2018, effluent nitrate levels fluctuated considerably until day 75, reaching maximums of 10 mg/L. Effluents from sand bed 4W showed especially high NO₃-–N content (Appendix 2). Plant uptake accounted for 6% and 2% of the N removal within sand and glass filters over the season, respectively (Table 11).



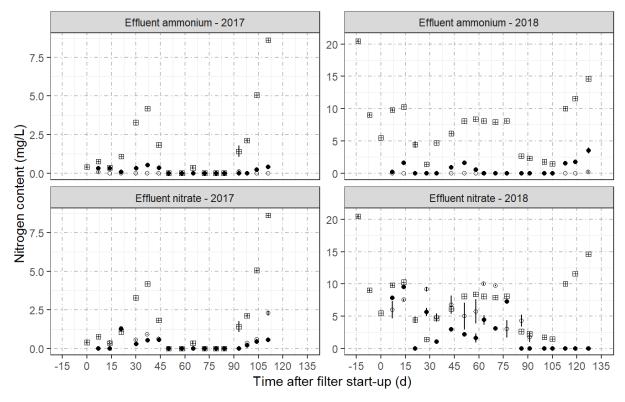


Figure 12 – Influent ammonium contrasted with nitrate and ammonium levels (mg/L) of treated effluents, the latter represented as means of the sand (1W, 4W) and glass (2W, 3W) filter bed duplicates.

Table 10 – Retention of NH_4^+ –N (g) within sand and glass filters, expressed for both bed duplicates and individual filter cells

		Removal (%)		Influent	Removal within filter beds (
		Sand	Glass	(g)	$1W_{\text{sand}}$	$2W_{\text{glass}}$	$3W_{\text{glass}}$	$4W_{\text{sand}} \\$
NH ₄ +–N	2017	99.4	90.8	933	926	850	845	928
	2018	99.8	90.4	4214	4214	3717	3902	4201

Table 11 – Percent NH₄+–N removal by plant biomass and other mechanisms within filter beds over seasons 2017 and 2018, expressed for both bed duplicates and individual filter cells

		NH ₄ +–N removal over the season (%)							
		Sand	Glass	$1W_{\text{sand}}$	$2W_{\text{glass}}$	$3W_{\text{glass}}$	$4W_{\text{sand}} \\$		
2017	Plant biomass Other mechanisms	27 73	8 92	38 62	11 89	5 95	17 83		
2018	Plant biomass	6	2	8	3	1	4		
2018	Other mechanisms	94	98	92	97	99	96		

Removal by plant biomass was based on values presented in Table 7. Season 2017 percentages represent estimates, as no biomass harvest was performed that year. These removal rates were calculated based on the 2018 biomass harvest instead.

3.1.3.6 Phosphorus

Figure 13 contrasts influent and effluents in terms of TP and orthophosphate. Influent early-season TP data are limited in 2017, due to measurement errors. Lagoon P levels reached peaks of 6 mg/L in 2018, with negligible particulate P after one week of operation. Both seasons showed insignificant differences between effluent TP and orthophosphate at the 95% confidence level (Appendix 3). Table 12 indicates that glass removed 57% (TP) and 65% (orthophosphate) less than sand in 2018 – a significant difference according to Fisher-Pitman permutations (Appendix 1). Percent P removal by plant biomass, concreted sand layers and filter beds is described in Table 13. P retention within concreted sand layers (250 g P, Table 20) accounted for approximately 50% of the difference in P removal between sand and glass filters (Table 12).



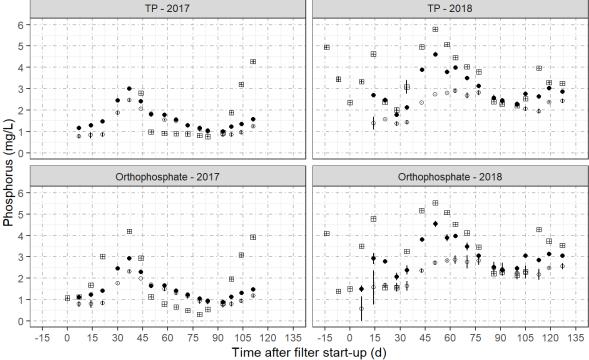


Figure 13 – Total phosphorus and orthophosphate (mg/L) of influent and treated effluents, the latter represented as means of the sand (1W, 4W) and glass (2W, 3W) filter bed duplicates.

Table 12 – Retention of TP and orthophosphate (g) within sand and glass filters, expressed for both bed duplicates and individual filter cells

		Remo	val (%)	Influent	Retenti	Retention within filter beds (g)			
		Sand	Glass	(g)	$1W_{\text{sand}}$	$2W_{\text{glass}}$	$3W_{\text{glass}}$	$4W_{\text{sand}} \\$	
TP	2017	21	6	917	250	111	5	129	
	2018	37	16	2224	891	379	329	739	
Orthophosphate	2017	28	12	942	348	158	73	186	
	2018	35	12	2139	834	306	208	645	

Season 2017 mass balances may not provide an accurate description of real TP retention, as early-season TP data are unavailable.

Table 13 – Percent P removal by plant biomass, concreted sand layers and filter beds in season 2018, expressed for both bed duplicates and individual filter cells

			P re	moval (%) - Seaso	n 2018	
		Sand	Glass	$1W_{\text{sand}}$	$2W_{\text{glass}}$	$3W_{\text{glass}}$	$4W_{\text{sand}} \\$
TP	Plant biomass	1.0	0.6	1.3	0.8	0.4	0.7
	Concreted layer	30.6	-	28.0	-	-	33.8
	Bed	68.3	99.4	70.7	99.2	99.6	65.5
	Plant biomass	1.1	0.9	1.4	1.0	0.7	0.8
Orthophosphate	Concreted layer	33.8	-	29.9	-	-	38.7
	Bed	65.1	99.1	68.7	99.0	99.3	60.5

Removal by plant biomass was based on values presented in Table 7.

Removal within concreted layers is only applicable to sand filter beds. Percentages were based on the standard retention of 250 g P within the concreted layer of a single sand filter cell (Table 20).

In the case of sand filters, 'bed' refers to the non-concreted media layer (i.e. virgin sand).

3.2 Investigation of concretious sand

Concretion occurred in both sand filters (1W, 4W), during the two field seasons (Figure 14). In 2018 concretion was first detected in 1W, two weeks after filter start-up.



