Development of Transitional Settling Regimen Parameters to Characterize and Optimize Solids-Liquid Separation Performance

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Dissertation submitted to the faculty of the Virginia Polytechnic Institute and State University in partial fulfillment of the requirements for the degree of

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ABSTRACT

Novel settling characteristic metrics were developed based on the fundamental mechanisms of coagulation, flocculation, and settling. The settling metrics determined parameters that are essential in monitoring and optimizing the activated sludge process without the need for expensive or specialized equipment. Current settling characteristic measurements that don't require specialized instruments such as sludge volume index (SVI) or initial settling velocity (ISV), have no fundamental basis in solid-liquid separation and only indicate whether settling is good or bad without providing information as to limitations present in a sludge matrix. Furthermore, the emergence of aerobic granulation as a potential pathway to mitigate solid-separation issues further stresses the need for new settling characteristic metrics to enable integration of the new technology with the current infrastructure.

The granule or intrinsic aggregate fraction in different types was of sludge was quantified by simulating different surface overflow rates (SOR). The technique named Intrinsic Settling Classes (ISC) was able to separate granules and floc by simulating high SOR values due to the lack of a flocculation time needed for granules. The method had to be performed in a discrete settling environment to characterize a range of flocculation behavior and was able to classify the granular portion of five different types of sludge. ISC was proven to accurately (±2%) determine the granule fraction and discrete particle distribution. The major significance of the test is its ability to show if a system is producing particles that will eventually grow to become granules. This methodology proved to be very valuable in obtaining information as to the granular fraction of sludge and the granular production of a system.

Flocculent settling (stokesian) was found to be predominant within ideally operating clarifiers, and the shift to 'slower' hindered settling (non-stokesian) causes both failure and poor effluent quality. Therefore, a new metric for settling characteristics was developed and classified as Limit of Stokesian Settling (LOSS). The technique consisted of determining the total suspended solids

(TSS) concentration at which mixed liquor settling characteristics transition from stokesian to non–stokesian settling. An image analytical technique was developed with the aid of MATLAB to identify this transition. The MATLAB tool analyzed RGB images from video, and identified the presence of an interface by a dramatic shift in the Red indices. LOSS data for Secondary activated-sludge systems were analyzed for a period of 60 days at the Blue Plains Advanced Wastewater Treatment Plant. LOSS numbers collected experimentally were validated with the Takacs et al. (1991) settling model. When compared to flux curves with small changes in the sludge concentration matrix, LOSS was found to be faster at characterizing the hindered settling velocity and was less erratic.

Simple batch experiments based on the critical settling velocity (CSV) selection were used as the basis for the development of two novel parameters: threshold of flocculation/flocculation limitation (TOF/α), and floc strength. TOF quantified the minimum solids concentration needed to form large flocs and was directly linked to collision efficiency. In hybrid systems, an exponential fitting on a CSV matrix was proposed to quantify the collision efficiency of flocs (α). Shear studies were conducted to quantify floc strength. The methods were applied to a wide spectrum of sludge types to show the broad applicability and sensitivity of the novel methods.

Three different activated sludge systems from the Blue Plains AWWTP were monitored for a 1 year period to explore the relationship between effluent suspended solids (ESS) and activated sludge settling and flocculation behavior. Novel metrics based on the transitional solids concentration (TOF, and LOSS) were also collected weekly. A pilot clarifier and settling column were run and filmed to determine floc morphological properties. SVI was found to lose sensitivity ($r < 0.20$) when characterizing ISV above a hindered settling rate of 3 m h^{-1}. ISV and LOSS had a strong correlation ($r = 0.71$), but ISV was subject to change, depending on the solids concentration. Two sludge matrix limitations influencing ESS were characterized by transition concentrations; pinpoint floc formation, and loose floc formation. Pinpoint flocs had TOF values above 400 mg TSS L-1; loose floc formation sludge had TOF and LOSS values below 400 mg TSS L^{-1} and 900 mg TSS L^{-1}, respectively. TOF was found to correlate with the particle size distribution while LOSS correlated to the settling velocity distribution. The use of both TOF and LOSS is a quick and effective way to characterize limitations affecting ESS.

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GENERAL AUDIENCE ABSTRACT

New parameters to determine how particles separate from water were developed. The new parameters didn't require expensive or specialized instruments. Current parameters that don't rely on specialized instruments give little information on how particles separate from water. The new parameters provide information on particle size, particle settling speed, and particle stickiness. The significance of this research is the ability for anyone in the field to gain a better understanding of the settling process they are monitoring using these parameters.

The first parameter named Intrinsic Settling Classes allows one to determine the particle size distribution within a sludge mixture based on how fast the particles settle. The parameter requires dilution of the sample to inhibit the particles from sticking together. Bigger particles will settle faster, due to more mass assuming density does not change significantly.

The second parameter named Limit of Stokesian Settling determines a particle's settling speed. The parameter involved finding the maximum particles that could occupy a particular space before the particle-particle interaction start to hinder settling. Particles with a faster settling distribution can have more particles occupying a certain space before hindering one another.

The third parameter named Threshold of Flocculation identified a particle's stickiness. Stickier particles attach together in a shorter time resulting in large clumps of particles that settle faster. This parameter involved determining the minimum amount of particles needed to form large clumps of particles at a predetermined time. Stickier particles require less particles and time to form large clumps of particles.

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ATTRIBUTION

Each co-author is duly credited for his or her contribution to this work, both in their sharing of ideas and technical expertise.

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INTRODUCTION

Figure 1.1: Aerial photograph of the Blue Plains Advanced Wastewater treatment plant where the experimental work presented in this thesis was conducted.

1.1 BACKGROUND ON THE PROBLEM

The increasing demand for clean water in the world triggered by the exponential growth of the human population and by industrial and economic development raises serious concerns for sustainable water management practices (Jury and Vaux, 2005). Figure 1.2 shows the projected population of the Earth by the year 2050 to be 9.3 billion (Glieck and Palaniappan, 2010) and the Earth's accessible freshwater will remain more or less constant (Postel et al., 1996). Humans already appropriate over 50% of all available renewable freshwater (Srinivasan et al., 2012), raising concern that the scarcity of fresh water in the future will limit agricultural, industrial, economic, and human well-being in the future. An important step to circumvent water scarcity is to efficiently clean and reuse wastewater. As the population increases, major cities will grow into "mega cities" (Abhat, et al, 2005) which will increase the production and contamination of wastewater. Furthermore, the Alcamo et al., 2008 study predicts approximately 5 billion people will face a water crisis. Therefore there is a need to intensify and optimize current wastewater treatment.

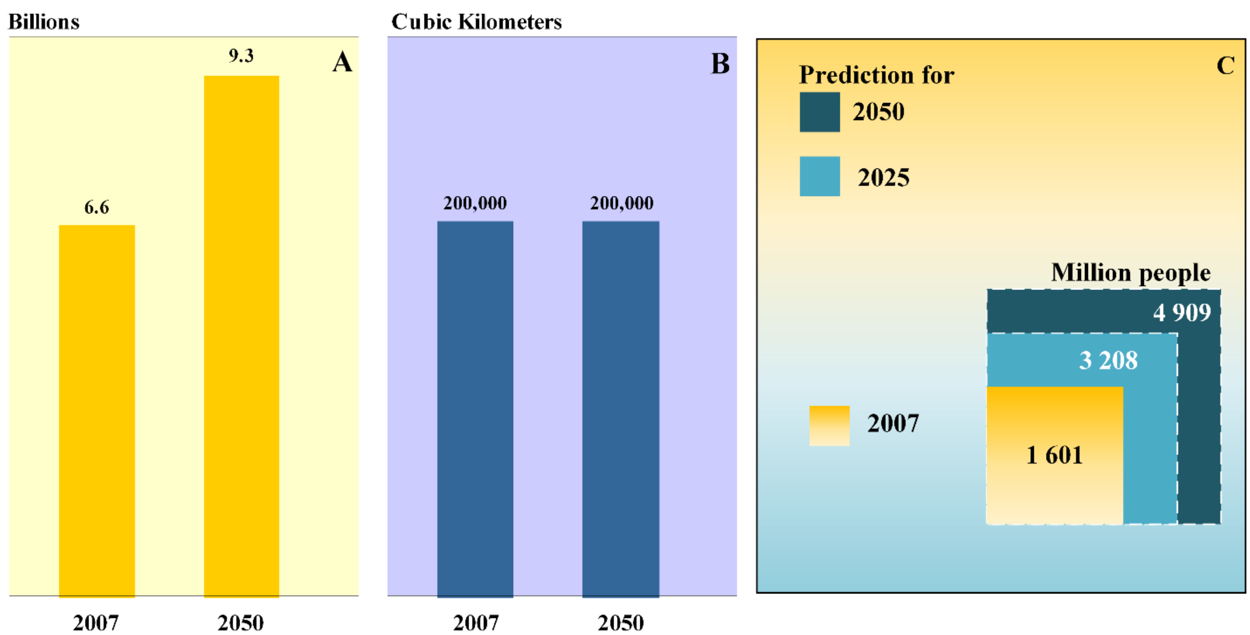

Figure 1.2: (A) Population increase and (B) water resources projections. (C) World population living in river basins with severe water stress. Water availability below 1000 m^3 per year was regarded as an indicator of water stress. Projections for 2025 and 2050 are computed considering socio-economic and climate driving forces from the B2 scenario of the IPCC (Alcamo et al 2008).

1.2 ACTIVATED SLUDGE TREATMENT PROCESS

Among all wastewater treatment processes, the most widespread and popular treatment of both municipal and industrial wastewater is the activated sludge process (Tandoi et al., 2006). The activated sludge process consists of three parts (Figure 1.3), reactor, transport channel, and clarifier. In the reactor, pollutant degradation by micro-organisms occurs and the micro-organisms grow as a suspended floc. After the water has been sufficiently treated, the combination of flocs and water, called mixed liquor, flows through the channels to the clarifier. At the clarifier the flocs settle out by gravity leaving a clear effluent water. The process effectiveness is related to both the pollutant degradation and floc-water (also referred to as solids-liquid) separation. If the solids-liquid separation is poor, suspended flocs will wash out of the system with the effluent. A study by Pujol and Canler (1992) estimated at least 70% of the activated treatment plants encounter solid-liquid separation problems at least once a year. Design, operation and control procedures have been under continuous development to optimize and mitigate sludge settling issues. However, most of the solids-liquid separation problems are related to the biological floc's formation and characteristics.

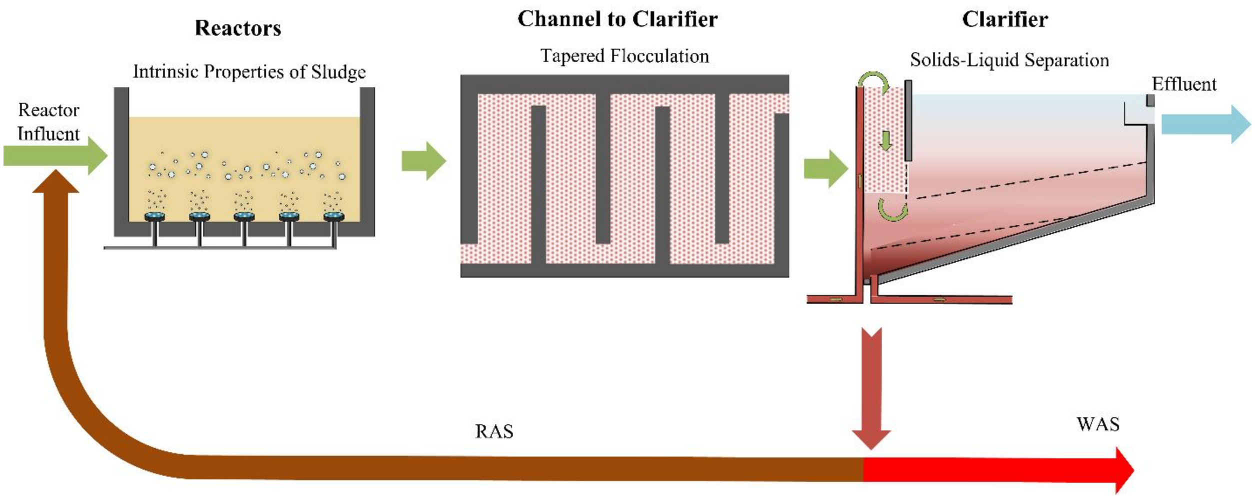

Figure 1.3: Basic concept of the activated sludge process for secondary/advanced reactor-clarifier

The emergence of aerobic granules for wastewater treatment has created a pathway to negate poor settling and remove clarifiers as the bottleneck in treatment (Beun et al., 1999, 2002). However, a tremendous amount of infrastructure has been geared towards flocculent systems that Anderson (2010) identified to be worth 3.1 trillion US dollars (2008). Upgrade of an existing facility to a fully granular treatment plant is not economically feasible for the next 20 years (2030), which is compounded by the projected population increase of 50 million (16.5%) in the U.S. (United Nations, 2014). There is an emerging need to intensify current treatment processes that includes integration of both flocculent and granular systems using the current infrastructure. To achieve this integration there is a need for physical separation processes that can aggressively select and waste granules as desired.

1.3 SETTLING CHARACTERISTIC METRICS AND THEIR LINK TO SOLIDS-LIQUID SEPARATION: HOW CAN WE IMPROVE THESE?

In the activated sludge treatment process, an optimally performing clarifier is required to meet the desired effluent solids concentration. Quantification of the maximal solids and flocculation capacity, a crucial operational parameter, can assist in optimization of a clarifier. However, determination of secondary clarifiers' solids and flocculation capacity has largely been ignored. This is in no part due to its significance, as clarification serves as the treatment limiting step in activated sludge systems (Ekama et al., 2002). Utilities design large clarifiers to increase the safety factor while disregarding the optimum and economical operational requirements. The presence of large clarifiers stemmed the need to understand fundamental mechanisms of solids-liquid separation in flocculant systems. Operators without tools of a reliable metric to gauge sludge settling performance rely on a reverse feedback loop to mitigate problems as they arise in activated sludge clarifier operations. Given the breath of limitation and understanding of activated sludge settling properties, we are often left with the following: *The search for a reliable and simple metric for activated sludge quality based on a fundamental understanding of flocculant settling properties.*

Solids-liquid separation can be predicted and simulated with or without the Stokes law, hereafter named as stokesian or non-stokesian settling, respectively. The stokesian settling includes two phases of settling, i.e. discrete and flocculant, while the non-stokesian settling includes hindered settling and compression (Tchobanoglous and Burton, 1991). Most adequate functioning clarifiers only display flocculant settling and compression phases, while hindered settling is only observed under clarifier failure condition (Mancell-Egala et al., 2016). Current conventional characterization methods such as sludge volume index (SVI) are good qualitative indicators of bad versus good settling but do not provide quantification of the settling characteristics. This is due to SVI being largely centered on compression settling that is a crude indicator of settling properties and cannot be linked with effluent quality. Alternatively, state point analyses generated from initial settling velocities (ISV) are based on hindered settling, which has been shown to be fraught with artifacts, operator error issues, and unreliability (Kinnear. 2002). The methods for characterizing the settling phases are very subjective and lack a clear boundary distinction as highlighted by the current settling characteristics measurements. These measurements have been unsuccessfully incorporated in computation fluid dynamic models for determining the capacity of secondary clarifiers (Ekama et al., 1997; Krebs, 1995; Krebs et al., 1996; Mazzolani et al., 1998; Lakehal et al., 1999; McCorquodale, 2004; DeClercq, 2003). The shortcomings of current methodologies lie in their focus on non-stokesian settling models to characterize the settling phenomena occurring in a clarifier.

Because conventional parameters only focus on non-stokesian settling and only provide rough indication of either good or bad settling, they do not allow for identification of factors that limit the settling of that system. Sludge settling characteristics can be limited by four factors, namely: (a) floc-size, (b) floc-compactness, (c) floc-adsorption, and (d) compression (Figure 1.4). Limitations of each factor and the efficient treatment can be linked to flocculation mechanics.

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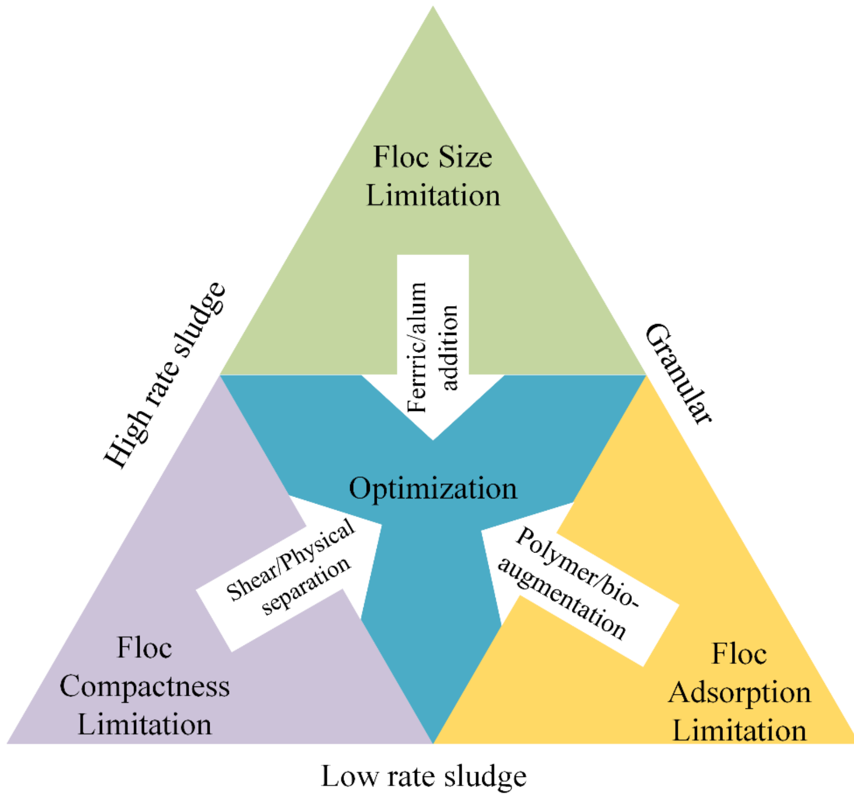

Figure 1.4: Diagram depicting the three limitations affecting solid-liquid separation and the engineered responses to the specific limitations. Examples of sludge type that fall into the categories are indicated.

The agglomeration of biomass through collisions is a simple definition for flocculation (Bartby et al., 2006). However, there are several models that simulate the different stages of flocculation. For the scope of this work flocculation can be narrowed down to three particle stages; dispersed solids or colloidal particles, micro-floc formation, and macro-floc formation (Figure 1.5). Each stage has different key factors. Colloidal particles have to be destabilized to mitigate the repulsive forces thus allowing them to stick to each other forming micro-flocs. The destabilization of colloidal particles is known as coagulation and a limitation here can lead to poor floc size distribution. After the micro-flocs are formed they stick to other micro-flocs to from macro-flocs. This mechanic is dominated by how effective the micro-floc can form attachments. This stage can be promoted by flocculants that are long heavy chain molecules that help form bridges between flocs or sweep up suspended particulates. Limitations here can cause problems with both floc size and compactness.

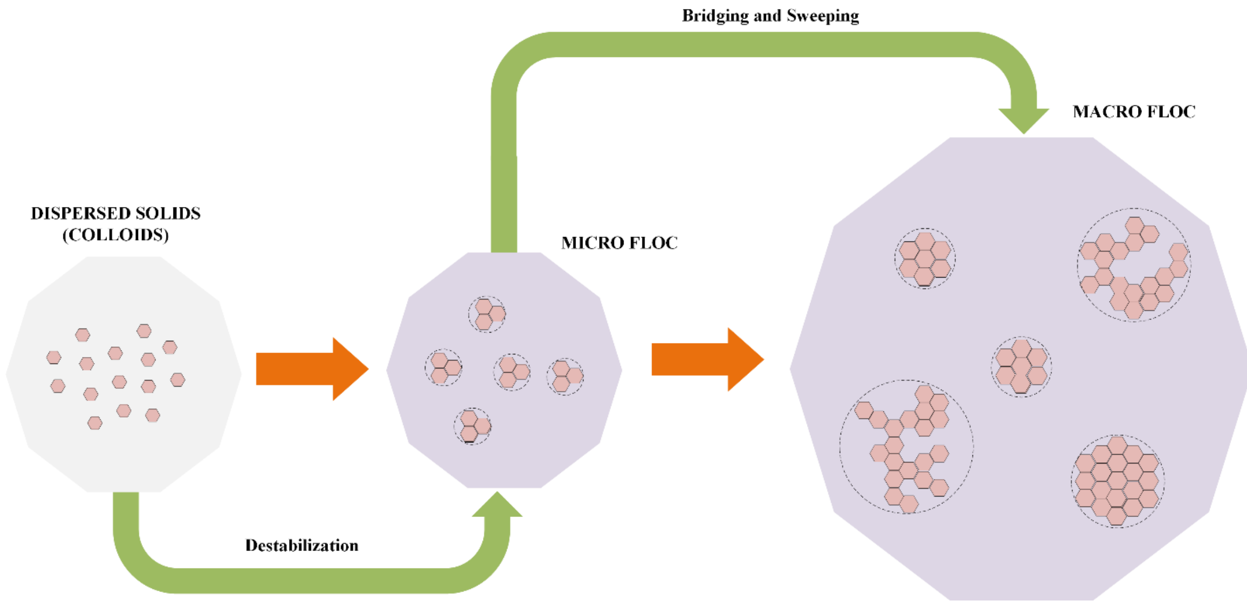

Figure 1.5: Mechanism of coagulation and flocculation to develop a floc.

A settling characteristic metric that gives information as to the limiting step in flocculation is essential to mitigate issues in solids-liquid separation. For example problems in micro-floc formation if identified by a settling metric can be solved by more extensive coagulation.

At the first stage of flocculation, the DVLO theory (Derjaugin and Landau, 1941; Verwey and Overbeek, 1948) explains colloidal interactions and the mechanisms that cause destabilization. The theory is based in a double layer model that considers all particles electrically charged surfaces existing in an ionic environment. Figure 1.6A illustrates this by showing a highly negative colloid surrounded by positive counter ions. At the surface of the colloid, negative attraction causes positive ions to form a firmly attached layer around the surface of the colloid, known as the Stern Layer. Beyond the Stern layer positive ions are still attracted by the negative colloid but are also repelled by the other positive ions and the Stern layer. A dynamic equilibrium forms a diffuse layer of counter ions. As the distance to the negative colloid increases, the concentration of positive ions decreases until it reaches equilibrium with the normal positive counter ion concentration in the environment. In a similar manner, there are no negative co ions near the surface of the negative colloid, because of repulsion. The concentration of negative co ions increases the greater the distance from the colloid. The diffuse layer is the charged atmosphere surrounding the colloid. At any distance from the colloid the charge density is positive and negative charge. The Stern layer and the diffuse layer is known as the double layer.

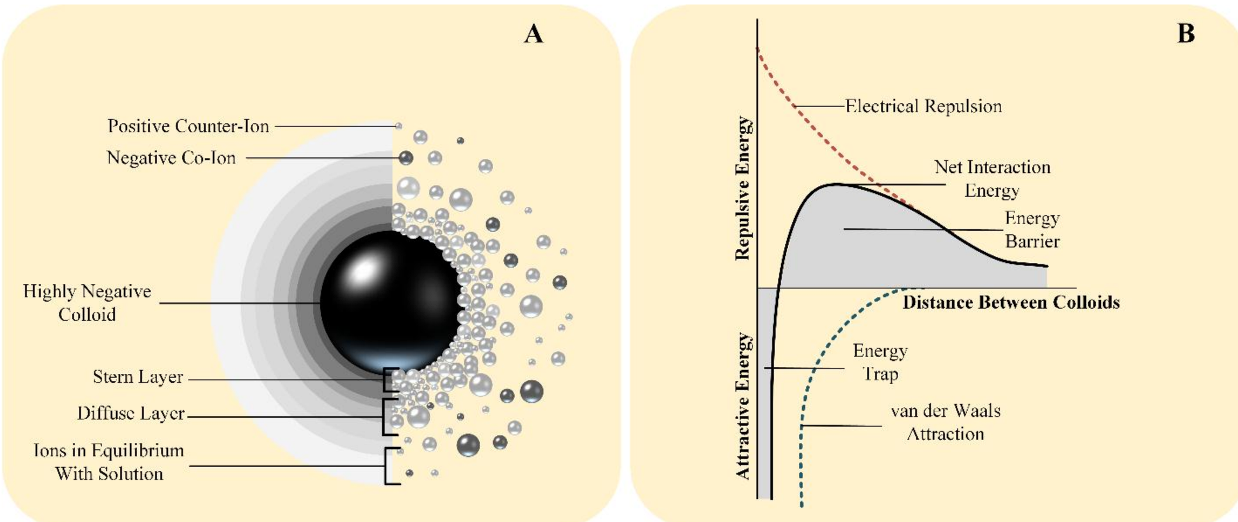

Figure 1.6: (A) two ways to visualize the double layer, the left shows the charge density and the right shows the distribution of positive and negative ions around the charged colloid. (B) The net interaction energy curve which is formed by subtracting the attraction energy from the repulsive energy.

The DVLO Theory explains why some colloids agglomerate while others don't by looking at the balance between two opposing forces; electrostatic repulsion and van der Waals attraction. Electrostatic repulsion becomes significant when two particles approach each other and their electrical double layers begin to overlap. Energy is required to overcome this repulsion and force the particles together. As the distance between two particles decreases the energy required to move them closer increases, this represented by the electrical repulsion curve (Figure 1.6B). Van der Waals attraction between two colloids is the result of forces between individual molecules in each colloid. Each molecule in the first colloid has van der Waals attraction to each molecule in the second colloid. This effect is additive and the sum of the forces is represented by the van der Waals attraction curve (Figure 1.6B). The DVLO theory combines both the van der Waals attraction and electrostatic repulsion curve to explain the tendency of colloids to remain discrete or agglomerate. The combined curve is the net interaction energy (Figure 1.6B) it can shift from either attraction or repulsion depending on the distance between colloids. There is a region called the repulsion energy barrier and the maximum height is the minimum kinetic energy (due to speed and mass) to pass the barrier and agglomerate. Destabilizing a colloid is the act of reducing the energy barrier thereby increasing the chances of agglomeration.

Kinetic energy to enable improved flocculation was modeled by both Smoluchowscki (1917) and Han et Lawer (1992). Figure 1.7 illustrates the sources of kinetic, random thermal 'Brownian' motion of particles (peri-kinetic), imposed velocity gradients from mixing (ortho-kinetic), or differences in settling velocities of individual particles (differential settling). Flocculation studies have mathematically related the flocculation mechanics in terms of two parameters that can be measured, collision efficiency and collision number (Serra et al 1997). Collision number (which is defined as the probability two flocs will collide) has been modelled by both Smoluchowscki (1917) and Han et Lawer (1992), and the knowledge of hydrodynamic interaction is strong and is function of shear gradient and solids concentration. However, collision efficiency (chances of successful attachment of two particles through collision) is a complex function, which makes determination of it difficult. Some studies have used an adjustable parameter (Biggs et al., 2002), or a constant value (Zhang et al., 2003). More mechanistic tools involve mathematical models developed heuristically. They integrate the physical characteristics of the process and allow for a better understanding of a system. Trajectory analysis (Adler et al., 1981) has been used successfully to develop collision efficiency models.

According to Serra et al., (1997) increasing the collision number by mixing will increase collisions, but decreases the efficiency of collisions. This is because there is another important flocculation parameter known as floc strength. Floc strength is ability of a floc to withstand shear without breaking apart. Using shear to characterize floc strength is a method that has been used in several studies (Leentvaar and Rebhun, 1983; Francois, 1987; Fitzpatrick et al., 2003). The protocols for floc strength have been studied thoroughly, but they are not employed by operators at municipalities. Floc strength can improved by better bridging through the addition of branched polymers that form threads and fibers that bind and capture particulates, or large doses of coagulants resulting in hydrous oxide flocs that enmesh particulates (Figure 1.8).

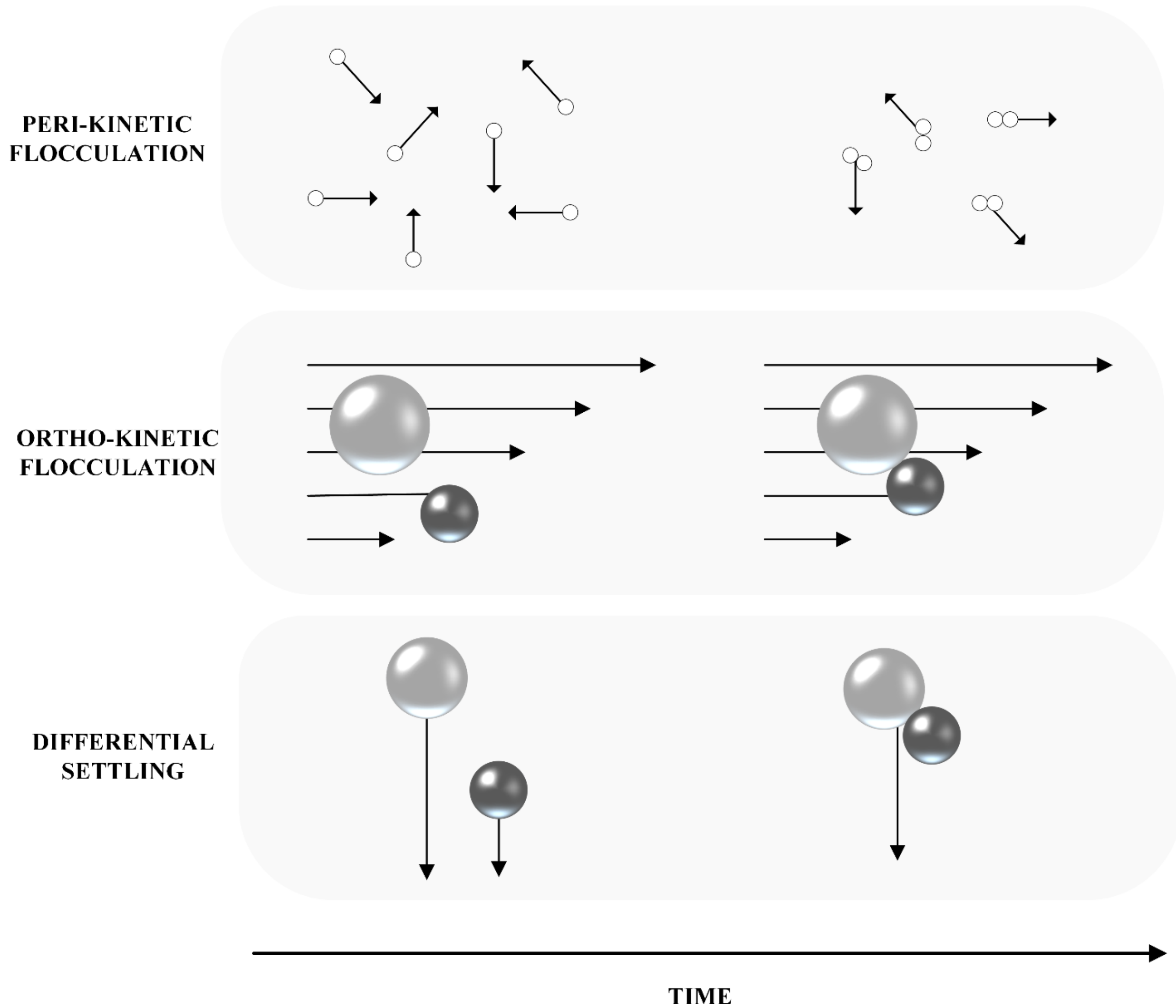

Figure 1.7: Illustrations of the different flocculation mechanisms that induce collisions

Current methods to determine floc strength use turbidity measurements (Cheng et al., 2011). Turbidity measurements have shown to correlate well with floc sizes (r^2 = 0.95), but the turbidimeter must be calibrated for different wastewater matrices or coagulants/flocculants used (Cheng et al., 2011).Collision efficiency models require particle size information. This can be collected via the use of small angle light scattering instruments (Francois, 1987; Spicer et al., 1998), video cameras with image analysis (Wu et al., 2003), or photometric dispersion analyzer (Fitzpatrick et al., 2003). Farrow and Warren (1993) conclude that light/laser scattering and transmission techniques are good for showing qualitative (rather than absolute) changes in particle sizes in activated sludge systems. All methods referenced to acquire collision efficiency or floc strength require specialized instrumentation that can be expensive and are most likely unavailable to municipalities. There is an immediate need for protocols to establish collision efficiency and floc strength that can easily be used at municipalities.

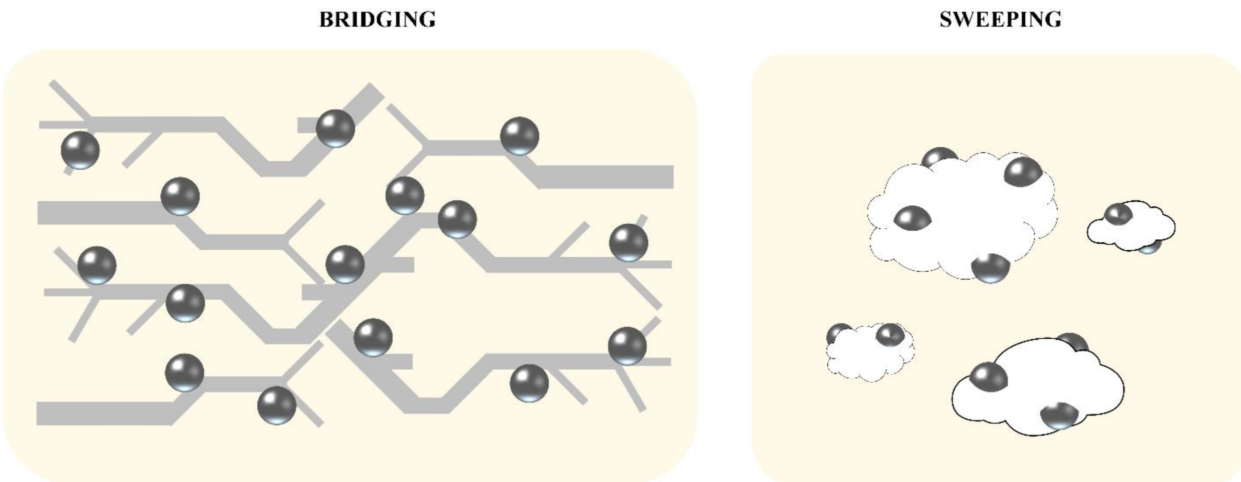

Figure 1.8: Illustrations of the bridging and sweeping mechanisms of flocculation

In summary, this study proposes the use of new settling character matrices utilizing simple tools, which are directly linked to specific settling limitation and therefore allow for development of approaches to improve settling and control clarifier operation.

1.4 RESEARCH QUESTIONS

Question 1: If integration of aerobic granules into conventional activated sludge processes is a potential pathway of intensifying the clarification process it possible to quantify the granular and flocculant fractions using differences in physical separation? Typical activated sludge systems rely on bio-flocculation to create large flocs that settle out faster than the individual particles. However a granular system as described by de Kreuk et al. (2007) consists of aggregates of microbial origin that do not need bio-flocculation to allow for fast settling properties. Granules are more compact than flocs, making them denser and more resistant to shear, with a faster stokesian and non-stokesian settling speed. A potential pathway to intensify the clarification process would be to combine the two sludge types, and use their different aggregation and settling mechanics to quantify the different fractions. An Ideal system would have the right balance of the flocculent portion that has minimum diffusion resistance based on Fick's law for diffusion, and a granular portion that has ideal settling properties based on stokes law. A hybrid system of flocs and granules has two key parameters that will benefit from a new approach. One is the ballasting function that expresses the degree of granules and good settling particulates that will be meshed in with floc to provide very fast settling. Secondly the clarification function, which will provide the operators knowledge as to the percentage of flocculent material that is present in sludge. Both these functions are essential, and monitoring and proper management of these functions provide balance in a hybrid system which will optimize the clarification process.

Question 2: Does a clarifier under normal operation display all four types of settling, and if not, in what conditions a clarifier display all four types of settling? Flux theory based techniques are often used for the design and operation of secondary clarifiers. However, there is strong evidence from pilot clarifier runs at the Central Davis County Sewer District (Kinnear 2002), that flocculent settling is the main settling mechanics that occurs in a clarifier outside of the sludge blanket, and the development of a hindered settling regimen is the precursor for clarifier failure. Failure occurs during hindered settling because non-stokesian settling velocities are a magnitude lower than stokesian flocculent velocities, and velocities recorded suggest a discontinuity in settling velocities as the mechanics of settling switches from stokesian to non-stokesian (Kinnear et al. 2001, McCorquodale et al. 2004). Furthermore, secondary clarifiers frequently fail by filling up with a dilute blanket as shown in numerous stress tests conducted by Daigger (1998).

Question 3: Conventional settling characteristic metrics used by flux theory does not take into account the differences in settling mechanics under different settling regimens, is there a different approach that does? According to current flux theory based on pioneering work by Kynch (1952), Kynch's constitutive assumption states that settling velocity of particles is a factor of solids concentration. The common assumption made is that sludge settling velocity decreases with increasing solids concentration. Takacs et al. (1991) built on flux theory and presented a widely accepted model predicting settling velocity over the entire solids concentration range. Takác's model identified that the highest settling velocity happens during flocculent settling and the transition to hindered settling results in a settling velocity that decreases with solids concentration. Since the mechanics of the different settling regimens does not change, it might be possible to characterize settling behavior by the transition concentration from one settling regimen to another.

Question 4: Is there a way to determine other flocculation properties (i.e. collision efficiency, floc strength) using simple batch jar tests? Current methods to determine floc strength use turbidity measurements (Cheng et al., 2011). Turbidity measurements have shown to correlate well with floc sizes ($r^2 = 0.95$), but the turbidimeter must be calibrated for different wastewater matrices or coagulants/flocculants (Cheng et al., 2011). Furthermore, collision efficiency models require particle size information. This can be collected via the use of small angle light scattering instruments (Francois, 1987; Spicer et al., 1998), video cameras with image analysis (Wu et al., 2003), or photometric dispersion analyzer (Fitzpatrick et al., 2003). All other methods referenced to acquire collision efficiency or floc strength require specialized instrumentation that can be expensive and are most likely unavailable to municipalities. Developing a methodology requiring inexpensive components which are readily available would allow operators to understand their systems better and make optimizations easier.