Figure 14 - Concretious sand formations in cells 1W and 4W of the pilot-scale filter.

Concreted sand occupied the area of half-cylinders that formed around the two wastewater delivery pipes in each cell. The phenomenon was observed within a radius of 15 cm of the tubing, along the entire length of the beds (10 m).

3.2.1 Physical characterization of concretious sand blocks

Physical characteristics of concreted sand blocks are shown in Table 14. Results indicate that bulk and particle densities are equal in concretious sand. Compressive strength of concreted sand varied from 2 to 9 MPa (Appendix 4), with an average of 4.6 MPa.

Table 14 – Physical properties of concreted sand blocks retrieved from the pilot-scale sand filter cells 1W and 4W

		Bulk Density	Particle Density	Permeability	Porosity	Void	
		(g/cm ³)	(g/cm ³)	(cm/s)	(%)	Ratio	
Concreted sand	Avg.	1.61	1.68	0.013	4.06	0.04	
(block)	SD	0.214	0.019	0.002	-	-	

3.2.2 Filter media washes, protein assay, lipid extraction and XRD analysis

Results of the water and acid washes are presented in Table 15. Both washes demonstrated that glass Ca, K, Mg, Na and P levels were higher at bed inlet. Sand water washes showed that concreted media held approximately 1 µg/g more Ca and Mg than virgin samples. Acid-washed concreted sand contained significantly more Ca (*p*-value 0.0070) and P (*p*-value 0.0101) than virgin sand. Both water and acid washes

indicated negligible Cu levels in all four media. Sand NH₄⁺ and NO₂⁻ yields were low in both washes, with virgin and concretious sands showing similar NO₃⁻ content.

Table 15 – Effluent Ca, Cu, K, Mg, Na, P, NH₄+, NO₂- and NO₃- yields (μg/g) of virgin sand, concreted sand, virgin glass and inlet glass washed with DI water and 0.2 M HCl

Water wash	Yield (μg/g)									
(DI Water)		Ca	Cu	K	Mg	Na	Р	NH_4^+	NO_2^-	NO ₃ -
Virgin sand	Avg.	10.6	0.04	3.6	2.4	5.5	0.3	0.2	0.2	5.9
	SD	0.03	0.01	0.42	0.02	0.14	0.03	0.03	0.02	0.43
Concreted sand	Avg.	11.2	0.05	0.9	3.7	5.4	0.2	0.6	0.2	6.5
	SD	0.29	0.01	0.1	0.13	0.09	0.06	0.07	0.03	0.31
Virgin glass	Avg.	1.1	0.01	8.0	0.4	0.9	0.2		_	
	SD	0.1	0.002	0.08	0.03	0.03	0.02			
Inlet glass	Avg.	4.4	0.01	3.9	1.5	5.5	0.9		_	
	SD	1.6	0.002	0.11	0.61	0.15	0.04			
Acid wash Yield (µg/g)										
(0.2 M HCI)		Ca	Cu	K	Mg	Na	Р	NH ₄ ⁺	NO_2^-	NO ₃ ⁻
Virgin sand	Avg.	1772.1	0.1	4.1	353.4	3.4	0.6	0.3	0.1	2.9
	SD	158.6	0.01	0.34	28.82	0.98	0.2	0.24	0.01	0.11
Concreted sand	Avg.	2712.8	0.2	2.3	148.7	6.3	17.3	0.5	0.2	3.2
	SD	237.41	0.02	0.06	23.13	1.11	2.97	0.32	0.03	0.14
Virgin glass	Avg.	274.9	0.1	2.2	68.6	1.3	1.2		_	
	SD	17.38	0.01	0.1	3.61	0.07	0.07			
Inlet glass	Avg.	947.4	0.2	6.9	271.7	5.2	3.9		_	
	SD	101.41	0.04	1.61	24.62	0.9	0.99			

^{&#}x27;Virgin' indicates that the media was retrieved from bed outlet, close to collection wells.

^{&#}x27;Inlet' indicates that the media was retrieved from bed inlet, close to where the wastewater first reaches the filter. Glass samples were not analyzed for nitrogen species.

Effluents obtained from filter media washes were also analyzed for Ca:P and Mg:P molar ratios (Table 16). Acid washes showed higher ratios in virgin sand and inlet glass. Sand water washes indicated greater Ca:P and Mg:P in concreted sand, whereas water wash ratios of virgin and inlet glass were almost equal.

Table 16 – Effluent molar ratios (Ca:P and Mg:P) of virgin sand, concreted sand, virgin glass and inlet glass washed with DI water and 0.2 M HCI

	Wate	r wash	Acid wash		
	Ca:P	Mg:P	Ca:P	Mg:P	
Virgin sand	29.1	11.0	2275.4	748.3	
Concreted sand	40.4	21.7	121.0	10.9	
Virgin glass	4.0	2.6	175.5	72.2	
Inlet glass	3.7	2.1	189.0	89.4	

Table 17 – Filter media protein and lipid content, obtained from Bradford Assay and organic solvent wash (1 C₆H₁₂: 1 CHCl₃), respectively

	Protein	(µg/g)	Lipids (mg/g)		
	Avg.	SD	Avg.	SD	
Virgin sand	0.90	0.26	0.20	0.098	
Concreted sand	0.44	0.30	0.30	0.050	
Virgin glass	0.65	0.19	0.02	0.005	
Inlet glass	0.71	0.12	0.19	0.051	

Filter media protein and lipid content were low in all four samples (Table 17). Protein levels of virgin and inlet glass were equivalent, whereas virgin sand contained twice as much protein as concreted media. Lipid yields were lowest in virgin glass, with virgin sand and inlet glass both containing 0.2 mg lipids/g. Lipid levels in concreted sand were 50% higher than in virgin samples.

XRD analysis of virgin and concretious sands (Appendix 5) showed that samples did not differ significantly with regards to crystalline composition. Both sands contained primarily dolomite (CaMg(CO₃)₂), with minor quartz (SiO₂), calcite (CaCO₃), calclacite (Ca(CH₃COO)CI.5H₂O) and albite (NaAlSi₃O₈). Dolomite was less abundant in virgin samples when both sands were similarly scaled to the dolomite 100% peak (Appendix 6). Calcite counts were similar in both sands (Appendix 6), with magnesium-rich calcite ((Ca,Mg)(CaCO₃)) detected exclusively in concreted sands (Appendix 7).