Question 5: Limitations affecting effluent quality of clarifiers has been documented by numerous studies. However, is there a way for a simple batch jar test to determine these

limitations? Studies have looked at developing settling characteristic metrics based on the transition between settling regimens (Chapter 3). There are four settling regimens; discrete, flocculant, hindered, and compression (Ekama et al., 1997). Metrics based on transitional concentrations, unlike conventional metrics, factor in the variance in settling at different solids concentration. The use of transitional settling metrics instead of conventional metrics is more practical for monitoring a system with short sludge retention time (SRT) also referred to as a high-rate systems. Initial studies into the transition between discrete to flocculant settling was suggested as a means to characterized collision efficiency and can be defined as the threshold of flocculation (TOF) (Chapter 3). Collision efficiency is a measure of the success of two particles sticking together after a collision. This mechanism is directly related to the energy barrier concept of the Derjaguin, Landau, Verwey, and Overbeek (DVLO) theory (Elimelech, 1991). If the energy barrier of a colloid is high, fewer colloids have enough kinetic energy for a successful collision. This limitation affects floc size and can be characterized by a TOF value. The transition from flocculant to hindered settling, defined as limit of stokesian settling (LOSS), characterizes the settling velocity of a floc (Mancell-Egala et al., 2016). According to stokes law, size and shape of a floc determines settling velocity. More compact flocs are more spherical according to fractal analysis (Bushell et al., 2002), and more spherical objects have less drag and settle faster according to stokes law. Therefore, using both TOF and LOSS in tandem can help characterize the compactness of a floc.

1.5 EXPERIMENTAL OUTLINE

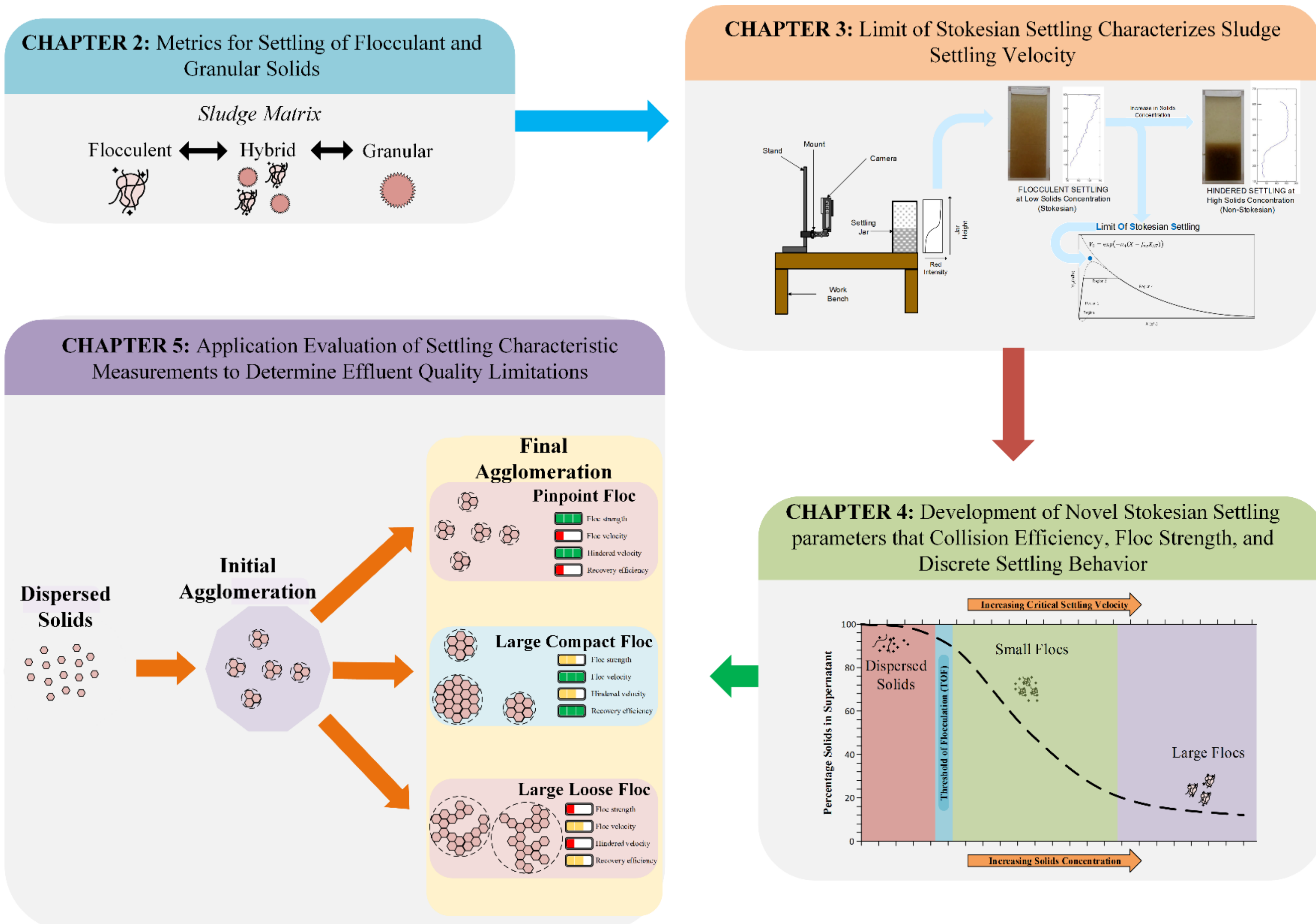

Figure 1.9: Experimental outline of the dissertation

1.6 ANNOTATED DISSERTATION OUTLINE

Chapter 1: ***Introduction*** The chapter provides background, defines the focus by stating the five major research questions, provides graphical abstracts for the experimental work and briefly summarizes the material presented in this dissertation by including an annotated dissertation outline.

Chapter 2: ***Metrics for settling of flocculant and granular solids***. This manuscript provides a new analytical technique to determine the intrinsic aggregate fraction in different types of sludge by simulating different surface overflow rates (SOR). Typical activated sludge (Flocculant) systems rely on bio-flocculation to create large flocs that settle out faster than the individual particles. However a granular system as described by de Kreuk et al. (2007) consists of aggregates of microbial origin that do not need bio-flocculation to allow for fast settling properties. The method uses high SORs that prevent bio-flocculation and separate out the granular fraction. *Research question 1* is directly addressed in this chapter. This work was presented at the 2014 Water Environment Federation Technology Conference in New Orleans, Louisiana, and was accepted for publication:

Mancell-Egala, WA.S.K., DeClippeleir, H., Murthy, S.N., Novak, J.T. (2014) Metrics for settling of flocculant and granular solids. *Proceedings of the Water Environment Federation, WEFTEC 2014*, Chicago, Illinois.

Chapter 3: ***Limit of stokesian settling characterizes sludge settling velocity***. This chapter provides a new analytical technique which is independent of solids concentration and characterizes sludge settling velocity. Flocculent settling (stokesian) is predominant within ideally operating clarifiers, and the shift to 'slower' hindered settling (non-stokesian) causes both failure and poor effluent quality. Therefore, a new metric for settling characteristics was developed and classified as Limit of Stokesian Settling (LOSS). The technique consisted of determining the total suspended solids (TSS) concentration at which mixed liquor settling characteristics transition from stokesian to non–stokesian settling. An image analytical technique was developed with the aid of

MATLAB® to identify this transition. *Research question 2 and 3* are addressed in this chapter. This work has been accepted for publication:

Mancell-Egala, W.A.S.K., Kinnear, D.J., Jones, K.L., De Clippeleir, H., Takacs, I., and Murthy, S.N. (2016) Limit of stokesian settling characterizes sludge settling velocity. *Water Research*, **90**. 100-110.

Chapter 4: ***Development of novel stokesian settling parameters that quantify collision efficiency, floc strength, and discrete settling behavior.*** This manuscript provides a three new novel parameters to predict the effluent quality and settling behavior in clarifiers that cannot conventionally be achieved using either the conventional flux theory or overflow rates. Simple batch experiments based on the critical settling velocity selection were used as the basis for the development of three novel parameters: intrinsic settling classes (ISC), threshold of flocculation/flocculation limitation (TOF/a), and floc strength. Research question 4 is directly addressed in this chapter. This work has been submitted to Bioresource and Technology.

Chapter 5: ***Yearlong application evaluation of novel settling characteristic measurements to determine effluent suspended solids limitations.*** This manuscript provides settling characteristic information on three activated sludge systems from the Blue Plains AWWTP for a 1 year period. The aim was to explore the relationship between effluent suspended solids (ESS) and activated sludge settling and flocculation behavior. Hindered settling rates (ISV) and sludge volume index (SVI) measurements were collected weekly. Novel metrics based on the solids concentration at which the transition between settling regimens occurred were also collected weekly. The transitional metrics were Threshold of Flocculation (TOF), and Limit of Stokesian Settling (LOSS). They marked the transition from discreet to flocculant settling, and from flocculant to hindered settling, respectively. A pilot clarifier and settling column were run and filmed to determine floc morphological properties and link these properties to ESS limitations and settling characteristic metrics. Research question 5 is directly addressed in this chapter. This work has been submitted to Water Research.

Chapter 6: ***Concluding remarks and engineering significance.*** The concluding chapter discusses specific significant contributions of this research to the wastewater engineering field, and proposes several valuable extensions to the work described throughout.

Appendix A: ***Settling transition concentration measurement to quantify sludge settling behavior.*** This paper was presented at the 2011 Water Environment Federation Technology conference in New Orleans, Louisiana. The manuscript presents a new analytical technique classified as settling transition concentration (STC) and was used to determine the total suspended solids (TSS) concentration at which mixed liquor settling characteristics transition from flocculent settling (Type II) to zone or hindered settling (Type II). An automated program was created with the aid of MATLAB program to determine STC. The program functioned by incorporating the settling sludge into a red green blue (RGB) matrix, the mixture is represented by varying color pixels. The MATLAB program analyzes these pixels via colors red, green, and blue with index values that range from 0 to 255. The program plotted the RGB values against the corresponding depth of the jar, and the plot was used to highlight the interface. STC data was analyzed for a period of six months in the Blue Plains Advanced Wastewater Treatment Plant to establish links to sludge settling characteristics. This paper was accepted and published by Water Environment Federation Technology:

Mancell-Egala, W.A.S.K., Kinnear, D.J., Jones, K.L., Takacs, I., and Murthy, S.N. (2012) Settling transition concentration measurement to quantify sludge settling behavior. *Proceedings of the Water Environment Federation*, WEFTEC 2012. New Orleans, Louisiana.

Appendix B: ***Quantifying flocculation capacity of activated sludge.*** This paper was presented at the 2015 Water Environment Federation Technology conference in Chicago, Illinois. The manuscript presented focuses on quantifying the flocculation capacity of a sludge matrix. The Critical Settling Velocity (CSV) was used to classify the fractions of sludge that flocculated and settled. A test matrix was implemented whereby CSV and solids concentration were varied. Two sludge types were tested; secondary activated sludge from high rate activated reactors and nitrification/denitrification (BNR) sludge, both from Blue Plains advanced wastewater treatment plant. Two new settling characteristic measurements were obtained from the test matrix; Threshold of Flocculation (TOF), and ‘α-values’. TOF is the transition concentration from discrete settling to flocculent settling, and was found to be directly related to flocculation capacity. Lower TOF corresponded to a high flocculation capacity, as flocs could be formed with fewer collisions. A solids removal rate was obtained from the test matrix, and followed an exponential trend. The curvature of the solids removal rate or ‘α-value’ also corresponded with flocculation. High curvature or α -value corresponded to high flocculation capacity and likewise for low α-values. This paper was accepted to be published by Water Environment Federation Technology:

Mancell-Egala, De Clippeleir, H., Su. C., Novak, J.T., and Murthy, S.N. (2015) Quantifying flocculation capacity of activated sludge. *Proceedings of the Water Environment Federation*, WEFTEC 2012, New Orleans, Louisiana.

Appendix C: *Supplemental information for Chapter 3.* Raw data for settling characteristic tests and simulations are presented, including settling velocity curves and associated model fits. Additionally, images and schematics of the instruments are included in this appendage.

Appendix D: *Supplemental information for Chapter 4.* Schematic of the materials used to determine the new parameters are presented in this appendage.

Appendix E: *Supplemental information for Chapter 5.* Raw data for settling characteristics and operational parameters are presented. Additionally, images and schematics of the experimental setup are included in this appendage.

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CHAPTER TWO

METRICS FOR SETTLING OF FLOCCULANT AND GRANULAR SOLIDS

2.1 AUTHORS

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Published

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2.2 ABSTRACT

A new analytical technique was used to determine the intrinsic aggregate fraction in different types of sludge by simulating different surface overflow rates (SOR). The technique involved placing a well-mixed low MLSS (<300 mg L^{-1}) in a 4000 mL graduated cylinder. Samples which consisted of the top 400 mL of the mixed liquor in the graduated cylinder were collected at specific times. These specific times simulate SOR values in an actual full-scale clarifier. The samples were tested for TSS, and the values correlate to the amount of TSS in the clarifier's effluent. The method proved capable in discrete settling environment and was able to classify the granular portion of five different types of sludge. ANNAMOX side stream pilot located at Blue Plains was 20% granular compared to Secondary mixed liquor from Blue Plains AWWTP which was 1% granular. The major significance of the test is its ability to show if a system is producing particles that will eventually grow to become granules. The mainstream ANNAMOX pilot in the Blue Plains AWWTP had very low granule fraction (2%) however it had 45% of the total TSS as particles that settled above 3 m/h, this is considerably significant especially when compared to the 2% in secondary mixed liquor. This methodology proved to be very valuable in obtaining information as to granular fraction of sludge and the granular production of a system.

2.3 KEYWORDS

Aerobic granules, intrinsic properties, activated sludge.

2.4 NOMENCLATURE

AWWTP	Advanced wastewater treatment plant
ISV	Initial settling velocity
MLSS	Mixed liquor suspended solids
SOR	Surface overflow rate
SVI	Sludge volume index
TSS	Total suspended solids

2.5 INTRODUCTION

Determining the capacity of secondary clarifiers based on sludge characteristics has been unsuccessful with conventional methodologies (Ekama *et al.*, 1997). The shortcomings of current methodologies lies in their focus on non-stokian settling models to characterize the settling phenomena occuring in a clarifier. Most operational clarifiers only display stokian settling through to the sludge blanket where compression takes over, and the presence of hindered settling is rather seen as the precursor for clarifier failure (Mancell-Egala *et al.*, 2011). Current conventional methods such as sludge volume index (SVI) are good indicators of bad vs good settling but the latter do not allow for providing information of the degree in the quality of settling. This is due to SVI being largely centered on compressional settling (when settling characteristics are good) and are crude representations of settling properties. Alternatively state point analyis generated from ISV are based on hindered settling, which has been shown (Kinnear. 2010) to be fraught with artifacts, operator error issues and unreliable in determining settling properties.

Stokian settling, which characterizes most of settling in a clarifier is separated into discrete and flocculent settling. Discrete settling occurs at very low MLSS concentrations (<400mg/L) by "non-sticky particles" or granules that settle with no interactions with other particles. Flocculent settling occurs at slightly higher MLSS (400mg/L-1000mg/L) and involves "sticky particles" or the formation of flocs from particles that stick together and settle out with minimum interactions with other particles. Typical activated sludge systems rely on bio-flocculation to create large flocs that settle out faster than the individual particles. However a granular system as described by de Kreuk *et al.* (2007) consists of aggregates of microbial origin that do not need bio-flocculation to allow for fast settling properties. Granular sludge is generally defined as sludge that has an $SVI_5 \approx SVI_{30}$ and a SVI_{30} lower than 70 ml/g (van Loosdrecht 2013). This definition is based on the fact that granules do not show any compression leading to SVI_5 measurements that are similar to SVI_{30}. However, the latter is not valid any more when a system consists of a combination of granules and flocs, as this will lead to compression. Alternatively, a minimum settling velocity of 10 m/h (Winkler *et al.*, 2013) was determined as a characteristic for aerobic granules and the latter was for example used by Liu *et al*. (2005) to design reactors that select for granulation.

The SVI and ISV measurements are not sensitive enough to provide degrees in settling properties and thus lack the ability to provide information on granular fraction and granular properties of activated sludge systems. A hybrid system of flocs and granules has two key parameters that will benefit from a new approach, one is the ballasting function that expresses the degree of granules and good settling particulates that will be meshed in with floc to provide very fast settling. Secondly the clarification function, which will provide operators knowledge as to the percentage of flocculent material that is present in sludge. Both these functions are essential, and monitoring and proper management of these functions provide balance in a hybrid system which will optimize settling characteristics. An Ideal system would have the right balance of flocculent portion that has minimum diffusion resistance based on Fick's law for diffusion, and a granular portion that has ideal settling properties based on stokes law. The goal of this research to develop a method based on stokian settling and design criteria for granular systems to distinguish between flocculent and granular fractions in activated sludge.

2.6 MATERIALS AND METHOD

The basis of this methodology is that SOR selects for granulation therefore at a certain SOR there will be a distinct difference in the granular and the flocculent fraction. Simulation of SOR in an actual clarifier was done by optimizing a setup by Peric et al., 2008 that simulated SOR in a primary clarifier. Also Peric et al., 2008 calculation of SOR in a clarifier as proportionally simulated by a graduated cylinder was used, and by changing the time of sample collection will allow the selection of SOR. At Specific SOR the sample (400 mL) was collected through the sampling ports. This volume represented the clarifier's effluent TSS. The mixed liquor tested came from for sources that are listed in Table 2.1.

2.7 RESULTS AND DISCUSSION

One of the characteristics of granular sludge is that it does not need bio-flocculation to allow for good settling (de Kreuk et al. 2004). Therefore the effect of bio-flocculation on the effluent quality was tested for a range of TSS concentrations (200 to 2000 mg TSS/L) at a SOR of 1.5 m h^{-1} (900 gpd ft^2). This SOR, according to literature (Daigger et al, 1998) is typically the point when a clarifier starts failing in stress tests and can be therefore seen as a threshold for good settling sludge. As expected a large difference in percentage effluent TSS between granular and flocculent system at the same SOR was observed in the discrete region (Figure 2.1). In the granular system 60% of the TSS could be maintained in the clarifier under discrete settling condition while for the flocculent systems only about 10% remained. This confirms the hypothesis that granular systems do not require bio-flocculation to settle out. In flocculent systems, a significant improvement in settling only occurs due to bio-flocculation (Figure 2.1). The latter was also shown by obtaining 2 SOR curves for flocculent system (Flocculent 2 sludge) at two different MLSS concentrations, namely at high MLSS of 923 mg TSS L^{-1}, representing the bio-flocculation region (Figure 2.2) and at low MLSS of 214 mg TSS/L, representing discrete settling. Figure 2.2 shows that for flocculent sludge bio-flocculation could improve the TSS in the effluent at an SOR at 1.5 m h^{-1} (900 gpd ft^2) from 95% to 26%. This shows that to classify granular from flocculent fractions operation at discrete settling range (200 – 400 mg TSS L^{-1}) is necessary, as higher MLSS concentration already incorporate bio-flocculation which plays a major role in settling characteristics of flocs.

Based on the latter experiments, granular and good settling particulate fractions were defined as the TSS fraction remaining at a SOR of 10 and 1.5 m h^{-1}, respectively (Figure 2.3A). The granular threshold of 10 m3/m2/h was based on individual settling velocities of granules (Winkler et al., 2013) and design criteria for granular systems (Liu et al 2005). The poor and non-settable solids (NSS) fractions were calculated from the predicted NSS at 0 m h^{-1}. The latter approach showed a clear difference in settling between granular sludge (granular 1 & 2), sludge from a hybrid system (Hybrid) and flocculent sludge (flocculent 1 & 2), while conventional settling parameters could not (Table 2.2, Figure 2.3B). SVI conflicted with ISV and the SOR test in the indication that Hybrid sludge had poor settling. Both ISV and SVI had very small shifts in values when indicating good settling which provide no information that can be pertained as to the characteristics of the sludge. A good settling particulate fraction (SOR > 1.5 m h^{-1}) of 45% and 65% was determined for hybrid and granular sludge, respectively, while this fraction was minimal for the flocculent types (Figure 2.3B). Only in the granular sludge, a granular fraction of about 20% was observed. Additionally, the NSS fraction was 55 and 20% of total TSS in Flocculent 1 and the Granular 1 Sludge. The hybrid system had the lowest NSS which was 9% of the total TSS. This showed that the hybrid system propagates the makeup of denser particles which comprise a majority of the TSS however they do not settle as fast as the granules.