3.2.3 Block dissolution experiments

Dissolution experiments showed that concreted sand blocks washed with citrate buffer at pH 4 and 5 achieved complete disfiguration after 7h of treatment, requiring less than 1.5 L of solution (Figure 15). The concreted block treated with pH 6 buffer took more than 12h to present signs of deformation.



Figure 15 – Temporal dissolution progression of a concreted sand block treated with citrate buffer at pH 4.

All four dissolutions indicated higher Ca, Mg and P levels in concreted sands (Table 18), with significant differences between virgin and concreted samples (Appendix 8). Phosphorus yields obtained in pH 4 and 5 dissolutions of concretious sand were similar, with an average of 0.23 mg P/g concreted sand.

Table 18 – Effluent Ca, Mg and P yields (mg/g) of virgin and concreted sands washed with DI water and citrate buffer at pH 4, 5 and 6

		Yield (mg/g)					
Wash	Sand	Са		Mg		Р	
		Avg.	SD	Avg.	SD	Avg.	SD
DI Water	Virgin	0.33	0.002	0.03	0.001	0.01	0.002
Di Watei	Concreted	0.76	0.019	0.06	0.000	0.02	0.001
pH 4	Virgin	55.92	0.106	6.89	0.015	0.02	0.001
Pi i	Concreted	79.94	1.798	9.85	0.254	0.21	0.002
pH 5	Virgin	50.00	0.713	4.09	0.007	0.02	0.002
ριτο	Concreted	75.32	0.163	5.91	0.109	0.25	0.003
pH 6	Virgin	32.28	0.127	1.86	0.002	0.01	0.001
P110	Concreted	64.15	0.231	3.64	0.037	0.46	0.003

Table 19 – Effluent molar ratios (Ca:P and Mg:P) of virgin and concreted sands washed with DI water and citrate buffer at pH 4, 5 and 6

Sand	DI Water pH 4		4 pH 5		pH 6			
Junu	Ca:P	Mg:P	Ca:P	Mg:P	Ca:P	Mg:P	Ca:P	Mg:P
Virgin	48.2	6.8	2197.5	446.4	2204.1	297.6	1665.4	157.9
Concreted	31.4	4.0	301.3	61.2	230.7	29.8	106.9	10.0

In terms of molar ratios (Table 19), Ca:P and Mg:P were always lower in concretious sand. Virgin sand ratios of acid dissolutions were about one order of magnitude higher than those of concreted samples.

Yields obtained in the dissolution experiment (Table 18) were used to estimate the P retention within virgin and concreted layers of a 20 m³ sand filter bed (Table 20). In scenario 1, where concreted sand represents 3.4% of the filter volume, P retentions were similar for pH 4 and 5 dissolutions. Taking the average of these two washes, 0.66 kg P would be retained within virgin sand and 0.25 kg P would be stored in the concreted fraction. In scenario 2, which considers a cell of only concreted sand, retention varied from 6 to 15 kg P according to acid-based dissolutions.

Table 20 – Average P mass retention within a 20 m³ sand filter bed according to two different scenarios: 1) Cell consisting of both virgin (19.3 m³) and concreted (0.7 m³) sands; and, 2) Cell consisting entirely of concreted sand

Scenario 1	Bed volume (m³)	Avg. P mass	Avg. P mass retention within bed (kg)			
	(/	DI Water	pH 4	pH 5	pH 6	
Virgin sand	19.3	0.19	0.69	0.62	0.53	
Concreted sand	0.7	0.02	0.22*	0.28*	0.51	
Total	20.0	0.21	0.92	0.89	1.03	
Scenario 2	Bed volume (m³)	Avg. P mass retention within bed (kg)				
Joshan Z	Doa volanie (m)	DI Water	pH 4	pH 5	pH 6	

Scenario 1 - Sand filter cell consists of two fractions: 1) Concreted sand that forms around flow delivery pipes (R = 15 cm), over the entire length of the bed (L = 10 m); and, 2) Virgin sand occupies the remaining bed volume. Scenario 2 - Sand filter cell consists entirely of concreted sand.

0.60

6.60

8.12

20.0

Concreted sand

14.93

^{*}The average of these values was used as the standard P retention (250 g P) within the concreted layer of a single sand filter cell (Table 13).

3.2.4 Column experiments

Sand and fine glass columns were inspected for concretion after receiving Dunnottar's secondary lagoon wastewater for 24 days. The phenomenon was absent in all three reactors, with no evident filter media hardening. No phosphorus removal was observed within the columns, as indicated in Table 21.

Table 21 – Orthophosphate concentrations (mg/L) of treated effluents collected from virgin sand and fine glass columns treating secondary lagoon wastewater

	Influent P (mg/L)	Effluent P (mg/L)				
		#1 Virgin sand	#2 Fine glass	#3 Fine glass		
Aug 29 th , 2018	1.9	1.7	1.8	1.8		
Sep 2 nd , 2018	1.7	1.8	1.9	1.8		
Sep 5 th , 2018	1.9	1.9	2.1	2.1		
Sep 7 th , 2018	1.9	2.2	2.3	2.3		
Sep 10 th , 2018	1.9	2.5	2.5	2.5		
Sep 13 th , 2018	3.8	3.2	3.4	3.6		
Sep 17 th , 2018	3.8	3.8	3.8	3.8		
Sep 20 th , 2018	3.3	3.6	3.5	3.4		

Influent orthophosphate does not represent concentrations inside the feeding container on indicated dates. Values refer to secondary wastewater lagoon concentrations on the field day that corresponds to the batch being delivered to columns on the above listed dates.

CHAPTER 4 - DISCUSSION

4.1 Recapitulation of research purpose

The objective of this study was to evaluate and compare the performance of sand and crushed recycled glass in pilot-scale subsurface filters treating municipal lagoon effluent. One of the research aims was to broaden the knowledge of P removal within sand and glass filters, as very limited information existed on this subject in the literature. The scope of the study was later expanded to include the investigation of the development of concretious sand – an unexpected finding that could affect filter efficiency and maintenance.