2.8 CONCLUSION

In summary sludge characterization based on discrete settling proved successful in its goal of accurately characterizing the granular portion of mixed liquor. Furthermore this methodology was sensitive enough to determine degrees in settling characteristics in areas conventional SVI and ISV measurement could not detect a difference. This methodology can be used to precisely indicate how good and bad settling will be in a clarifier and can show operators the degree of flocculation needed to remove a majority of the TSS in the effluent. The major significance of the test is its ability to show if a system is producing particles that will eventually grow to become granules. The mainstream deammonification pilot in the Blue Plains AWWTP (Hybrid sludge) had very low granule fraction (2%). However, due to the ballasting provided by the granules, it had 44% of the total TSS as particles that settled above 1.5 m/h. The latter is a significant improvement compared fully flocculent sludge (Figure 2.3). It is believed that characterizing the discrete settling properties of the mixed liquor could provide valuable information for clarifier designs and operations.

2.9 SIGNIFICANCE OF RESEARCH

Knowing merely whether a sludge has good or bad settling is not enough. This paper quantifies between the grades of good and great settling, and sheds light on areas such as granule and flocculent fractions that are not possible with SVI or other rudimentary conventional methods. The ability of having a more refined metric for settling that can distinguish sludge quality at a higher degree helps in computing the ballasting function and clarification function of a sludge that until now has not been possible.

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2.11 TABLES

Table 2.1: Location and description of all of the sludge types used in the experiment

Name	Location and description
Flocculent 1	Secondary mixed liquor (A-stage) from Blue Plains AWWTP, a predominately flocculent system
Flocculent 2	Nitrification mixed liquor from Blue Plains AWWTP, a predominately flocculent system
Hybrid	Mixed liquor from Mainstream deammonification pilot from Blue Plains AWWTP, a hybrid system of both granular and flocculent
Granular 1	Mixed liquor from deammonification Side Stream pilot SBR D1 from Blue Plains AWWTP , a predominately granular system
Granular 2	Mixed liquor from deammonification Side Stream pilot SBR D2 from Blue Plains AWWTP , a predominately granular system

Table 2.2: Conventional settling measurements of the types of sludge (Table 2.1) used

Sludge Type	SVI_{30} (mL g^{-1})	ISV (m h^{-1})
Flocculent 1	116	3.3
Flocculent 2	88	4.0
Hybrid	121	4.4
Granular 1	81	--
Granular 2	86	4.8

2.12 FIGURES

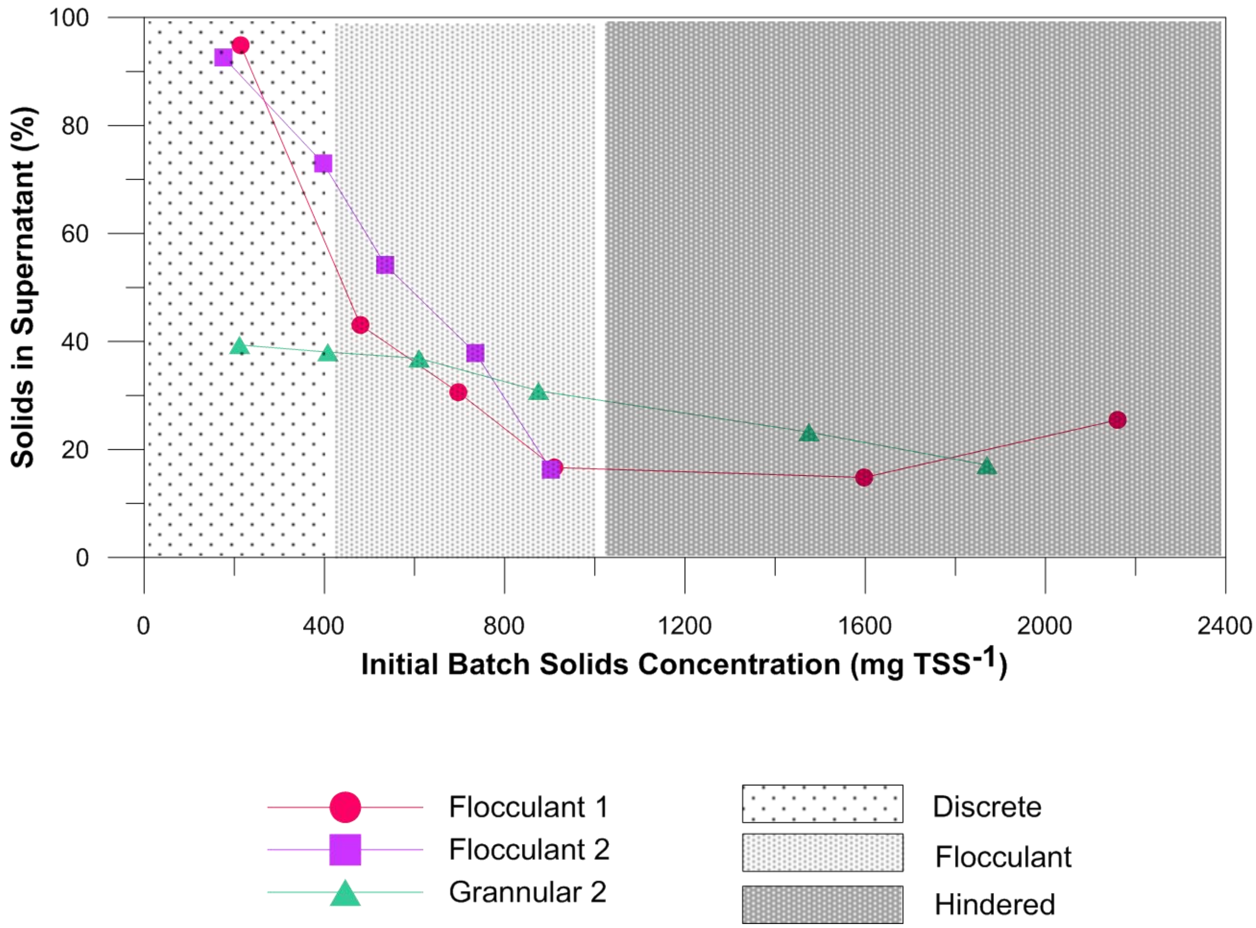

Figure 2.1: Graph showing percentage TSS in effluent versus ranging TSS of mixed liquor concentrations at an SOR of 1.5 m h^{-1}, and the TSS zones that exhibit discrete, flocculent and hindered settling. The test was done for 2 flocculent systems (Flocculent 1 & 2) and 1 granular system (Granular 2).

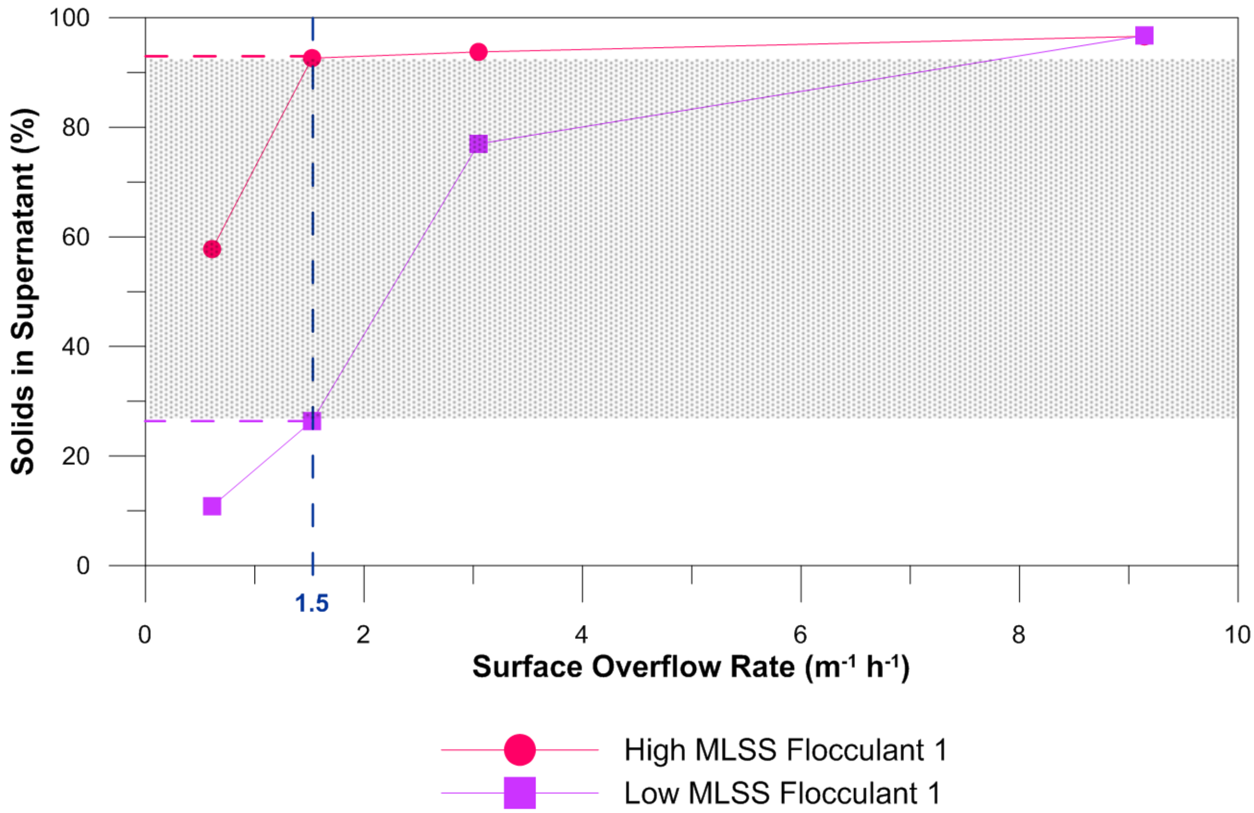

Figure 2.2: Graph shows how percent of TSS in the effluent at different SOR changes for Discrete settling environment (214 mg L^{-1}) and flocculent environment (923 mg L^{-1}). The shaded area signifies the greatest difference in flocculent and discrete settling at an SOR of 1.5 m h^{-1}.

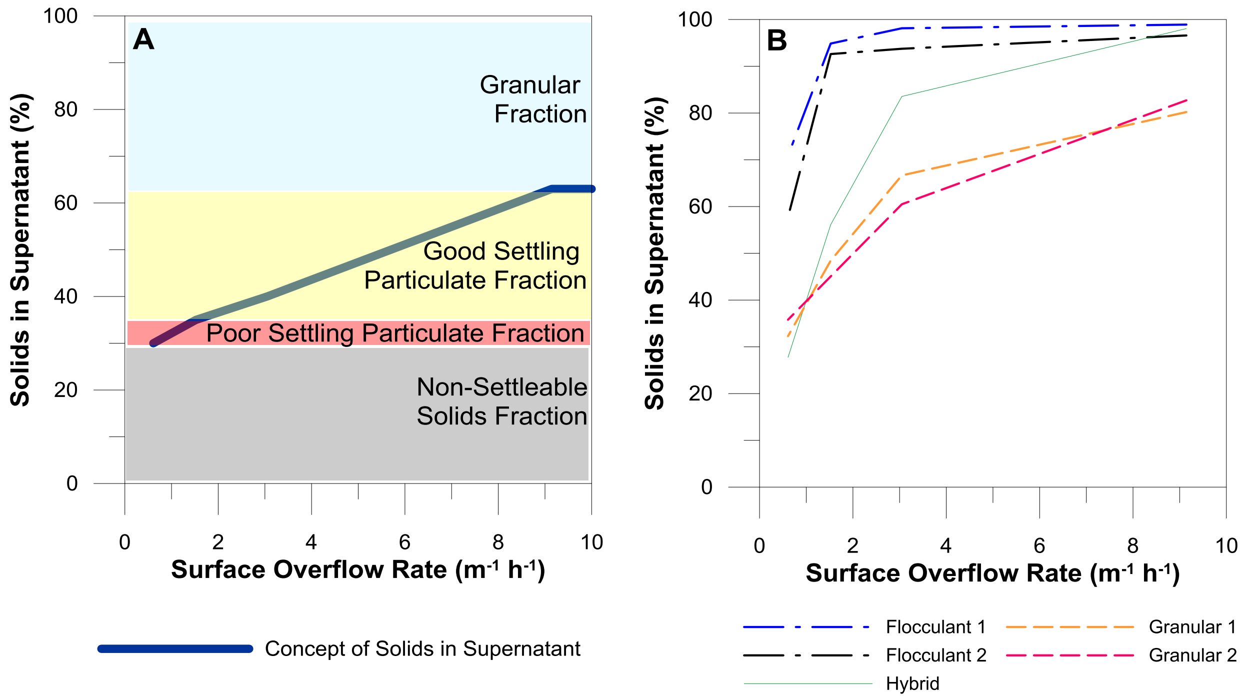

Figure 2.3: (A) Concept of the graph output of SOR test for differentiation between granular, good and poorly Settling Particulate Fraction, and predicted NSS. (B) Graph details results of SOR test done on five different types of sludge (Table 1).

CHAPTER THREE

LIMIT OF STOKESIAN SETTLING CONCENTRATION CHARACTERIZES SLUDGE SETTLING VELOCITY

3.1 AUTHORS

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3.2 ABSTRACT

Flocculent settling (stokesian) is predominant within ideally operating clarifiers, and the shift to 'slower' hindered settling (non-stokesian) causes both failure and poor effluent quality. Therefore, a new metric for settling characteristics was developed and classified as Limit of Stokesian Settling (LOSS). The technique consisted of determining the total suspended solids (TSS) concentration at which mixed liquor settling characteristics transition from stokesian to non–stokesian settling. An image analytical technique was developed with the aid of MATLAB® to identify this transition. The MATLAB tool analyzed RGB images from video, and identified the presence of an interface by a dramatic shift in the Red indices. LOSS data for Secondary activated-sludge systems were analyzed for a period of 60 days at the Blue Plains Advanced Wastewater Treatment Plant. LOSS for secondary systems typically occurred between 600-700 mg TSS/L but reached 1000 mg TSS/L for a good settling secondary sludge and 500 mg TSS/L for a poor settling secondary sludge, settling quality was based on hindered settling rates. In addition, LOSS was collected for granular systems seeded with cyclone underflow from Strass Wastewater Treatment Plant, it was observed that LOSS was higher for granular systems ranging from 1600 to 5500 mg TSS/L for low and high levels of granulation, respectively. The monovalent to divalent cation ratio (M/D) was increased with the addition of sodium ions to deteriorate settling properties. Samples adjusted with higher M/D consistently had 100 mg TSS/L (15%) decrease in LOSS from the control. LOSS numbers collected experimentally were validated with the Takacs et al. (1991) settling model. When compared to flux curves with small changes in sludge matrix, LOSS was proven to be faster at

characterizing hindered settling velocity and was less erratic. This is the first time a measurement method has been developed to characterize the transition from stokesian to non-stokesian settling. Additionally, this is the first step in developing new metrics to predict clarifier failure, and determine effluent quality through the development of flocculent settling metrics.

3.3 KEYWORDS

Final clarifier, conventional activated sludge, settling characteristics, settling velocity distribution.

3.4 NOMENCLATURE

AWWTP	Advanced wastewater treatment plant
BNR	Biological nutrient removal
f_{ns}	Fraction of non-settable solids in the feed
ISV	Initial settling velocity
LOSS	Limit of stokesian settling
M/D	Monovalent/divalent ratio
r_p	Parameter associated with settling behavior in low solids concentration
r_h	Parameter associated with settling behavior in hindered settling zone
SLR	Solids loading rate
SOR	Surface overflow rate
SRT	Sludge retention time
$sSVI_{30}$	Stirred sludge volume index at thirty minutes
TSS	Total suspended solids
v_0	Settling velocity constant
v_s	Settling velocity
WWTP	Wastewater treatment plant
X	Solids concentration
X_f	Solids concentration in feed
X_{min}	Non-settable solids concentration in feed

3.5 INTRODUCTION

In wastewater treatment secondary clarifiers fulfill three tasks. They serve as clarifiers, sludge thickeners, and sludge storage zones. As clarifiers they provide low total suspended solids (TSS) supernatant, as sludge thickeners they provide a constant underflow of thickened sludge, and as sludge storage zones they safeguard the storage of sludge during peak flow rates (Ekama and Gunthert 1997). Accurate prediction of the capacity limitation of a clarifier is essential to mitigate failure. When the capacity of a clarifier is surpassed the clarifier will begin to fail in two of its tasks. Firstly as a clarifier because, higher TSS supernatant will leave the system. Secondly as a sludge storage zone, as solids will be escaping in the supernatant. The basis of estimating clarifier capacity is rooted in sludge quality which includes but is not limited to variables such as degree of bio-flocculation, non settleable solids (NSS), settling velocity, monovalent to divalent ratio (M/D), pH, and coagulant addition (De Clercq 2003, Lakehal et al. 1999, Li and Ganczarczyk 1987).

Unfortunately it is difficult to estimate the overflow capacity of a clarifier with current methods, and secondary clarification is frequently a limiting factor for a wastewater treatment plant (Ekama and Marais 2002). There are currently two conventional sludge quality methods popularly used: Sludge Volume Index (SVI) and Flux curves. The former being the most widely used due to simplicity, the latter requires plotting a solids flux curve against solids concentration. The solids flux curve is generated from Initial Settling Velocities (ISV) from a range of solids concentrations. Compared to SVI that requires one solids concentration and takes thirty minutes to complete, Flux curve requires at least five concentrations and can take about half a day to complete. However, SVI has “no basis in solid-liquid separation theory” according to Standard Methods for the Examination of Water and Wastewater (American Public Health Association et al. 1980). Several authors (Daigger and Roper 1985, Ekama and Marais 1986, Koopman and Cadee 1983, Pitman 1985), have investigated relationships between SVI and the Flux Curve , but the main drawback is the inconsistencies of SVI and suspended solids concentration (Dick and Vesilind 1969). Efforts have been made to circumvent SVI’s dependence on solids concentration by dilution. Diluted Sludge Volume Index test were noted by Ekama and Marais (1984) to not vary with solids concentration, but were proven by Bye and Dold (1998) to bear no relationship to settling characteristics.