4.2 Filter performance

4.2.1 Suspended solids

TSS removal was achieved equally well in sand and glass filters, with the latter exceeding previously reported treatment efficiencies of 75% (Horan & Lowe, 2007) and 79% (Hu & Gagnon, 2006). In spite of the high TSS retention within filter beds (Table 8), clogging was not observed. Backwash was therefore not necessary, facilitating filter operation and maintenance (Crittenden et al., 2012; Horan & Lowe, 2007). The provincial discharge limit of 25 mg TSS/L was met by both filters at all times. Effluent TSS levels remained stable despite fluctuations in the influent (Figure 9), indicating consistent removal in both media. This diverged from full-scale results obtained by Horan & Lowe (2007), where effluent quality depended on influent concentrations.

4.2.2 Chemical oxygen demand

Both sand and glass filters presented lower total COD removal within the first month of operation (Figure 10). Filter beds failed to deliver effluents free of non-soluble COD during that period. This occurred due to elevated particulate COD in the influent, which was on average 7 times higher at that time. First-month effluent BOD, estimated based on a BOD/COD ratio of 0.36 (Al-Hashimi & Hussain, 2013; Pirsaheba et al., 2015), would have exceeded the provincial guideline of 25 mg BOD/L.

Dissolved COD removal was also lower during this period. The establishment of microbial communities on filter media – a process that can take up to six months in colder climates (Hu et al., 2017) – was first induced at system start-up. At this point, moisture and nutritional substrate became available within filter beds, enabling biofilm development. Late spring temperatures were still relatively low (Figure 7), which not only retarded biofilm maturation (Lessard & Bihan, 2003) but also decreased oxidation rates within the film (Gray, 2004). This, combined with influent loads on average 1.5 times higher, resulted in reduced removal of soluble COD during the initial filter operation phase.

Crushed glass removed significantly less dissolved COD in comparison to sand. The biodegradable fraction of soluble COD is mostly metabolized by heterotrophic bacteria within the biofilm (Wentzel et al., 2003). Filter media particle size, therefore, plays an important role: the smaller the media, the greater the surface area available for biofilm growth (Lessard & Bihan, 2003; Morgenroth, 2008b). Given these considerations, soluble COD removal efficiencies were expected to be superior in sand media, which had an effective size 95% smaller than that of crushed glass. This

explains why Gill et al. (2011) and Hu & Gagnon (2006) – who compared media with similar ES – observed insignificant differences between sand and glass filters in terms of organic material removal.

4.2.3 Nitrogen

Influent NH₄+–N levels were on average 4.4 times higher in the second season, a result of increased sewage loads added to the primary lagoon until late May. During that time, the facility treated wastewater from both Dunnottar and Gimli. This might have led to different operating conditions, such as reduced retention times within primary and secondary stabilization ponds. In addition, N and P were substantially increased in the secondary lagoon, resulting in considerable eutrophication – a phenomenon that had not been observed in the previous year.

Sand filters performed better than glass in reducing NH₄+–N (Table 10), providing effluents without ammonium nitrogen in both seasons (Figure 12). Removal efficiencies were above 90% in both media, which agrees with values reported by Elliott (2001b) and Hu & Gagnon (2006). Effluents from glass media filters were in accordance with the federal guideline of 1.25 mg NH₃–N/L in 2017. The following year, the limit was exceeded during 1/3 of the time. This might be a reflection of increased influent NH₄+–N that season.

Results obtained in the first year showed that plant uptake was not the only N removal mechanism within filter beds (Table 11). Nitrification seemed to have contributed to NH₄+–N reductions in sand and glass-filtered effluents, which is in agreement with previous studies (Elliott, 2001b; Gill et al., 2011). Higher NH₄+–N

removal in sand filters could be a consequence of increased plant growth on beds 1W and 4W (Table 7) but might also indicate superior nitrification in sand media. Nitrification and media surface area are inversely proportional (Nakhla & Farooq, 2003). This would therefore favour the process within sand cells, due to higher biofilm densities. Low effluent NO₃-N concentrations in relation to influent NH₄+N suggest that denitrification also occurred (Laaksonen et al., 2017). Gaseous N was most likely produced at lower filter depths, as previously seen in glass beds (Gill et al., 2011); in anaerobic or anoxic biofilm fractions, which occur closest to the support medium (Gray, 2004; Lessard & Bihan, 2003); and within microniches inside filter media pores, especially in the sand (Cook et al., 2017).

Ammonium removal in the second season was probably achieved through the same aforementioned mechanisms. N data obtained that year were inconsistent and might have been compromised by adapted sampling methods (days 21 - 51) and backflow problems encountered along the season (see section 2.2.2). Backflow was caused by malfunction of the drainage pump located in the final emptying well (EW). The water exchange between EW and holding tanks could explain effluent NO₃⁻–N fluctuations and justify the especially high nitrate concentrations in 4W (closest to EW). Since the drainage pump was only replaced on day 105, backflow phenomena cannot be discarded as a factor until that day. This could account for highly variable NO₃⁻–N levels in 4W even after the troubleshooting phase.

4.2.4 Phosphorus

Effluent P was mostly found in the form of orthophosphates, which was expected. Both sand and glass filters failed to reduce P levels below the provincial discharge limit of 1 mg P/L, similarly to what was observed by Hu & Gagnon (2006) and Gill et al. (2011). In 2017, effluent P levels were sometimes higher than in the influent. The assumption is that part of the P removed early in the season – where influent TP was above 4 mg/L – was gradually released by filter beds later on. This would explain why effluent P slowly decreased while influent loads remained constant between days 50 and 90. In 2018, influent and effluent P were on average twice as high as in the previous year. This increase can be attributed to additional wastewater loads from Gimli, treated within Dunnottar's facility until late May that year. Sand media removed significantly more P than glass in 2018. This finding was consistent with Gill et al. (2011), who detected better removal in sand filters 72% of the time. This raised the hypothesis that P retention could be related to sand concretion phenomena, which might have been accountable for removal differences between sand and glass filters.

4.2.4.1 Investigation of concretious sand

The investigation of concreted sand relied on the assumption that media hardening occurred due to precipitation. Based on that premise, precipitate would occupy media voids and bind sand grains together, increasing compressive strength and possibly reducing permeability.

This hypothesis was supported by physical characterization and mechanical testing results. Virgin and concreted sands differed with regards to porosity and void

ratio. The latter were 7 and 9 times smaller in concreted samples, respectively, indicating a clear reduction in void space. This might explain the higher flow resistance observed in concreted samples, which presented permeability rates 14% lower than those of virgin sand. The average compressive strength of concreted media equaled ¼ of the strength of residential concrete (NRMCA, 2003). In one of the sand blocks this proportion reached ½.

Low protein and lipid levels (Table 17) suggested that the precipitate was not organic in nature, which seemed unlikely from the start. Concreted sands did not present any odour or dark colour, which would have been signs of organic clogging due to accumulation of bacterial biomass and suspended solids (Grace et al., 2016; Leverenz et al., 2009). Magnesium ammonium phosphate (NH₄MgPO₄·6H₂O, struvite) was discarded as a potential precipitate based on acid wash results (Table 15). NH₄+–N levels were below the minimum concentration of 1 mM for struvite precipitation (Ackerman et al., 2016) and molar proportions of 1:1:1 were not met.

Higher dolomite content in concreted sands (Appendix 6), in addition to complete absence of Mg-rich calcite in virgin samples (Appendix 7), indicated precipitation of these compounds within concreted layers. This was supported by block dissolution results (Table 18), which demonstrated consistently higher Ca and Mg in concretious sands. The presence of Mg⁺² in sand filters played an important role in carbonate precipitation within these systems. Magnesium ions were naturally available in the media and also found in the influent (Figure 8). This favoured the precipitation of Mg-rich calcite over CaCO₃ (Jenkins et al., 1971), besides enabling the formation of dolomite precipitates (Fukue et al., 2011).

Carbonate precipitation within sand beds certainly contributed to media concretion and increased compressive strengths, effects commonly reported in the literature (Abo-El-Enein et al., 2013; Al-Salloum et al., 2017; Cheng et al., 2013; Fukue et al., 2011). Acid washes and dissolution tests, however, suggested that concretion might not have been caused by carbonate precipitation alone. Both experiments showed significantly higher Ca and P levels in concreted samples, providing strong evidence towards calcium phosphate precipitation within concreted sand. Field conditions were favourable for this: 1) Sand filters contained an abundance of Ca+2 and Mg+2; and, 2) Wastewater pH levels were alkaline throughout both seasons (Figure 7). This combination is known to promote calcium phosphate precipitation within sand media (Arias et al., 2001; Aulenbach & Meisheng, 1988; Bubba et al., 2003; Prochaska & Zouboulis, 2006).

XRD analysis did not detect crystalline phosphates bound to concreted sands. A possible explanation is that calcium phosphates had not transitioned to crystalline phases yet, existing predominantly as amorphous compounds within concretious sands. This is a reasonable assumption, as the maturation process of these precipitates is relatively slow (Eanes & Meyer, 1977; Jenkins et al., 1971). To that end, sand concretion appears to be a result of both carbonate (dolomite and Mg-rich calcite) and calcium phosphate precipitation. Precipitation reactions and products, combined with the natural buffer capacity of sands (Fukue et al., 2011), might explain why the pH of sand filter effluents was consistently lower than that of glass (Figure 7).

4.2.4.2 Differences in P removal between sand and glass filters

The investigation of concreted media confirmed that it represented an important P sink in sand filters. Concreted layers retained 30 to 35% of the total P removed within sand cells (Table 13), a substantial amount considering that they occupied less than 4% of the total bed volume. This demonstrates that P was further removed within non-concreted media. These findings explain why retention in concreted sands (250 g P) did not account for the total difference in P removal between sand and glass filters.

Phosphate removal within sand filters seemed to have been predominantly accomplished through mineral precipitation with Ca and Mg compounds. Media mineralogy and alkaline wastewater conditions provided the right environment for that. Top layer concretion suggests that precipitate formation was highest close to the surface. Further precipitation could have taken place within non-concreted sand media, which might have become concreted over time.

Adsorption to sand grains and carbonate precipitates might also have contributed to P reductions. This mechanism, however, does not promote P removal in the long-term, as media saturation can be reached relatively quickly (Aulenbach & Meisheng, 1988; Bubba et al., 2003; Tofflemire & Chen, 1977). Arias et al. (2001), who studied P-sorption capacities of 13 different sands, demonstrated that P removal was mostly achieved via precipitation reactions with calcium. Gill et al. (2011) also indicated that phosphate removal within sand filters could not have been achieved by adsorption processes alone. These studies support the hypothesis of calcium phosphate precipitation as principal P removal mechanism in Dunnottar's sand filters.

Phosphorus removal in glass filters did not reach half the efficiency recorded in sand beds. This can be attributed to lower Ca content in glass media. Neither sand nor glass filters removed substantial amounts of Ca from influent wastewater (Figure 8), which indicates that the Ca consumed during P removal was primarily available from within the media. Decreased Ca availability in glass beds reduced the media's ability to adsorb and/or precipitate phosphates (Arias et al., 2001; Bubba et al., 2003). This has been confirmed by Gill et al. (2011), who found very little P adsorption affinity in glass. The latter reported glass filter P removal efficiencies that exceeded the ones observed in this study by 2.5 times. This difference might be explained by the additional limestone layer used by Gill et. al (2011), which accounted for 55% of the total P removal within glass.

4.3 Engineering significance of this study

This research expanded the knowledge on tertiary filtration of municipal wastewater using sand and crushed recycled glass as filter media. Results show that media selection should be based on contaminant removal targets, to ensure that environmental guidelines are met by the filter system.

TSS was the only parameter that was removed equally well by sand and crushed glass, with both filters providing effluent TSS concentrations close to zero. This indicates that crushed glass does not have to be fine-graded to successfully reduce TSS. COD removal seems to be determined by media size and surface properties, especially when most of it is dissolved. Differences in COD removal between sand and glass filters could be minimized by using smaller-graded glass. This would expand

surface area available for biomass growth, resulting in increased removal efficiencies. Media biomass densities seem to also play an important role in NH₄+–N removal. Finer media gradations would improve nitrification and denitrification within glass filters. This could approximate glass filter removal rates to those observed in sand, which delivered effluents with undetectable NH₄+–N.

If the main concern is P removal to levels below 1 mg P/L, neither sand nor crushed glass filters seem to be an appropriate option. P retention tends to be greater in sand beds, due to higher Ca availability within the media, but still insufficient to reach effluent discharge guidelines. Under alkaline wastewater conditions, sand concretion should be expected, especially close to the surface. This might pose limitations to filter operation and maintenance. Depending on the rate of concretion, it may be necessary to periodically replace the media with virgin sand, which would increase costs.