In contrast, Flux theory has had successes quantifying settling characteristics, and has produced models that are backed with experimental data (Cho et al. 1993, Dupont and Henze 1992). Flux theory's basis lies in the pioneering work by Kynch (1952), Kynch's constitutive assumption states that settling velocity v_s of particles is a factor of solids concentration. The common assumption made is that v_s decreases with increasing solids concentration (Åström et al. 2012), and a number of empirical models have been proposed relating ISV's to solids concentration. The most commonly used is the semi logarithmic Vesilind (1968) equation:

$$v_S = V_0 e^{-r_h X} \qquad (1)$$

where X is solids concentration, V_0 and r_h are sludge settling constants. Takacs et al. (1991) expanded flux theory and presented a widely accepted model predicting v_s over the entire solids concentration range (Figure 3.1). The model presented four regions; region 1 represented the NSS, region 2 represents the low solids concentration, whereby discrete settling is in effect, region 3 represents flocculent settling which is assumed to be independent of solids concentration and is a function of the maximum settling velocity, and region 4 is the non stokesian region that described hindered and compression settling. Takacs et al. (1991) model identifies that the highest settling velocity occurs during flocculent settling and the transition to non-stokesian settling results in a settling velocity that decreases with solids concentration. Flux theory quantifies sludge quality by the area underneath the solids flux curve. A majority of the area is located after the peak solids flux. A larger area signifies better sludge quality. This paper studies the implications of the solids concentration variance with the start of region 4. The Limit of Stokesian Settling (LOSS) is the solids concentration marking the beginning of region 4, a higher LOSS will correlate with a larger area under region 4.

Batch initial settling velocity tests conducted at solids concentration above LOSS ensures hindered settling conditions and the ability to determine the interface location. As the test concentration decreases, the interface becomes more difficult to determine as one approaches LOSS. However, with current technologies, determination of an interface at solids concentration close to LOSS is possible. Kim et al. (2011) with the aid of MATLAB wrote a program to accurately identify a sludge interface. Utilizing cameras and computer programing will allow for a degree of automation that will remove subjectivity from LOSS tests. This new approach is to devise a new simple bench scale metric that is based on the fundamentals of settling characteristics, and also provides information on flux curves.

Flux theory based techniques are often used for the design and operation of secondary clarifiers. However, there is strong evidence from pilot clarifier runs Central Davis County Sewer District (Kinnear 2002) that flocculent settling is the main settling mechanics that occurs in a clarifier (Supplemental data S1) outside of the sludge blanket, and the development of a hindered settling regimen is the precursor for clarifier failure. Failure occurs during hindered settling because non-stokesian settling velocities are a magnitude lower than stokesian flocculent velocities and velocities recorded suggest a discontinuity in settling velocities as the mechanics of settling switches from stokesian to non-stokesian (Kinnear et al. 2001, McCorquodale et al. 2004). Furthermore, secondary clarifiers frequently fail by filling up with a dilute blanket as shown in numerous stress testing conducted (Daigger 1998). LOSS ability may not solely lie in discerning a flux curve but extend to identifying a critical solids concentration. A new hypothesis was formulated from this study, stating that LOSS may indicate the maximum solids concentration to exist in a feed zone to avoid failure.

The goal is to make a method that can be applied in the field for estimation of clarifier performance. Flocculent settling (stokesian) is predominant within ideally operating clarifiers, and the shift to 'slower' hindered settling (non-stokesian) causes both failure and poor effluent quality. Therefore, an investigation was started to characterize settling transition and relate it to clarifier performance. In this paper, LOSS is determined for different sludge matrices. Additionally, LOSS for secondary systems are monitored over a 60 day period, and its role compared to conventional settling characteristics measurements was evaluated.

3.6 MATERIALS AND METHODS

3.6.1 Experimental Design, Sample Location, and Preparation

Blue Plains AWWTP is the largest advanced wastewater treatment plant in the world. The plant treats on average 375 million gallons of raw sewage per day, originating from Washington DC, and neighboring counties in Maryland and Virginia. Blue Plains was chosen to test out the proposed methodology due to its high rate (1-1.5 day SRT) secondary systems. Large changes in sludge quality over a short period of time is a byproduct of short SRT systems due to aggressive wasting strategies. This allows for the sensitivity and variance testing of the proposed methodology. On the other hand, low rate systems that are more stable in terms of sludge quality can gauge the reproducibility of the proposed methodology. Secondary and Biological Nutrient Removal BNR samples were collected at the end of the reactor passes, at a channel before being fed to the clarifiers. Secondary samples were taken from the high rate process over a 60 day period, flux theory parameters and LOSS were compared based on their assessment of clarifier performance. Basic characteristics of the mixed liquor samples are presented in Table 3.1. Sampling was done using a bucket instead of a pump to avoid shearing of the floc. Samples were collected at the same time every day to avoid variability due to diurnal flow. No less than 250 L of samples were collected during trials. Supernatant from settled samples was used for dilutions to avoid altering the water chemistry.

Mainstream sludge was obtained from mainstream deammonification pilot at Blue Plains. The mainstream pilot simulates the BNR system at Blue Plains with the difference that aggressive aerobic SRT (1-5 days) are applied under transient anoxia and anammox granules are bio-augmented. Therefore this system can be seen as a hybrid system with a small fraction of granules. Sidestream sludge was obtained from sidestream deammonification pilot at Blue Plains. The sidestream pilot was a sequencing batch reactor, which was treating centrate from the Alexandria Sanitation Authority. The reactor operated at high SRT of 30 days to allow retention of anammox and is characterized by a large fraction of granules with only a small fraction of flocs. Seed sludge is granular sludge provided by the Strass WWTP, and was collected from the DEMON® cyclone's underflow. The DEMON® cyclones are physical separation devices that select sludge based on particulates density (Wett et al. 2010). This sludge was used as a granular example; it was artificially obtained by the use of a cyclone and thus not directly related to an operated system. Basic characteristics of the granular hybrid activated sludge samples are also presented in Table 3.1.

3.6.2 Materials for Image Acquisition and Analysis

A Kodak® Zi8 High definition camcorder (F2.8/f=6.34 mm) was used to capture videos. The videos were recorded at 1080p at 30 frames per second. The camera was set 0.35 m away from the settling jar. A detailed diagram of the experimental setup can be seen in Figure A.2 of Supplementary Data. Videos recorded were converted to an avi format for analysis with Mplayer ver 1.1.1. Analysis was conducted with MathWorks® MATLAB® R2010b.

3.6.3 Materials for LOSS Determination

A 2 L Thermo Scientific™ Nalgene™ Settleometer made from clear polycarbonate was used to determine LOSS. A polypropylene paddle was used to stir dilutions and stop vortexes forming. Thermo Scientific™ Nalgene™ graduated cylinders made from polypropylene ranging from 500mL to 4000mL were used for measurements of mixed liquor samples. Large batches of sludge were stored in Sterlite® 62.5 L storage bins.

3.6.4 Column Testing

A settling column was fabricated in accordance with the Clarifier Research Technical Committee (CRTC) of the American Society of Civil Engineers and is identical to that used by Wahlberg and Keinath (1988), and Wahlberg et al. (1995). State point analysis and Stirred Sludge Volume Index ($sSVI_{30}$) procedures were conducted in this column to obtain a solids flux curve (Wahlberg and Foundation 2001). The TSS range was modified to be between 900 mg TSS/L to 2500 mg TSS/L for the generation of solid flux curves. This range gave a representation of what occurs close to the LOSS zone.

3.6.5 LOSS Experimental Procedure

In order to generate the LOSS, a series of dilutions were made of mixed liquor collected from secondary and BNR reactors (Table 3.1). The dilutions were produced between the ranges of 300 mg TSS/L and 1200 mg TSS/L with a maximum gap between dilutions of 100 mg TSS/L. Two liters of each batch was placed in a clear settleometer. The content of the settleometer was then mixed without a vortex forming and settling was observed visually and filmed for five minutes. The videos were then analyzed with the commercial MATLAB program to determine LOSS. TSS of each batch was measured using the protocols from Standard Methods (Eaton et al. 2005) and the observed LOSS was recorded, illustrating the point at which the LOSS occurs in relation to TSS concentration. To detect LOSS, one must start with a high solids concentration mixture, ensuring an interface which is easy to detect visually. One must then make serial dilutions using the same sludge. LOSS is the highest solids concentration dilution with no detectable interface. LOSS was done in triplicates to assess reproducibility.

3.6.6 Image Analytical Tool

The program goal is to highlight an interface and graph it. The MATLAB program was based on Kim et al. (2011) , and characterized the interface by changes in color intensities. The program collects still images from videos and converts all still images into a RGB matrix. The RGB matrix describes each pixel in the still as three number which reflect the pixel's color. The three numbers represented color intensity values for red, green, and blue, respectively. Intensity values range from 0 to 255, lighter colors have higher intensities and vice versa for darker colors, resulting in black being expressed as 0,0,0 and white as 255,255,255. Secondary and BNR sludge from Blue Plains AWWTP have a reddish brownish hue attributed to biomass present (brown) and ferric salts added (red). Out of all three RGB values, the red index changes the most when characterizing pixels from supernatant and sludge. Varying degrees of the red intensity values correspond to a solids concentration, red intensity values increases with a decrease in solids concentration. Specific red intensities were selected according to their location coordinate, arrays of red intensities were collected spanning the width and the depth of the jar excluding the walls. The red intensity values spanning the width of the jar are averaged and plotted on the x axis, with corresponding depth on the y axis.

For each video of batch settling there was a series of graphs that detailed chronological evolution of red intensities according to depth. There are three states in a batch settling test, with accordance to time line there is flocculation, settling, and compression, respectively. Flocculation time is the period of solids aggregating to larger particles. Next comes the settling period as larger particles that were formed settle. The last period is defined as the compression phase, when solids that have collected at the bottom of the jar began to compress. The interface is always present during the compression phase, but only present in the settling phase for batch mixtures that exhibit hindered settling. The difference between a hindered interface and an interface produced by compression alone is that the former starts within the top quartile of the settling jar and progresses downwards whiles the latter starts at the bottom quartile and progresses upwards a magnitude slower than the previous.

If an interface is present the graph generated will be similar to a step input curve, with three distinct sections (Figure 3.2A, B). Starting from the water surface and moving towards the bottom of the jar there are three key regions: supernatant, interface, and compacting sludge. Supernatant section will be a vertical line with a steady red index value, the interface section details a sharp slope with a sudden decrease in red index values with increasing depth. The compacting sludge section is another vertical line spanning the remainder of the jar depth with significantly higher red intensity values than the first section. When there is no interface present, there will be a steady slope of ranging red intensities across the entire depth of the jar (Figure 3.2C). As the solids concentration in a series of dilution approaches LOSS, the interface weakens, the dramatic shift in red index values becomes less pronounced. An interface is no longer present when there is no shift in red intensities greater than 10 within 2 cm jar depth during the settling period. The highest concentration that does not exhibit an interface is classified as LOSS.

3.6.7 Pilot Clarifier

A pilot clarifier was built in accordance to Kinnear's (2002) dissertation. The pilot clarifier is an acrylic column with an inner diameter of 20 cm and total height of 305 cm. Samples are fed through two opposing valves situated at the midpoint of the column (152 cm). The pilot clarifier was operated under an open system, with recurrent 300 L batch collection of secondary mixed liquor fed continuously to the clarifier, while overflow and underflow are disposed down the drain. Masterflex® peristaltic pumps were used for feed and underflow pumps. Sample ports were located at 15 cm intervals such that undisturbed samples could be collected throughout the column depth.

3.6.8 LOSS Validation

To validate LOSS as a precursor for settling characteristics, LOSS was determined for five biologically distinctive types of sludge. All the flocculent sludge types were collected from operational reactors at Blue Plains AWWTP. Table 3.1 indicates that BNR has faster non-stokesian settling sludge that compresses better than secondary. The granular sludge was provided by the Strass WWTP, and was collected from the DEMON® cyclone's underflow.   The hybrid systems sludge was collected from pilot systems ran by the research department at the Blue Plains facility. These pilots were seeded from the same Strass cyclone underflow.

Moreover, the effect of the chemical matrix on the settling properties and the determination of LOSS was determined. According to Novak et al. (2002), increasing the M/D ratio retards settling characteristics. Therefore to see if chemical alteration of settling characteristics causes a shift in LOSS, sodium ions in the form of sodium hydroxide were added to secondary mixed liquor. Sodium hydroxide was chosen based on their use at Blue Plains AWWTP to alter pH and by increasing pH above 8 will deteriorate settling characteristics. A 2 L jar of secondary was spiked with 420 mg NaOH/L and LOSS values were compared to a control.

In the third step of validation fourteen trials spanning a period of 60 days were conducted on secondary sludge at Blue Plains AWWTP. LOSS was carried out alongside column testing which involved generating solid flux curves and $sSVI_{30}$ determinations in order to compare each method. All the analyses were conducted within half an hour of each other in order to minimize any effects due to sludge aging. Trials were a minimum of two days apart in order to ensure sample variability, since high-rate secondary systems at Blue Plains were run at an SRT between 1.5 to 2 days.

In the fourth step, Takacs et al. (1991) model was run with data obtained during 60 days of column testing. LOSS data collected from experimental approach was plotted against Takacs model's percieved LOSS (maximum peak or solids concentration before settling velocity declines with solids concentration). This showed whether Takacs et al. (1991) model and LOSS experimental procedure were in agreement on the beginning of non-stokesian settling. Furthermore, sensitivity of the methodology and variance in detection could be examined across a large range of clarifier performance.

Lastly, a pilot clarifier was run under three scenarios: operational baseline, peak rate scenario, and failure scenario (Table 3.2). The first two scenarios were run until steady-state conditions were met (3 HRTs) before samples were collected. Two samples were collected from each sample location, one HRT apart. Sample locations included effluent, underflow, feed, and clarifier column. The clarifier column was divided into three sections: feed layer, top layer, and bottom layer. The feed layer comprised of 0.15 m (6 in) above and below the feed valves. The top layer spanned the section above the feed layer. Three samples were collected every 0.30 m (1 ft) starting 0.30 m below the water surface from the top layer. The bottom layer is the portion below the feed layer. Three samples (every 0.30 m) were also collected from the bottom layer starting 0.30 m above the bottom of the clarifier. The operational baseline was the first scenario and the pilot clarifier was run in according to Blue Plains' secondary system's six week operational hydraulic surface flow rate average. The goal of this scenario was to validate the absence of hindered settling in an operational clarifier. The second scenario was defined as peak rate, where the operational parameters were set at Blue Plains' peak flow rates. The goal of the peak rate scenario was to determine whether non-stokesian settling became prominent when a clarifier is pushed closer to its capacity. The last scenario involved artificially thickening the feed through decanting of supernatant as well as increasing the SOR past Blue Plains' designed limits to obtain a failure scenario. In the final scenario samples were taken from the feed layer and top layer every 15 minutes until clarifier failure. Failure was defined when effluent solids concentration was above 10% of the feed concentration.

3.6.9 Statistical Analysis

To establish variance in LOSS determination, t-test was conducted using Microsoft Excel®. For trials on biological and chemical alteration there is a directional hypothesis thus two-tail was assumed. Biological differences were assumed to be unequal variance, while chemical alterations of the sample is a repeated measure design. The fourteen LOSS numbers collected during 60 days secondary clarifier observation underwent statistical analysis to elucidate any variances. A matrix was constructed to compare p values between trials and a t-test was conducted on the modeled and observed values at low (below 600 mg TSS/L) and high (above 600 mg TSS/L) measured LOSS values. This was done to see if there was a notable difference in measuring poor or superior settling sludge.

3.6.10 Settling Velocity Models

Takacs et al. (1991) developed a double empirical equation (eq. 2) that was built upon Vesilind (1968) exponential equation (eq. 1). Vesilind equation entails two parameter estimations (V_0 and r_h), however Takacs model requires three parameter estimations (V_0, r_p, and r_h). Employing similar analytical methods as Torfs et al. (2013), Takacs parameters were estimated from the Vesilind equation.

$$v_S = V_0 e^{-r_h(X_f - X_{min})} - V_0 e^{-r_p(X_f - X_{min})} \qquad (2)$$
$$with\ X_{min} = f_{ns} \cdot X_f$$

Firstly, plots from several dilutions of solids concentration were analyzed (S3 A, see Supplementary data), linear portion's slope of the settling curve represented hindered settling velocity at corresponding solids concentration (S3 B, see Supplementary data). Hindered settling velocities were plotted against solids concentration, yielding an exponential trend line. Both Vesilind equation parameters (V_0 and r_h) were acquired from the exponential equation of the trend line (S3 C, see Supplementary data).

Secondly, initial V_0 and r_h for Takacs model were obtained from previously obtained Vesilind parameters, and initial r_p value was set to tenfold of the r_h (Takacs et al. 1991). Starting from initial estimates, a parameter optimization was performed by minimizing the Sum of Squares Errors between the settling velocities that were measured from column tests and the velocities that were predicted by the settling velocity function (Torfs et al. 2013). The Nelder-Mead algorithm (Nedler and Mead 1965), which is based on the Simplex algorithm was used for optimization with MATLAB. The resulting parameters were computed and graphed (Figure A.3D, Table A.1, see Supplementary data).

3.7 RESULTS

3.7.1 Sludge Matrix Effects on LOSS

Table 3.1 shows $sSVI_{30}$ indices were highest for Mainstream and Secondary with values of 121 mL/g and 116 mL/g, respectively. BNR $sSVI_{30}$ was 88 mL/g and Side Stream had similar $sSVI_{30}$ indices that ranged from 81 to 88 mL/g. Seed had the lowest recorded $sSVI_{30}$ with a value at 21 mL/g. Flocculent systems had the lowest ISV values, Secondary and BNR had values of 3.3 m/h and 4.0 m/h, respectively. Side stream had an ISV of 4.8 m/h which was the highest recorded value. Mainstream and Seed had similar ISV values of 4.4 and 4.5 m/h, respectively. However Seed's ISV could not be detected at 2000 mg TSS/L as no interface was present at that concentration. To achieve an interface that can easily be tracked, a minimum of 6500 mg TSS/L was required. Seed with 5500 mg TSS/L was documented with the highest value for LOSS which corresponds to the highest settling velocity. Side Stream was the second highest with a LOSS of 1600 mg TSS/L. Secondary, Mainstream, and BNR LOSS were closer together with values of 1000, 1100, 1200 mg TSS/L, respectively.

3.7.2 Wastewater matrix effects on LOSS

Sodium hydroxide effect on LOSS is illustrated in Figure 3.3. All three trials showed that LOSS decreased significantly ($p < 0.001$) by an average of 100 mg TSS/L when spiked with sodium hydroxide. Error bars increased for samples spiked with NaOH due to a weak interface that fluctuated in appearance at solid concentrations close to LOSS.

3.7.3 $sSVI_{30}$, gravity flux curves, and LOSS over 60 day period

3.7.3.1 Secondary Clarifiers Operations

Figure 3.4 highlights operational configurations of secondary clarifiers that were the basis for settling characteristics tests. Over the 60 days of observation, effluent TSS averaged around 11.4 ± 5.3 mg TSS/L. For the first 22 days effluent TSS was stable at 9.8 ± 3.7 mg TSS/L, but that period was followed by a sudden rise in TSS to 19 mg TSS/L that lasted two days. Effluent TSS for the next 16 days averaged 7.6 ± 2.6 mg TSS/L. It was in between operational day 41 and day 60 that effluent TSS deteriorated and averaged 16.2 ± 3.8 mg TSS/L.

Daily solids wasting was scattered for the first 20 days ranging from 0 to 24 t/d, peaking during days 16 and 17 when effluent quality was at its worst. Days 21 to 32 shows wasting was more consistent at around 20 t/d, with five outliners when wasting ranged between 40 to 100 t/d. The first two outliners were days 23 and 24, when effluent quality was very high at 19 mg TSS/L. The remaining three outliers that happened at days 26, 31, and 32, do not correlate with a high effluent TSS. After Day 32, wasting was consistent at 14 ± 3 t/d. Faulty equipment led to over wasting during days 23, 24, 26, 31 and 32. The excessive wasting caused a visible change in microbiology of the sludge at day 41 (see Figure A.4 in Supplementary Data).