Nutrient removal could be improved by using vegetated filters. Plant growth on filter surface would consume inorganic phosphates, nitrate and ammonium, besides providing additional surface area for biofilm development. Potential complications could arise during biomass harvest – a necessary step to effectively remove contaminants from the system. Plant roots tend to get wrapped around wastewater delivery pipes, making it difficult to extract them without lifting the tubing. This could lead to pipe ruptures and damaged pipe connectors.

This study indicates that crushed recycled glass filters have potential as tertiary wastewater treatment systems, especially with regards to TSS, COD and NH₄+–N removal. Employing crushed recycled glass as filter media represents an opportunity to promote environmental sustainability by diverting waste from landfills. In most cases,

recycled glass can be obtained at no charge. Costs would mostly arise from crushing the material to specific gradations and transporting it to the site. Hence, it is advantageous to rely on a local glass supplier. Unlike sand, glass media tends to present little or no concretion due carbonate and calcium phosphate precipitation. This would lead to lower maintenance costs in these systems, as well as better reliability in operation and performance.

CHAPTER 5 – LIMITATIONS AND RECOMMENDATIONS FOR FUTURE WORK

A number of limitations and improvement opportunities were identified for this study, as well as future research priorities in the field of sand and crushed recycled glass as wastewater filter media:

- ❖ Power outages posed limitations to this study. The timer that controlled the main pump shut down every time these events occurred. Wastewater delivery to filter beds was therefore interrupted until the next sampling date, when the timer was manually reset. Effluent samples on these days could have been standing for a while, which might have compromised their quality. A controller that resets automatically after power failures would have avoided the problem.
- ❖ Backflow between holding tanks and EW caused major complications in 2018. Interconnected wells draining into a common container did not represent the best system configuration. Ideally, drainage would be kept separate for each holding tank, with treated effluent being discharged directly into the secondary overflow lagoon rather than into an intermediate emptying tank such as EW.

- Nitrogen species monitored in this study were NH₄+−N, NO₂−N and NO₃−N, measured in both influent and effluent samples. It would have been beneficial to quantify Total Nitrogen (TN) and Total Kjeldahl Nitrogen (TKN) as well, so that a more comprehensive mass balance could have been carried out. This would have contributed to a better understanding of N removal mechanisms within filter beds.
- The sampling methodology adopted by Gill et al. (2011) would have been a valuable addition to this study and should be considered by future researchers in the field. The author collected effluent at different media gradients, enabling the assessment of contaminant removal across filter depth. This approach would have been particularly useful to evaluate P retention in sand beds, given the high removal observed within top layers (i.e. concreted sand). It could also have provided information on N dynamics within the filter.
- SEM-EDS analysis (Scanning Electron Microscope with Energy Dispersive Spectroscopy) could have complemented the investigation of concreted sands, especially with regards to calcium phosphate precipitation. SEM would enable the visualization of concreted sands at high magnifications, which could potentially help identify precipitates within the sample. These regions could be further analyzed with EDS, for the semi-quantitative determination of their chemical composition.

- It would have been helpful to perform isotherm studies on virgin sand samples, so that P-sorption capacities could have been determined. This would have provided more information on P removal mechanisms (i.e. adsorption and precipitation) within sand filters. A better fit obtained with the Freundlich isotherm, for instance, would strengthen the hypothesis of calcium phosphate precipitation.
- The effect of hydraulic residence time (HRT) on filter efficiency was not evaluated in this study. Filters operated at a single HRT of 24 hours during both seasons. It would be interesting to assess filter performance at HRTs of 6 and 12 hours, for instance. The purpose would be to investigate whether reduced HRTs result in decreased contaminant removal within filter beds.
- Information on the commercial viability of crushed recycled glass in wastewater filtration represents a big gap in the literature. Financial specifications on glass filters treating municipal wastewater were only provided by Elliott (2001a, 2001b). Cost-benefit analyses could facilitate system selection and implementation by sanitation companies and municipal governments. Economic analyses should therefore be included in future research priorities, as already highlighted by Horan & Lowe (2007).

CHAPTER 6 – CONCLUSIONS

This study evaluated the performance of sand and crushed recycled glass in pilotscale subsurface filters treating municipal lagoon effluent. Filters operated at a 24-hour HRT, with active treatment seasons from May to September. System performance was assessed over a two-year period (2017 - 2018), which lead to the following conclusions:

- ❖ TSS were removed equally well in sand and glass filters. Removal efficiencies exceeded 90% in all beds, leading to effluents within the provincial limit of 25 mg TSS/L in both seasons. Effluent TSS levels did not fluctuate with variations in the influent, indicating consistent removal in both media.
- ❖ Both sand and glass filter effluents contained mostly dissolved COD, except for the first month of operation, where influent particulate COD exceeded 100 mg/L. During that time, both filters would have failed to achieve the guideline of 25 mg BOD/L. Dissolved COD removal was 29% less in glass filters, a significant difference when compared to sand. This resulted from the smaller surface area of crushed glass, which had an ES 21 times greater than that of sand.
- ❖ NH₄+–N reductions were above 90% in both filters. Results suggest that removal was predominantly achieved through nitrification and denitrification within the media. Plant uptake also contributed, accounting for less than 10%

of the NH₄+–N removed within filter beds in the second year. Sand filters were able to provide effluents without detectable NH₄+–N throughout both seasons. Glass filters met the guideline in 2017, exceeding the limit 33% of the time in the following year. This might be a reflection of increased influent NH₄+–N levels, which were on average 4.4 times higher that season.

- ❖ Treated effluent P existed mostly in the form of orthophosphates. Both sand and glass filters failed to reduce P levels below 1 mg P/L. In 2018, TP and orthophosphate reductions were 57% and 65% less in glass filters, respectively. Plant biomass accounted for approximately 1% of the total P removed by filter beds over the season.
- Concretion resulted from the precipitation of carbonates (dolomite and Mg-rich calcite) and calcium phosphates within sand beds. Concreted media, which occupied less than 4% of the bed volume, retained 30 35% of the P removed within sand filters. Retention in concreted layers accounted for approximately 50% of the difference in P removal between sand and glass filters.
- P removal within sand filters seemed to have been predominantly accomplished through mineral precipitation with Ca and Mg compounds. Adsorption to sand grains and carbonate precipitates might also have contributed. Decreased Ca availability in glass media represents the major reason for low P removal in these filters.

❖ Crushed recycled glass can be used as filter media in tertiary wastewater treatment systems, especially to achieve TSS, COD and NH₄⁺–N removal. Satisfactory TSS reductions can be obtained with coarser glass, whereas smaller-graded media could improve COD and NH₄⁺–N removal. Crushed recycled glass filters might present lower maintenance costs in comparison to sand, as concretion phenomena are not that common. Most importantly, crushed glass media filters pose an opportunity to contribute to sustainability by employing a locally recycled waste product.

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APPENDICES

Appendix 1 – Fisher-Pitman permutation p-values at the 95% confidence level, comparing sand duplicates (1W, 4W), glass duplicates (2W, 3W) and sand vs. glass filters

Fisher-Pitman p-values					
		F	Permutation Group	os	
		Sand (1W, 4W)	Glass (2W, 3W)	Sand and Glass	
	2017	0.8332	0.4311	1.5E-05	
рН	2018	0.2375	0.6470	0.0003	
	2017 vs. 2018	0.2890	0.4135	3.0E-08	
	2017	0.6932	0.9244	0.8480	
EC	2018	0.5967	0.7944	0.8403	
	2017 vs. 2018	0.5638	0.8292	0.9540	
	2017	0.6359	0.7790	0.6300	
Alkalinity	2018	0.1996	0.7863	0.7341	
	2017 vs. 2018	0.2611	0.7466	0.9449	
	2017	0.8573	0.9377	0.2379	
Temperature	2018	0.8892	0.9703	0.8359	
	2017 vs. 2018	0.9358	0.9496	0.5365	
	2017	0.7014	0.9470	0.3852	
Ca	2018	0.3664	0.8849	0.8899	
	2017 vs. 2018	0.5618	0.8974	0.7323	
	2017	0.6924	0.9635	0.9729	
Mg	2018	0.4076	0.9501	0.7187	
	2017 vs. 2018	0.3676	0.9375	0.7967	

Appendix 1 – Fisher-Pitman permutation p-values at the 95% confidence level, comparing sand duplicates (1W, 4W), glass duplicates (2W, 3W) and sand vs. glass filters

Fisher-Pitman <i>p</i> -values					
		F	Permutation Group	os	
		Sand (1W, 4W)	Glass (2W, 3W)	Sand and Glass	
	2017	0.5088	0.9319	0.9815	
Na	2018	0.2782	0.9161	0.9723	
	2017 vs. 2018	0.3026	0.9589	0.9740	
	2017	0.8026	0.9184	0.6693	
K	2018	0.1333	0.5016	0.5672	
	2017 vs. 2018	0.5988	0.7270	0.6260	
	2017	0.1920	0.3410	0.7527	
TSS	2018	0.7857	0.9283	0.0776	
	2017 <i>vs.</i> 2018	0.8948	0.8232	0.0968	
	2017	0.0834	0.5566	0.6297	
VSS	2018	0.5503	0.6491	0.0770	
	2017 vs. 2018	0.8752	0.8233	0.0914	
	2017	0.5047	0.4961	0.0009	
COD _{total}	2018	0.8024	0.9290	0.4041	
	2017 vs. 2018	0.7093	0.8190	0.1151	
	2017	0.5104	0.6653	0.0119	
$COD_{dissolved}$	2018	0.6473	0.9277	0.0043	
	2017 <i>vs.</i> 2018	0.9560	0.7957	0.0004	
	2017	0.8126	0.8896	0.0061	
NH_4^+ $-N$	2018	0.3173	0.3837	0.0121	
	2017 <i>v</i> s. 2018	0.4845	0.4261	0.0035	

Appendix 1 – Fisher-Pitman permutation p-values at the 95% confidence level, comparing sand duplicates (1W, 4W), glass duplicates (2W, 3W) and sand vs. glass filters

Fisher-Pitman <i>p</i> -values							
		Permutation Groups					
		Sand (1W, 4W)	Glass (2W, 3W)	Sand and Glass			
	2017	0.9535	0.7696	0.2788			
NO ₃ ⁻ –N	2018	0.2542	0.8285	0.1722			
	2017 vs. 2018	0.3348	0.8246	0.1935			
	2017	0.2412	0.2931	0.1516			
TP	2018	0.2249	0.7605	0.0040			
	2017 vs. 2018	0.1932	0.6210	0.0169			
	2017	0.1061	0.4472	0.1540			
Orthophosphate	2018	0.1793	0.5344	0.0030			
	2017 vs. 2018	0.1158	0.5235	0.0161			

All p-values were evaluated at the 95% confidence level.

p < 0.05 - failure to accept the H_0 of mean equality.

p > 0.05 - failure to reject the H_0 of mean equality.

Appendix 2 – Seasonal mass balances of NH_4^+ –N and NO_3^- –N (g) in influent and treated effluents

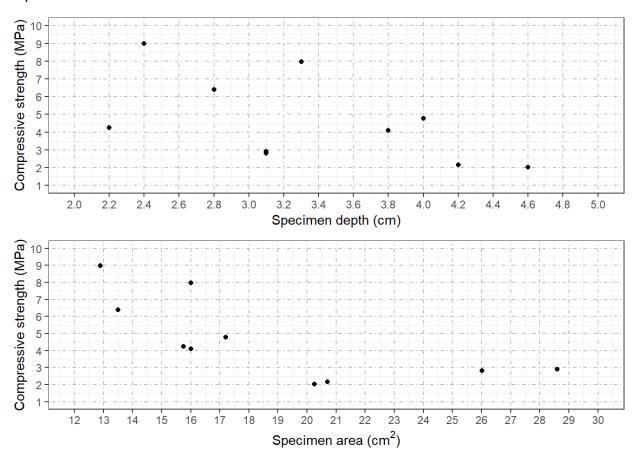
		Influent		Efflue		
		(g)	$1W_{\text{sand}} \\$	$2W_{\text{glass}}$	$3W_{\text{glass}}$	$4W_{\text{sand}} \\$
	NH ₄ +–N	933	7	83	88	5
2017	NO ₃ N	159	244	122	142	237
	NH ₄ +–N removed		926	850	845	928
	NH ₄ +–N	4214	0	497	312	13
2018	NO ₃ ⁻ –N	64	2084	1521	1665	3078
	NH ₄ +–N removed		4214	3717	3902	4201

Season 2018 mass balances may not be completely accurate, as technical field issues were frequent along the season (see section 2.2.2).