From Figure 3.4, days 1 through 27 show a rise in daily solids loading rate (SLR), with a peak solids load of 121 kg/(m^2·d) on day 18. From day 28 to 34 solids loading halved from 80 to 40 kg/(m^2·d). Days 35 to 60 show that solids loading was kept around 40 kg/(m^2·d) with intermittent days when solids loading averaged around 60 kg/(m^2·d).

Figure 3.5 presents how $sSVI_{30}$ changed during 60 days of settling characteristics observations. In the first week, $sSVI_{30}$ was below 100 mL/g, however $sSVI_{30}$ recorded on day 15 which was 135 mL/g showed a sharp rise. $sSVI_{30}$ continued to steadily rise reaching a record of 163 mL/g on day 44, before steadily dropping to 88 mL/g on Day 60. Operational period between day 53 and 60 showed $sSVI_{30}$ stayed below 100 mL/g. SVI determination on samples ranging from 900 to 2500 mg TSS/L showed a significant variance with a standard deviation of ± 17.5% (Figure 3.6).

3.7.3.2 Gravity flux curves

Figure 3.6A and B show the various gravity flux curves during the length of observation. Figure 3.6A shows the observation during the first 32 days whiles Figure 3.6B shows the remainder. Flux curves from the first 32 days follow two trends, the first fifteen days show superior sludge quality than the remaining 17 days. The descending curve after the peak for the first 15 days are almost identical with r_h values averaging 0.76 ±5, were as the 17 days after have k values averaging 0.96 ±4.5. Contrary to the previous operational days, the remaining period suffered from severe fluctuation in sludge quality. Day 39 and 60 had the lowest and highest V_0 of 6.97 and 22.26 m/h, respectively. Day 46 and 51 had the smallest and largest r_h values of 0.53 and 1.2, respectively. The peak solid flux for a majority of the 60 day observation period fell between 4 and 6 kg^2(m^2•hr), with four flux curve exceeded that range.

3.7.3.3 Comparison of sludge quality metrics

Figure 3.7 plots LOSS, Clarifier Operational Area, and Average sSVI30 over the 60 day observational period. Day 3 had the highest LOSS recorded at 830 mg TSS/L, LOSS then proceeded to drop over the next 25 days to its lowest of 555 mg TSS/L on day 25. A peak was observed on day 29 when LOSS rose to 760, but then dropped to 630 mg TSS/L on day 39. LOSS then steadily dropped over the next 10 days to 590 mg TSS/L, its second lowest value. LOSS then increased and stayed above 650 mg TSS/L for the remainder of time with minimal fluctuation.

The workable clarifier operation is the area under the flux curve, and represents the capacity of a clarifier in terms of storage, overflow, and underflow. The workable clarifier operation started off at 15.5 $kg^2(m^5 \cdot hr)$ and rose over the next five days to around 20.5 $kg^2(m^5 \cdot hr)$. After day 5 the clarifier operational area declines and reaches 9.3 $kg^2(m^5 \cdot hr)$ on day 22, there it stays stable up to day 32. However, the remaining period showed severe fluctuation in sludge quality. A brief stable period on days 39 and 44 where the flux curves had similar operational areas of around 13 $kg^2(m^5 \cdot hr)$, lead to a rapid increase in operational area on Day 46 followed by the lowest recorded operational area on day 51, where operational area was around 17 and 8 $kg^2(m^5 \cdot hr)$, respectively. Days 53 to 60 showed dramatic improvements in sludge quality, where the clarifier operational area rose continuously in those 7 days to a record high of 29 $kg^2(m^5 \cdot hr)$ on the 60^{th} day.

The average $sSVI_{30}$ was first recorded at around 95 mL/g, it then dropped to the lowest recoded value of 81 mL/g on the 5^{th} day. $sSVI_{30}$ rose and stayed above 120 mL/g over the next 30 days, reaching a peak of 163 mL/g on day 39. The following days after day 39 showed a decrease of SVI30 around 95 mL/g where it stayed stable for the remainder of the observational period.

3.7.3.4 Pilot clarifier study

Figure 3.9 shows the solids concentration profile in the pilot clarifier. Sludge tested in the pilot clarifier had an ISV, sSVI30, and LOSS of 4.2 m/hr, 30 mL/g TSS, and 1200 mg TSS/L, respectively. Under the first scenario, effluent solids concentration remained at 46 ± 3 mg TSS/L. Furthermore, the top and bottom layer both averaged 50 ± 10 mg TSS/L and 272 ± 9 mg TSS/L, respectively. There appeared to be no solids concentration gradient, or sludge blanket (A.5A, see supplementary data Figure). The second scenario also exhibited no solids gradient or sludge blanket but an elevated solids concentration with the top and bottom feed layer averaging 72 ± 4 mg TSS/L, and 403 ± 8 mg TSS/L, respectively (A.5B, see supplementary data Figure). The third scenario failed in 0.5 HRT (30 min), allowing two sample collections. Fifteen minutes into the failure scenario, effluent solids concentration and feed layer solids concentration were 130 mg TSS/L, and 1375 mg TSS/L, respectively, thus reaching LOSS at the feed layer. The top layer's solids concentration profile starting from the highest elevation were 130, 150, and 687 mg TSS/L. After thirty minutes, the effluent solids concentration had reached 590 mg TSS/L and the feed layer was recorded at 2400 mg TSS/L. The top layer concentration profile starting from the top were 723, 1000, and 1510 mg TSS/L. At the failure point there was a solids concentration gradient in the top layer and a sludge blanket was recorded at a height of 0.91m.

3.8 DISCUSSION

This study firstly hypothesized that LOSS would characterize settling velocity. From Table 3.1, similar trends between LOSS and ISV are observed, however correlation is rather low (r = 0.40). This might be caused by the different nature of the different sludge types. Granular sludge suggested by (Beun et al. 2002) as a pathway to negate poor settling in secondary clarifiers due to their superior settling characteristics was shown to have the highest LOSS (seed, Table 3.1). Additionally, the portion of granules within a sludge matrices affected LOSS, the larger the granular fraction the higher the LOSS and settling velocity as evident in mainstream, sidestream, and seed (Table 3.1).The extremely high LOSS for granular sludge is in accordance with literature that documents settling velocities for granules to be between 10 – 60 m/h (Winkler et al. 2013), compared to floc settling velocities that are between 1- 14 m/h (Griborio and McCorquodale 2006, Kinnear et al. 2001). When looking within the same system, i.e. secondary system over a period of 28 weeks, a stronger correlation (r = 0.71) between ISV and LOSS was detected (see supplementary data Figure A.6A). Shortcomings have been shown for $sSVI_{30}$ when settling is good ($sSVI_{30}$ below 50 mL TSS/g). When settling is good, compression settling limits $sSVI_{30}$'s ability to characterize differences in settling, as observed by the plateauing in $sSVI_{30}$ data below 50 mL/g TSS (see supplementary data Figure A.6B). Consequently, LOSS marks the transition point between settling regimens and is not skewed by non-stokesian artifacts and differences in sludge matrices, making it a better indicator of settling velocity. If non-stokesian settling described as region 4 (Takacs et al. 1991) starts later, so at higher LOSS, it would mean that the settling velocity distribution is localized at a higher range. Mainstream compared to BNR sludge had a marginally faster hindered settling velocity but a larger $sSVI_{30}$ value indicating poorer compression. This characteristic cannot be captured in one ISV at a given value (i.e. 2000 mg TSS/L) but a series is needed to create a flux curve that illustrates the full nature of settling characteristics. LOSS is however at an advantage in two ways, firstly, a single LOSS value characterized BNR as having better settling properties than mainstream. Secondly, since the prevalence of hindered settling in a clarifier is a precursor for failure, LOSS provides a maximum solids loading rate. Previous studies have found correlations between SVI-type measurements with flux theory (r_h and V_0 in equation

1) (Daigger 1995, Pitman 1985). SVI measurements in this study, which were all done at 2000 mg TSS/L correlated with LOSS data, indicating LOSS's proportionality with settling characteristics.

Increased M/D ratios have shown before to decrease settleability due to impair flocculation. The latter was shown by Novak et al. (2002) who showed higher M/D deteriorated the di-cationic bridging which is necessary for flocculation. This study showed a significant decrease in LOSS at higher M/D ratios (Figure 3.3). However the degree of change in LOSS was not as drastic as expected (Novak et al. 2002). This might indicate that the lower LOSS values are more limited by flocculation. Therefore, the lower limit of LOSS will be dependent on a minimum TSS concentration, which allows for the formation of flocs, rather than the settling velocity distribution as such.

Varying degrees in settling characteristics helped test the sensitivity, and proportionality of $sSVI_{30}$, ISV, and LOSS. The primary basis of design and control of secondary clarifiers in the United Kingdom is $sSVI_{30}$ due to its quickness and ease (Bye and Dold (1998) . According to Ekama and Marais (1984), $sSVI_{30}$ produces reproducible results between solids concentration and $sSVI_{30}$ for solids concentrations of up to 10 or 7 g TSS/L for good and poor settling sludge, respectively. However, White's (1976) observation conflicts with Ekama and Marais (1984), White (1976) studied wastewater sludge from 30 activated sludge systems and denoted a poor correlation between hindered settling and solids concentration below 3.5 g TSS/L. White's observation aligns with the $sSVI_{30}$ results recorded for this study, $sSVI_{30}$ from Figure 3.7 shows a standard deviation $> \pm 17.5\%$. This large deviation strongly suggests a non-linear correlation between $sSVI_{30}$ and solids concentration between the ranges of 900-2500 mg TSS/L and emphasizes $sSVI_{30}$ inability to resolve small increments in sludge settling characteristics. Therefore any $sSVI_{30}$ would have to be done at the same solids concentration, and above 3.5 g TSS/L to be comparable. $sSVI_{30}$ data identified three distinct periods of comparable settling characteristics; initial period (day 1-10) good settling characteristics, mid period (day 15-50) poor settling characteristics, and final period (day 53 -60) a return to good settling characteristics. The three periods correlated with both flux curves and LOSS but lacked the dynamic capabilities to spot spikes in settling characteristics (Figure 3.7). Plants such as Blue Plains AWWTP that have an MLSS below 3.5 g TSS/L will not benefit from $sSVI_{30}$, due to $sSVI_{30}$ limitation in being comparable and identify minor changes in settling characteristics.

Flux curves unlike $sSVI_{30}$ seemed much more capable of showing consistency in measurements for sludge quality evident from the negligible differences in ISV duplicates (std. = 4.04%). Flux curve and LOSS on a macro level showed the same message of three distinctive periods during 60 days of secondary sludge observations. The periods were good settling followed by a period of bad settling then a return to good settling sludge. However, flux curves and LOSS did not correlate in terms of scale of improvements and there were two occurrences whereby LOSS and $sSVI_{30}$ were in disagreement with the corresponding flux curve. The first occurrence was on days 44-46 when both LOSS and $sSVI_{30}$ recorded a decrease in settling characteristics but flux curves indicated the opposite. LOSS decreased by 50 mg TSS/L, sSVI30 increased by 5 mL/ g TSS, and clarifier operational area increased by 5 $kg^2(m^5•hr)$. The second occurrence was on days 53 to 58. LOSS and $sSVI_{30}$ remained stable but flux curves showed a 10 $kg^2(m^5•hr)$ increase. It should be noted that day 29 was an outlier for LOSS. The short SRT of 1.5-2 days completely altered the settling characteristics of the sludge in 3 days leaving LOSS to show a different snapshot of settling characteristics on Day 29 to a flux curve on Day 32. During the observed operation, RAS averaged 3000 mg TSS /L more than MLSS, however, on day 29 the difference was at its lowest of 380 mg TSS/L. The negligible difference in RAS and feed concentration suggests that the clarifier was operating with no blanket. SLR increased by 28% from the previous day, and SOR decreased by 3.7%, leaving only superior settling sludge as the prime culprit for the absence of a sludge blanket. By day 32 when a flux curve was determined, sludge settling characteristics reverted back leading to the highest recorded RAS of over 13000 mg TSS/L suggesting a very high sludge blanket. At this time a problem in the wasting strategy occurred leading to the highest recorded waste of 98 t/d.

Flux curves show dramatic improvement in settling characteristics during the last four curves. Day 53 to 58 show an increased operational area for a clarifier which does not correlate to $sSVI_{30}$ and LOSS (Figure 3.7). According to operational data, day 53 to 60 show a reduction in SLR and SOR, from 80 to 36 $kg/(m^2•d)$ and 1.3 to 0.63 m/hr, respectively. The influent solids concentration remained constant at 2400 mg TSS/L while the underflow solids concentration dropped by 200 mg TSS/L. Furthermore, on day 60 feed solids concentration was lower by 400 mg TSS/L while SOR and underflow solids concentration increased by 0.1 m/hr and 600 mg TSS/L, respectively. Unlike LOSS's peak on day 29, flux's peak on day 60 has no operational backing, reinstating the hypothesis that hindered settling is not present in a clarifier and velocities gained from them are poor modeling parameter. Additionally, the lack of a correlation between flux curves and LOSS supports claims stating 1D flux theory models being a major weakness in CFD models (Ekama and Marais 2002). Flux theory's focus on hindered settling conditions alone to predict clarifier capacity might be what is limiting clarifier capacity estimation. LOSS inclusion of stokesian settling properties might be more advantageous in clarifier capacity estimation LOSS during the 60 days of operation varied significantly ($p \leq 0.003$) when operational conditions were vastly different. Similarly, days when conditions were very similar (i.e. days 53 – 60), no significant difference in LOSS was detected ($p>0.05$). Operational conditions dictate sludge properties, especially in a high rate system. This suggests that LOSS unlike flux curves does not fluctuate easily under similar conditions, but can indicate when a key shift in sludge quality arises. Day 4 and day 10 show a key shift, which is mimicked in operational data by elevated effluent quality and low SLR to counter sludge quality. Day 29 and 32 show a major shift as well: Day 29 LOSS was at its highest and operators increased SLR due to an absence in sludge blanket. At day 32 LOSS was 20% lower than day 29, operators quickly tried to increase the underflow rate to mitigate the high sludge blanket resulting in over wasting.

From Figure 3.8 shows modeled versus experimental LOSS, and a favorable trend can be seen. A total of fourteen LOSS values were collected. However only ten data points were compared. The omitted days are highlighted in supplementary data Table A.1. The first five LOSS values are compared to the ISV data values collected within the 24 hour period. Column data on day 32 was not compared to LOSS on day 29. The three days that were omitted are (days 2, 15, and, 53), was due to model failure. The method used was sensitive to ISV data collected at low solids concentration, ISV values on the omitted days had multiple entries below 1 g TSS/L/. All model runs for the omitted days predicted LOSS at 0.1 g TSS/L, even with varying corresponding Vesilind parameters. Furthermore, there was no significant difference ($p > 0.05$) between low LOSS values and high LOSS values. This suggests that the accuracy of the LOSS test is not function of sludge quality.

The sludge quality from the pilot clarifier study differed significantly from the previous experiments with sSVI30 and ISV being comparable to granular sludge. However, effluent quality was poor in the pilot clarifier and full scale clarifiers at Blue Plains at 46 ± 3 mg and 37 ± 8 TSS/L, respectively. There is an apparent disconnect between effluent quality and metrics tested (ISV, sSVI30, and LOSS). All these measurements focus on what settles and the rate whiles providing no information on the non-settleable fraction. The sludge matrix's settling fraction was very good, and as a result the last scenario had to be modified for failure to occur. In the original failure scenario, clarifier was operated at a SOR of 4.0 m/hr (2360 gpd/ft2) and was fed with unmodified secondary mixed liquor. However, mixed liquor concentrations fluctuated dramatically due to diurnal flow during the 24 hour study. The feed solids concentration was 1600 mg TSS/L during the commencement of the final scenario, and the effluent quality was 87 mg TSS/L at steady state. The feed layer was unable to reach LOSS, and therefore a hindered profile was never reached. To guarantee that the feed layer would reach LOSS and achieve failure, mixed liquor collected for the feed was thickened.

In the first two scenarios, Figure 3.9 showed stokesian as the main settling mechanism. Furthermore, when hindered settling was observed in the feed layer during the failure scenario (after 15 min), the bottom layer exhibited flocculent settling up to the sludge blanket. Additionally, a hindered profile was captured (S7A&B, see supplementary data) extending up from the feed layer, just before failure. These observations in addition to knowledge of the original failure (low feeds concentration, but high SOR) scenario validate that hindered settling in an operational clarifier is a precursor for failure. LOSS measured corresponded to the appearance of a hindered profile in the clarifier. Fifteen minutes into the failure scenario when solids in the feed layer were above LOSS, a solids concentration gradient was beginning to form in the top layer. Since there had never been a solids gradient in the last two scenario the presence of one forming strongly suggests that failure is imminent, which it was.

Consequently, the LOSS number just focuses on mechanics of settling and forgoes the flocculation and coagulation aspects of settling. LOSS number is still lacking a significant amount of information such as distributions of particle size, density, settling velocity. Indications on these key parameters can be integrated into computational fluid dynamic models to improve their solid separation predictive capabilities. Further work needs to be done to find correlations between LOSS and these stated parameters. Additional stress tests needs to be performed to isolate effects of SOR and SLR limits with LOSS. Additionally, LOSS gives no information as to non-settleable solids, or whether there is bulking occurring. The focus on stokesian settling metrics is the necessary step forward to provide data for better modelling, but more work is necessary to provide secondary effluent predictions.

3.9 CONCLUSION

LOSS was obtained experimentally and validated with Takacs et al. (1991) model. LOSS was proven to increase for better settling sludge and vice versa for bad settling sludge and proven to not significantly change in its accuracy when compared to Takacs et al. (1991) model. Gravity flux curves unlike LOSS exaggerated good settling sludge and seemed to change dramatically for small increments in settling characteristics. LOSS was able to identify good settling sludge and quantify how good or bad they are. Moreover LOSS test takes half an hour whereas generating a gravity flux curve takes over four hours. $sSVI_{30}$ was a good measure for drastic changes in sludge characteristics, but a more poor method to quantify settling characteristics. The pilot clarifier study validated two hypothesis; there is no hindered settling in a functioning clarifier and LOSS is the maximum feed layer concentration before clarifier failure occurs. Furthermore, future work needs to be done to test the hypothesis that LOSS values can be directly used by operators to ensure that the feed zone in a clarifier does not exceed LOSS enabling for an efficient way of maximizing the load to a clarifier without threat of failure.

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3.11 TABLES

Table 3.1: Type of samples taken from the Blue Plains AWWTP and Strass WWTP with their respective SVI30, ISV and LOSS.

Sludge Type	Description	Location	SVI (mL/g)	ISV* (m/hr)	LOSS (mg/L as TSS)
Secondary	Flocculent	BPAWWTP West channel	116	3.3	1000
BNR	Flocculent	BPAWWTP Even Channel	88	4.0	1500
Main Stream	≈80% Flocculent (Hybrid)	DCWRD	121	4.4	1100
Side Stream	≈60% Flocculent (Hybrid)	DCWRD	81-88	4.8	1700
Seed	Granular	Strass WWTP Cyclone underflow	21	4.5**	5500

*ISV was measured at 2000mg TSS/L
**ISV was measured at 6500mg TSS/L, a high solids concentration was necessary for an interface to occur.

Table 3.2: Operating parameters for the three scenarios run on the pilot clarifier with SOR = 1, 2, and 3 m/hr, RAS = 75, 50, and 25%, SLR = 60, 150, 300 kg(m^2d).

Scenario	SOR (m/hr) [gpd/ft^2])	RAS Ratio (%)	SLR (kg(m^2d)	Feed Concentration (mg TSS/L)
Operational Baseline	1[590]	75	60	2400
Peek rate Scenario	2[1200]	50	150	3100
Failure Scenario	3[1800]	25	300	4100

3.12 FIGURES

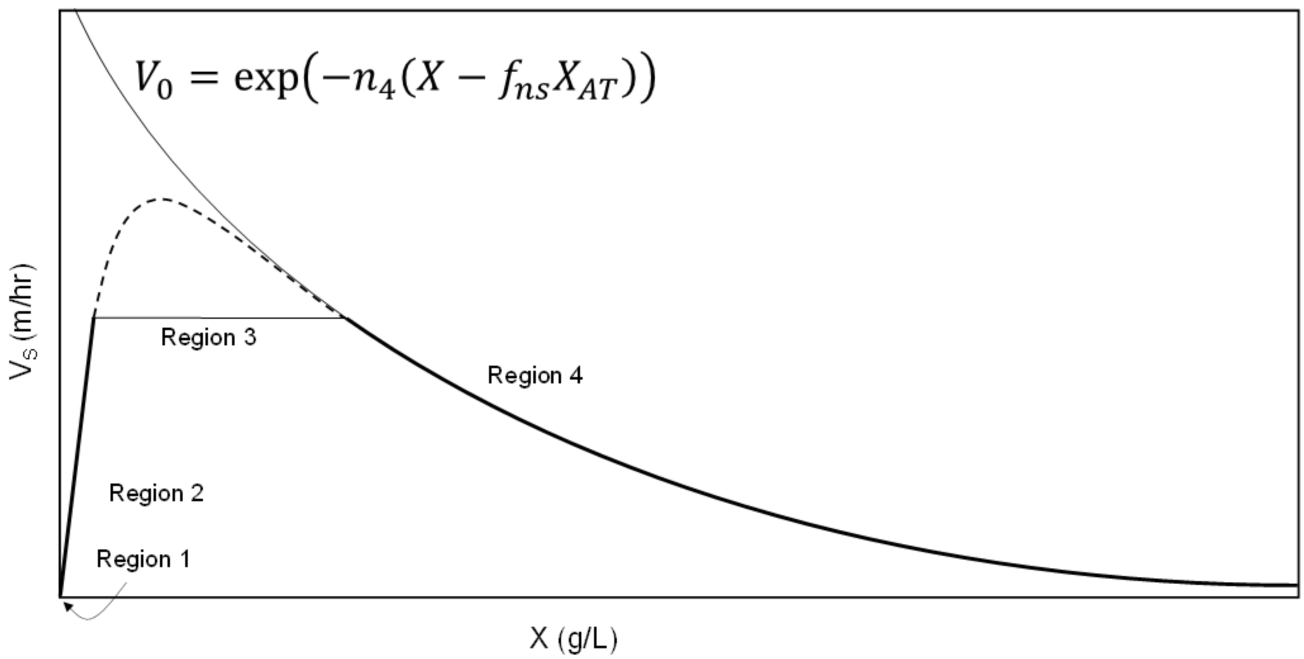

Figure 3.1: Settling function of Takacs et al., (1991) incorporating the settling characteristics of both dispersed and flocculated suspended solids.

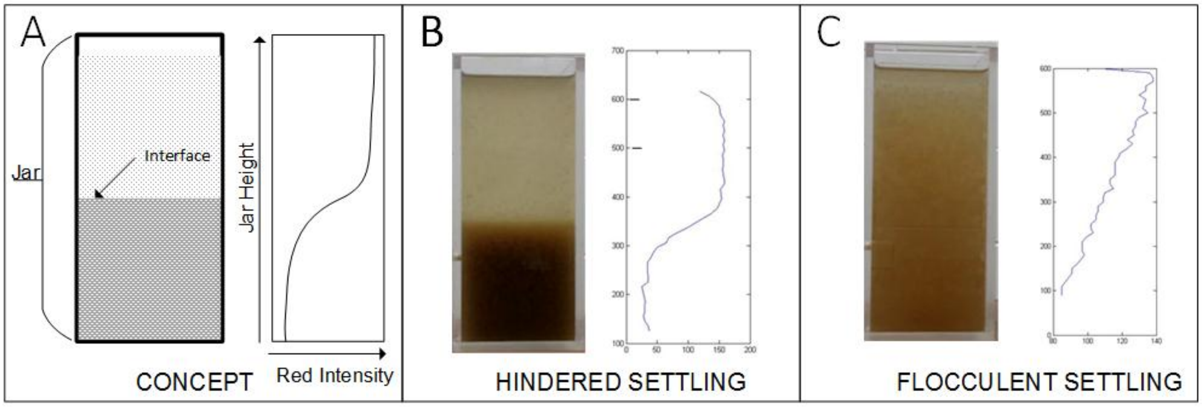

Figure 3.2: (A) A jar with sludge settling in a hindered manner with an interface, and to the right is the jars height in pixels on the y axis plotted against red index value on x axis. (B) and (C) depict hindered and flocculant settling respectively.

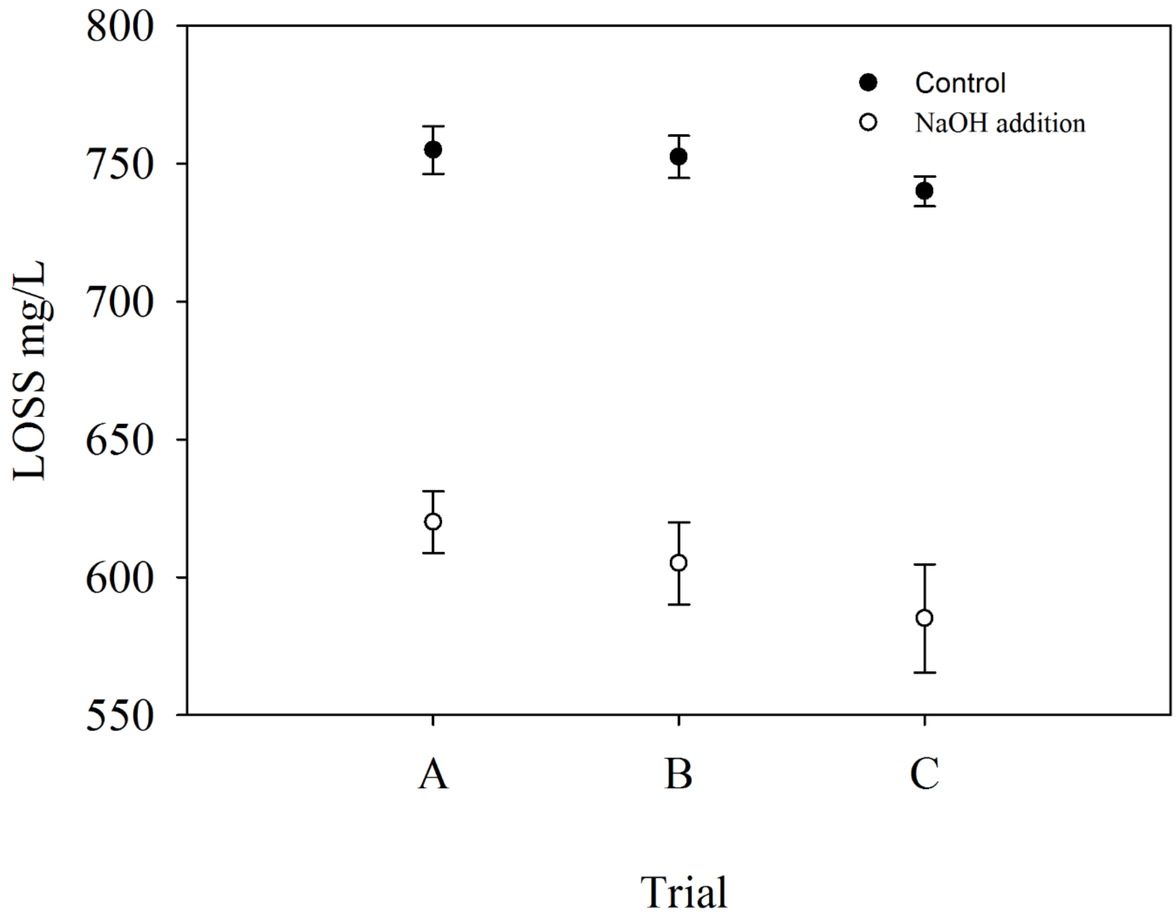

Figure 3.3: LOSS for Secondary mixed liquor with addition of 420 mg NaOH/L for 3 time points (triplicate measurements).

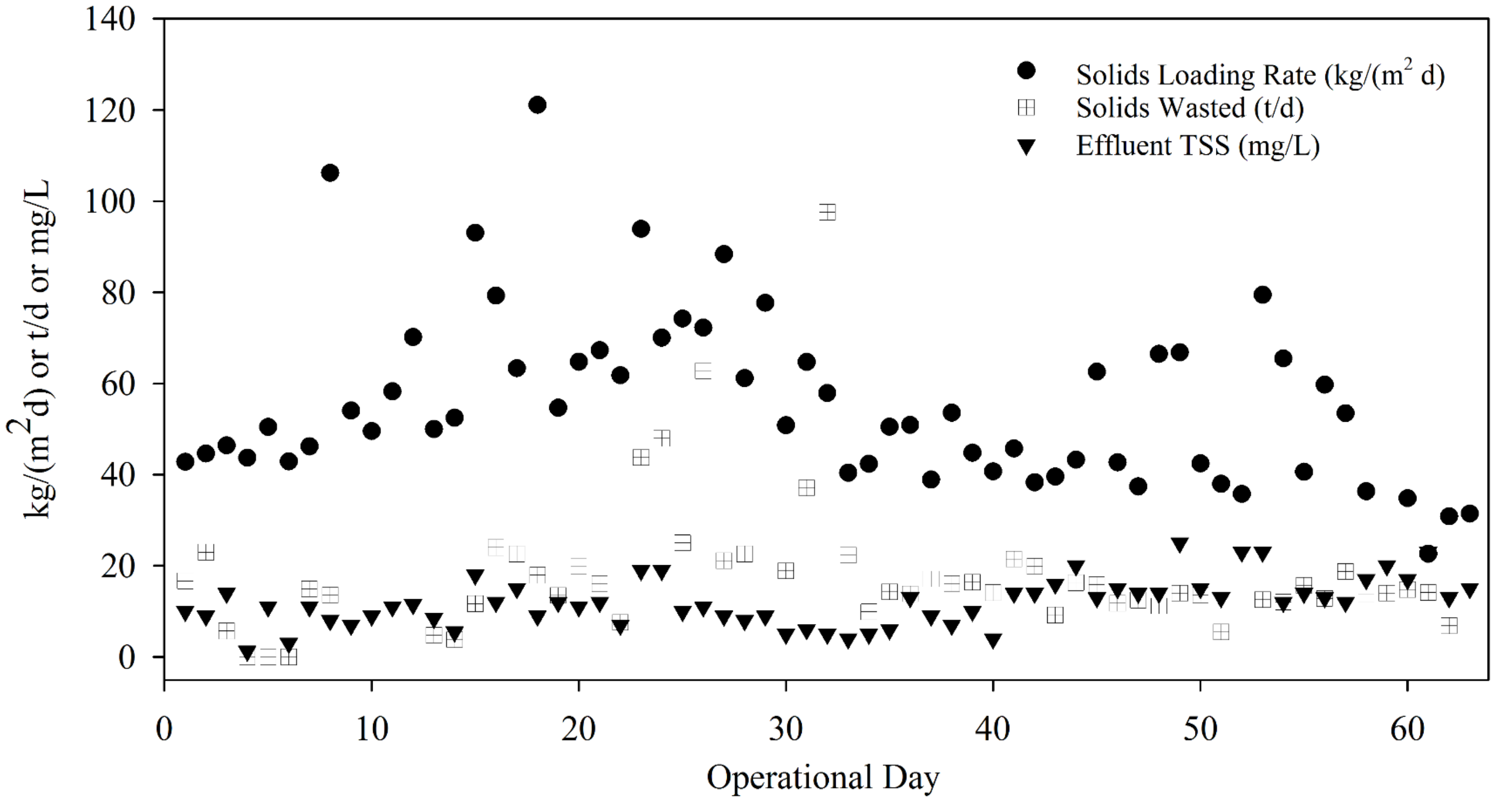

Figure 3.4: Blue Plains AWWTP secondary clarifier operational data plotting solids loading, solids wasting, and effluent TSS over 60 days of settling characteristics observations.

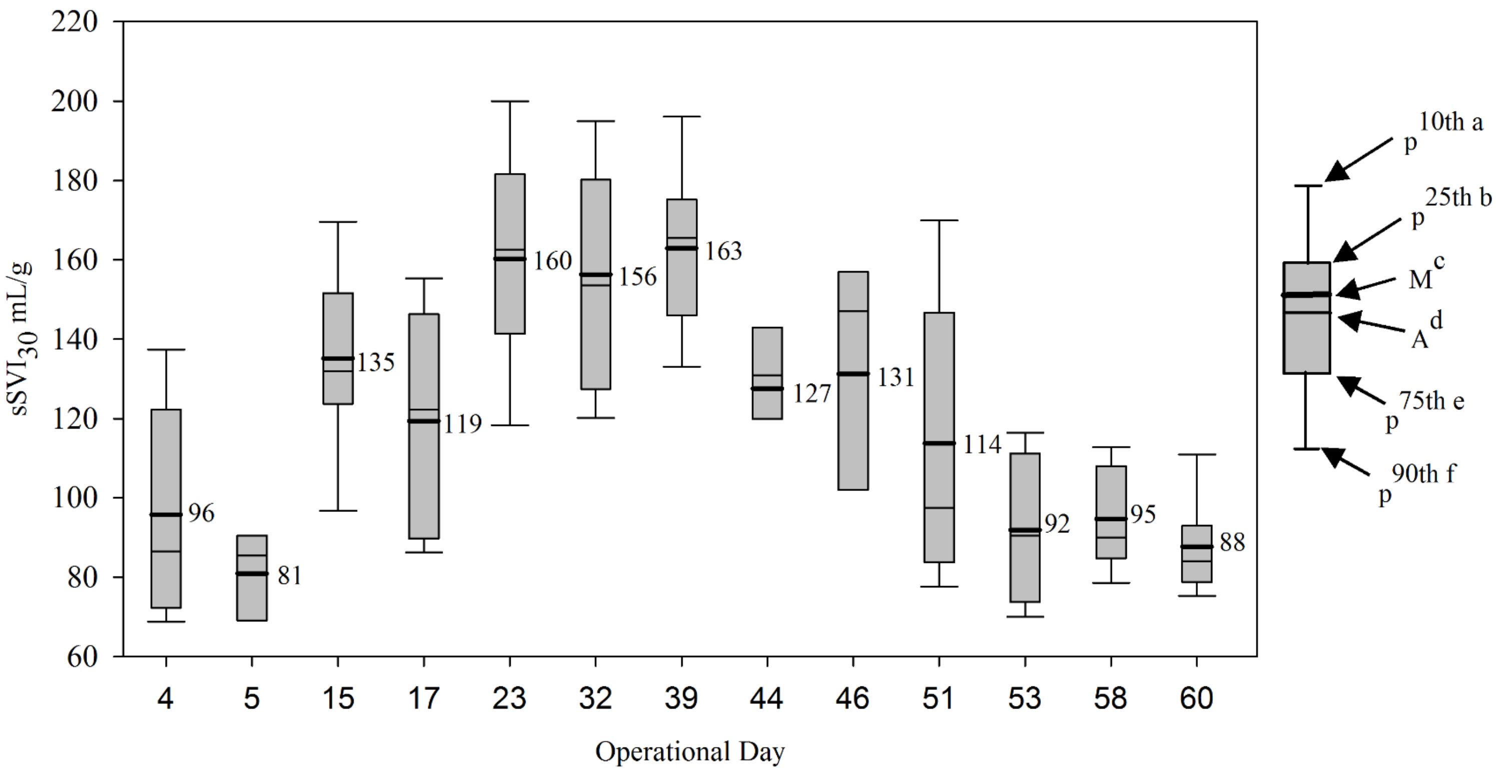

Figure 3.5: Box plot graph representation of sSVI30 data over 60 days of observation, the distribution is of the 5 different batches of MLSS ranging from 900 mg/L to 2000 mg/L used to generate the gravity flux curves. (a) is the 10th percentile, (b) is the 25th percentile, (c) is the median, (d) is the average, € is the 75th percentile, and (f) is the 90th percentile.

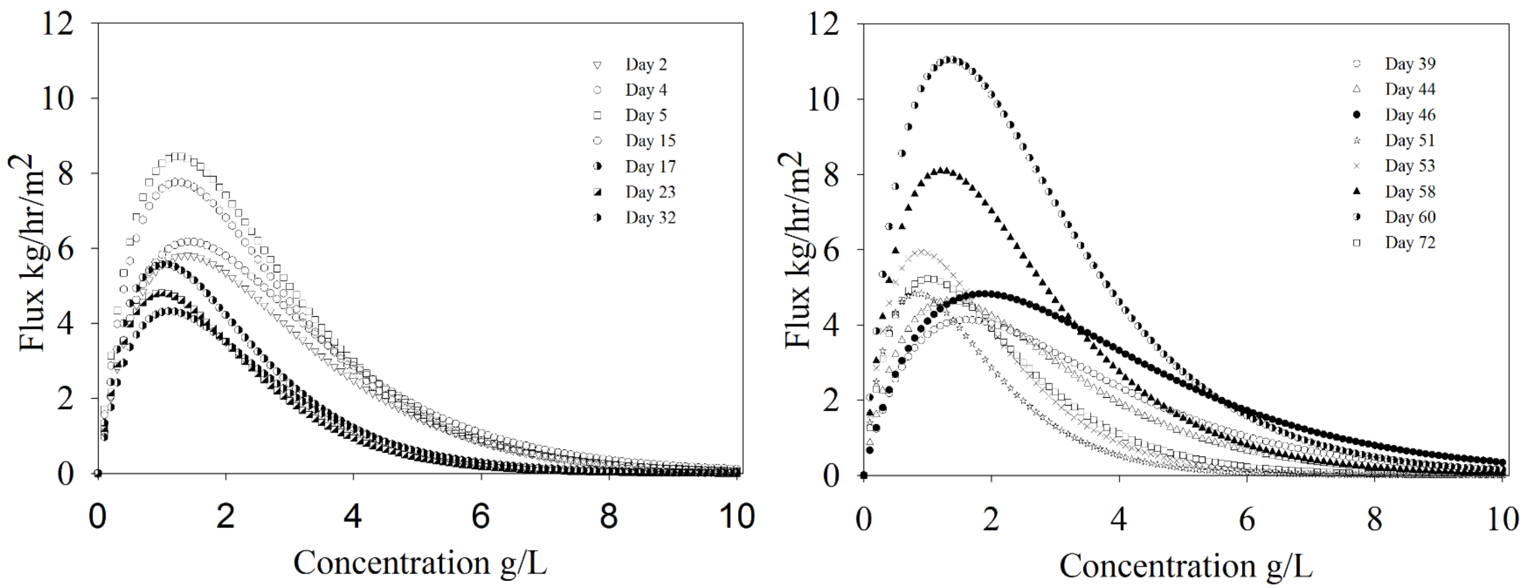

Figure 3.6: Gravity flux curves from 15 column testing trials conducted over a 60 day period. (A) shows the first 32 days, while (B) shows the remainder.