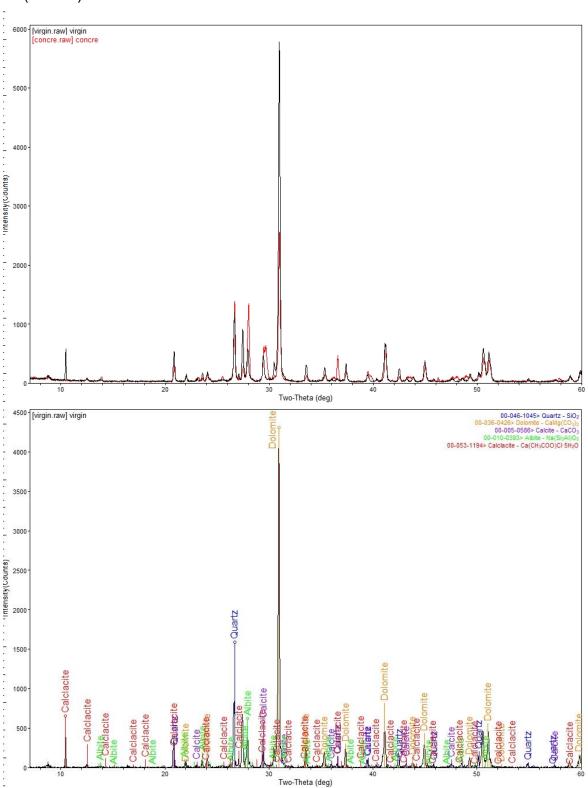
Appendix 3 – Two-sample *t*-test *p*-values at the 95% confidence level, comparing TP and orthophosphate concentrations in sand and glass filter effluents

Two sample <i>t</i> -test <i>p</i> -values						
	Season	Sand	Glass			
TP vs. Orthophosphate	2017	0.6384	0.6593			
	2018	0.6357	0.6675			
All <i>p</i> -values were evaluated at the 95% confidence level.						
$p < 0.05$ - failure to accept the H_0 of mean equality.						
$p > 0.05$ - failure to reject the H_0 of	of mean equal	ity.				

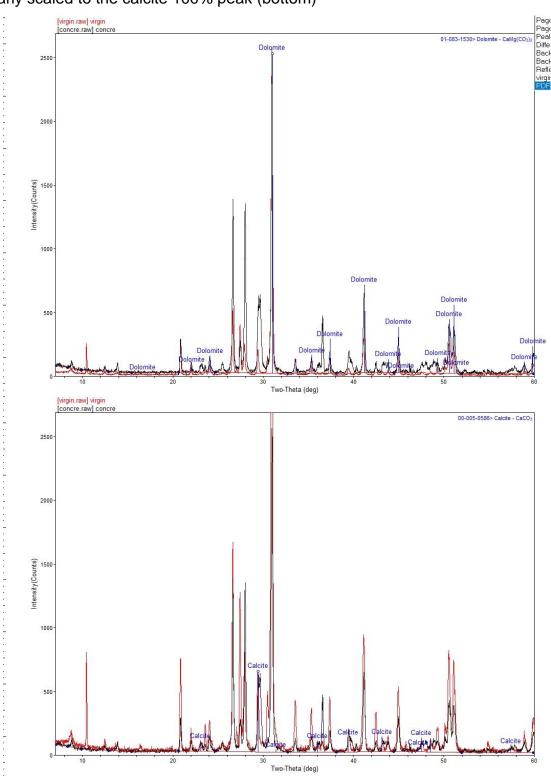
Appendix 4 – Compressive strength of concreted sand blocks, according to specimen depth and area



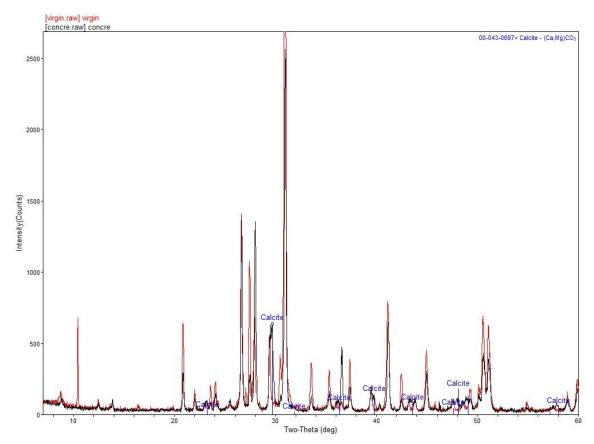
Appendix 5 – Crystalline structures of virgin and concreted sands obtained from XRD analysis: overlay of virgin (black) and concreted (red) sands (top); composition of virgin sand (bottom)



Appendix 6 – Overlay of crystalline structures of virgin (red) and concreted sands (black) obtained from XRD analysis: similarly scaled to the dolomite 100% peak (top); similarly scaled to the calcite 100% peak (bottom)



Appendix 7 – Overlay of crystalline structures of virgin (red) and concreted sands (black) obtained from XRD analysis similarly scaled to the calcite 100% peak: fitting of the magnesium-rich calcite to the concreted sand



Appendix 8 – Welch two-sample *t*-test *p*-values at the 95% confidence level, comparing Ca, Mg and P concentrations in virgin and concreted sands washed with DI water and citrate buffer at pH 4, 5 and 6

Welch two-sample t-test p-values						
Wash	Ca	Mg	Р			
DI water	0.0180	0.0100	0.0324			
pH 4	0.0331	0.0379	0.0001			
pH 5	0.0089	0.0264	0.0004			
pH 6	0.0003	0.0090	0.0012			
	Wash DI water pH 4 pH 5	Wash Ca DI water 0.0180 pH 4 0.0331 pH 5 0.0089	Wash Ca Mg DI water 0.0180 0.0100 pH 4 0.0331 0.0379 pH 5 0.0089 0.0264			

All *p*-values were evaluated at the 95% confidence level.

p < 0.05 - failure to accept the H_0 of mean equality.

p > 0.05 - failure to reject the H_0 of mean equality.