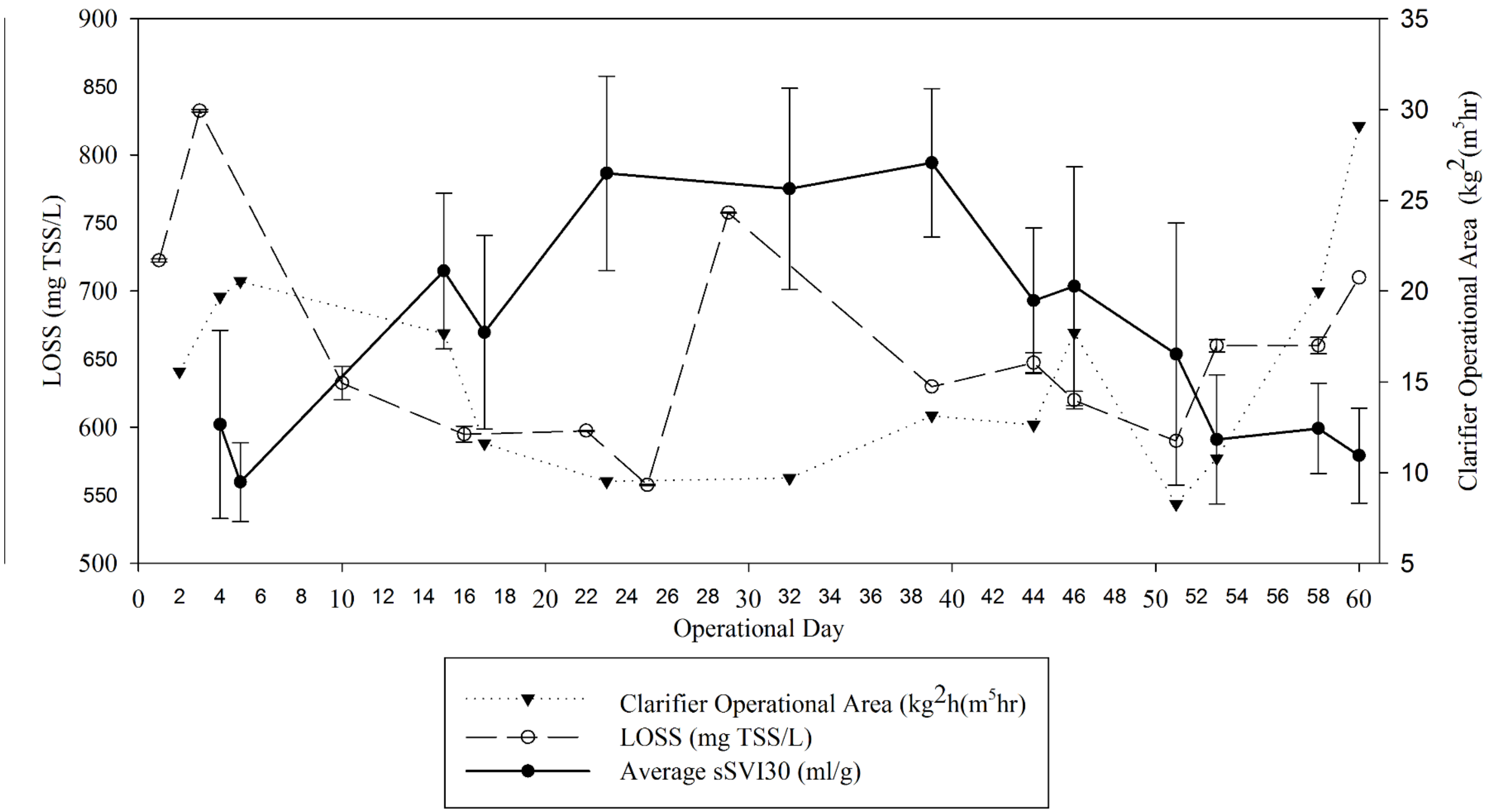

Figure 3.7: Correlations of sVI30, LOSS, and clarifier operational area (extrapolated from the flux curves) covering 60 days of observations.

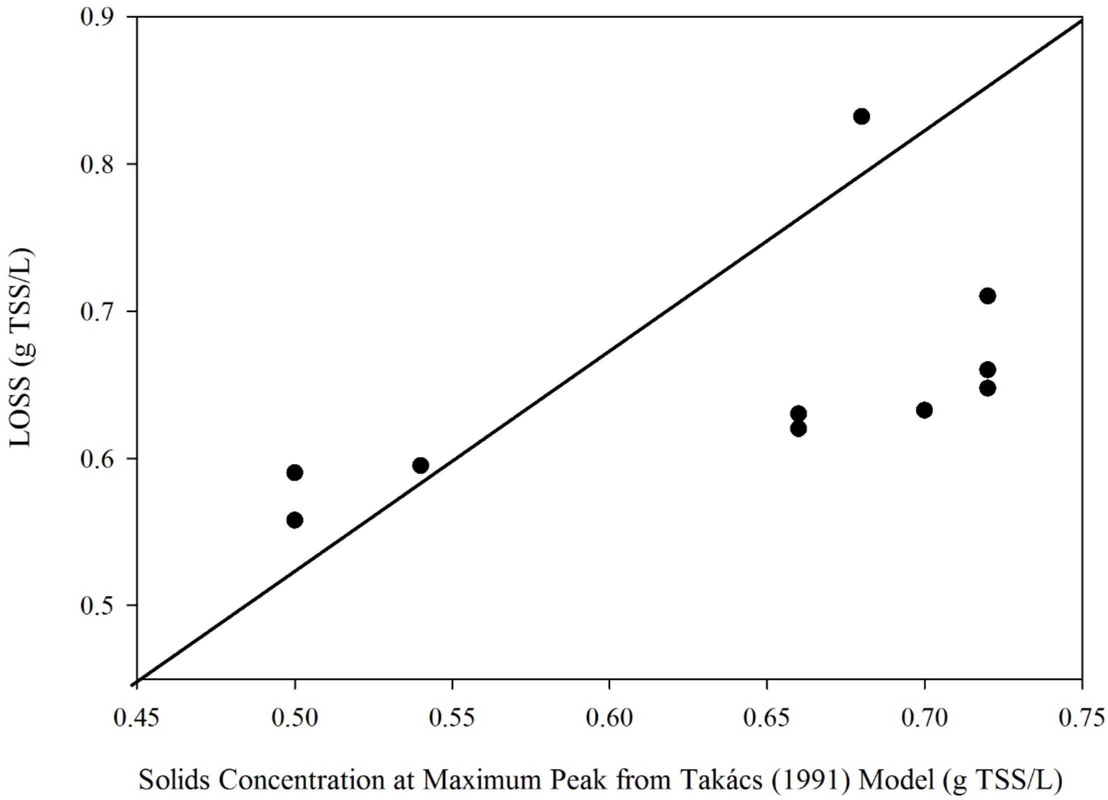

Figure 3.8: Takacs et al., (1991) model prediction of LOSS (maximum peak) versus experimentally measured LOSS.

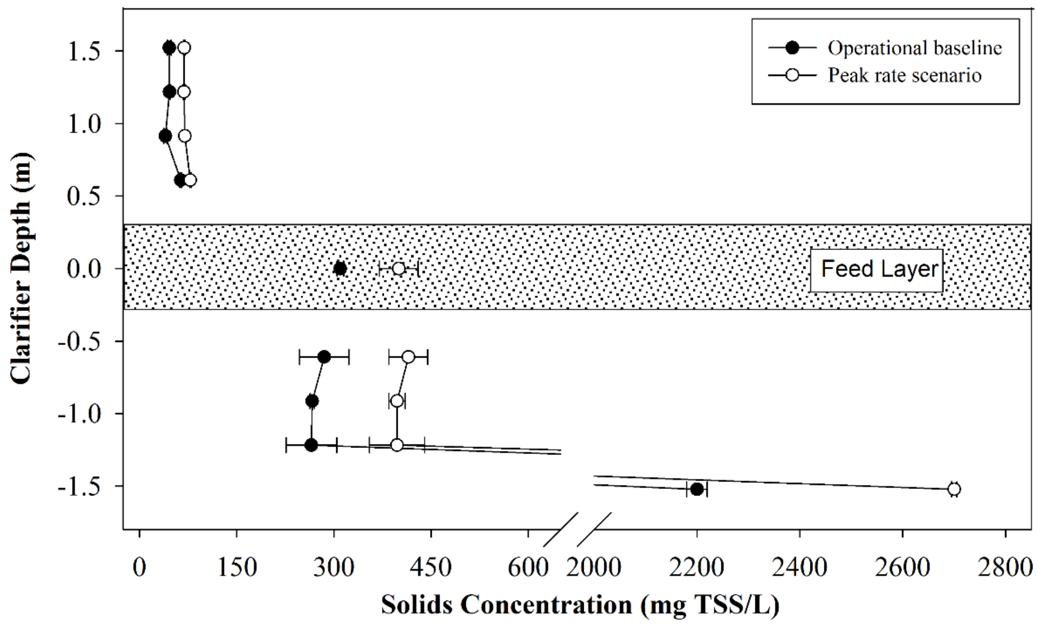

Figure 3.9: Average solids concentration and depth profile based on two collections, 1 HRT apart, for current operational and peak rate conditions in the pilot clarifier at Blue Plains AWWTP. Feed layer (shaded area) is designated a depth of 0.5 to -0.5 m. Top and Bottom layer are designated 0.3 to 1.3 m and -1.3 to -0.3 m, respectively. Effluent and underflow are at 1.5 m and -1.5 m, respectively.

CHAPTER FOUR

NOVEL STOKESIAN METRICS THAT QUANTIFY COLLISION EFFICIENCY, FLOC STRENGTH, AND DISCRETE SETTLING BEHAVIOUR

4.1 AUTHORS

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Submitted to Water Environmental Research

4.2 ABSTRACT

Novel parameters were developed to predict the effluent quality and settling behavior in clarifiers that cannot conventionally be achieved using either the conventional flux theory or overflow rates. Simple batch experiments based on the critical settling velocity (CSV) selection were used as the basis for the development of three novel parameters: intrinsic settling classes (ISC), threshold of flocculation/flocculation limitation (TOF/a), and floc strength. ISC was proven to accurately (±2%) determine the granule fraction and discrete particle distribution. TOF quantified the minimum solids concentration needed to form large flocs and was directly linked to collision efficiency. In hybrid systems, an exponential fitting on a CSV matrix was proposed to quantify the collision efficiency of flocs (a). Shear studies were conducted to quantify floc strength. The methods were applied to a wide spectrum of sludge types to show the broad applicability and sensitivity of the novel methods.

4.3 KEYWORDS

Flocculation, Activated sludge, Settling regimens, Granulation, Critical settling velocity

4.4 NOMENCLATURE

BNR	Biological nutrient removal
CAS	Conventional activated sludge
CSV	Critical settlings velocity
EPS	Extracellular polymeric substances
ISC	Intrinsic settling classes
ISV	Initial settling velocity
LOSS	Limit of stokesian settling
SOR	Surface overflow rate
SRT	Solids retention time
$sSVI_{30}$	Stirred sludge volume index at thirty minutes
SVI	Sludge volume index
t_F	Batch flocculation time
TOF	Threshold of flocculation
t_S	Batch settling time
TSS	Total suspended solids
t_T	Total batch experiment time
α	Collision efficiency

4.5 INTRODUCTION

The design of secondary settling tanks has typically been conducted using the flux theory (Ekama et al., 1997). This theory assumes that flocculated solids settle as a blanket, leaving a clarified overflow layer above. According to this theory, the overflow liquid (secondary effluent) is either of low solids, or the blanket rises and overflows, resulting in a high solids overflow. In reality, the effluent solids can vary over a range of solids concentrations due to changes in floc characteristics and operation of the activated sludge system. In particular, there are situations where the mixed liquor fed to the clarifier does not form a blanket due to either the characteristics of the solids or a feed concentration below that needed to form a blanket and clarifier performance is dominated by stokesian settling behavior (Mancell-Egala et al., 2016).

Solids separation can be predicted and simulated with or without the application of Stokes law, here after named as stokesian or non-stokesian settling, respectively. Stokesian settling includes two phases of settling, i.e. discrete and flocculant, while the non-stokesian settling includes hindered settling and compression (Ekama et al., 1997). Current conventional settling characterization methods such as sludge volume index (SVI) are good qualitative indicators of bad versus good settling but do not provide quantification of the settling properties. This is due to SVI being largely centered on compression settling; it is a crude indicator of the settling properties and cannot be linked with effluent quality. Alternatively, state point analyses generated from initial settling velocities (ISV) are based on hindered settling, which has been shown to be fraught with artifacts, operator error issues, and unreliability (Kinnear, 2002). These measurements have been unsuccessfully incorporated in computation fluid dynamic models for determining the capacity of secondary clarifiers (Ekama et al., 1997). The shortcomings of current methodologies lie in their focus on non-stokesian settling models to characterize the settling phenomena occurring in a clarifier, while this settling behavior only becomes relevant when failure occurs. A need for a more direct measurement related to stokesian settling properties is necessary to improve the current state of practice.

It is important to understand both discrete as well as flocculant settling behavior, as both follow Stokes' law. Conventional activated sludge (CAS) systems rely on bio-flocculation to maintain solids separation by creating large flocs that settle out faster than the individual particles. Therefore, flocculation kinetics are most important for CAS systems. In contrast, fully granular systems, as described by Kreuk et al. 2007, do not need bio-flocculation to occur in a low hydrodynamic shear environment to settle out fast and be maintained in the system. Therefore, discrete settling is the main settling mechanism determining solids capture. One can of course envision that in several cases hybrid systems (flocs + granules) will occur, making the combined effect of discrete settling and flocculation kinetics responsible for the effluent quality in clarifiers. In general granular sludge is described by conventional settling parameters such as sludge types with $SVI_5 \approx SVI_{30}$ and SVI_{30} smaller than 70 ml g TSS^{-1} (Winkler et al., 2013). Increasing the granule fraction in a system is possible through selection for intrinsic particle that settle faster than 10 m h^{-1} (Liu et al 2005). No method has been proposed for quantifying a discrete settling velocity distribution that could be applied for both flocculant, granular, and hybrid systems. This study tries to fill this gap.

In addition to characterizing discrete settling, no easy and practical tools have been developed to characterize flocculant settling behavior, is a second goal of this study. Flocculation is dependent on two functions: collision efficiency and the number of collisions (Serra et al., 1997). Collision efficiency results in the successful attachment of two flocs through collision, while the number of collisions is directly related to the solids concentration and shear. According to current solid flux theory based on the Kynch (1952) assumption and bio-flocculation studies (Serra et al., 1997), there should be a solids concentration linked to the minimum collision rate needed to form larger aggregates. The transition concentration from discrete settling to flocculant settling should be directly related to collision efficiency. This paper aims to develop a parameter that characterizes collision efficiency and therefore, the flocculation property.

In addition, floc strength is an important characteristic of a floc as both formation and breakup determine solid capture in clarifiers. Flocculation can be enhanced with the addition of energy (shear), commonly known as ortho-kinetic flocculation. Sludge experiences a transport shear within channels from reactors to clarifiers. Popular belief is that flocculation happens within feed channels, and that flocs are already formed before introduction to the clarifier (Bratby et al., 2006). This study's final goal was to characterize floc strength by quantifying floc size formation at varying shears.

Overall, in this research several new activated sludge parameters; (i) discrete settling velocity distribution, (ii) collision efficiency and (iii) floc strength, are used to characterize the flocculation and settling characteristics of a broad spectrum of sludge types (activated sludge, granular sludge and hybrid systems). These parameters allow prediction of the effluent quality and settling behavior that cannot be achieved using either the conventional flux theory or overflow rate to describe secondary clarification.

4.6 MATERIAL AND METHODS

4.6.1 Sludge Types

Five systems were sampled and their characteristics are summarized in Table 4.1. (i) Secondary sludge was obtained from the west secondary treatment full-scale reactors from the Blue Plains Advanced Waste-water Treatment Plant (Washington, DC, USA). The secondary treatment was a high-rate activated sludge system (1-2 days of sludge retention time) focused on the removal of organics. (ii) Biological -Nitrogen Removal (BNR) sludge was obtained from the Blue Plains tertiary treatment process operated at an aerobic SRT of 10-15 days which received methanol as external carbon source. This flocculant system has shown good settling properties over the years, resulting in reliable effluent quality (Table 4.1). (iii) Mainstream sludge was obtained from a mainstream deammonification pilot plant at Blue Plains (Al-Omari et al., 2015). This pilot simulated the BNR system at Blue Plains with the difference that an aggressive aerobic SRT (1-5 days) was used under transient anoxia and anammox granules were bio-augmented. Therefore this system can be seen as a hybrid system with a small fraction of anoxic ammonium-oxidizing granules. (iv) Sidestream sludge was obtained from a sidestream deammonification pilot at Blue Plains (Zhang et al., 2016). The Sidestream pilot plant was a sequencing batch reactor, which was treating centrate from the Alexandria Sanitation Authority. The reactor was operated at a SRT of 30 days to allow retention of anoxic ammonium-oxidizing bacteria and was characterized by a large fraction of granules with only a small fraction of flocs. (v) Seed sludge was a granular sludge provided by the Strass wastewater treatment plant, and was collected from the DEMON® cyclone’s underflow. The DEMON® cyclones are physical separation devices that select sludge based on particulate density (Wett et al. 2010). This sludge was used as a granular example although artificially obtained by the use of cyclone and thus not directly related to an operational system.

4.6.2 Conventional Settling Parameters

A settling column was fabricated in accordance with the Clarifier Research Technical Committee of the American Society of Civil Engineers and was identical to that used by Wahlberg (1995). ISV and Stirred Sludge Volume Index at 3.5g ($sSVI_{30}$) procedures were conducted in this column to obtain a solids flux curve. Both $sSVI_{30}$ tests and Total Suspended Solids (TSS) determination followed protocols listed in standard methods (APHA et al., 2005). The Limit of Stokesian Settling

(LOSS) methodology followed the protocol described in Mancell-Egala et al., (2016). LOSS characterizes hindered settling velocity by identifying the transitional solids concentration whereby flocculant (stokesian) settling transitions to hindered (non-stokesian) settling).

4.6.3 Setup for Novel Settling Parameter Determination (CSV Test)

A settling cylinder was fabricated after a design used by Perric et al., (2008). The device was constructed from a modified 4 L graduated cylinder acquired from Thermo Scientific™ Nalgene™. The cylinder had two sample ports located opposite to each other at a distance of 5 cm from the water surface (supplementary Figure B.1). A well-mixed (i.e. hand mixed) batch of activated sludge at a known solids concentration sludge was poured into the settling column with the aid of a funnel. The funnel was necessary to ensure unified flow into the center of the column to eliminate vortexes. After the sample was poured, the funnel was removed and a timer was started. When the required time had elapsed the sample ports were opened and the entire portion above the sample ports was removed. This is referred to as the supernatant (shaded area in supplementary Figure B.1). The supernatant then underwent TSS determination. A normalized percentage of TSS for corresponding batches and supernatant was calculated. The required batch settling time was determined by equation 1. The above method will be referred to in general as a critical settling velocity test or CSV test. This test was either performed at different CSVs at one initial TSS concentration, or at different initial TSS concentrations at one CSV, or a combination of both.

$$Critical\ Settling\ Velocity\ = \frac{Distance\ travelled}{time} = \frac{0.05m}{time}\ \dots\ \ \dots\ (Eq.1)$$

4.6.3.1 Intrinsic Settling Classes (ISC) Determination

One biomass mixture of 20 liters was collected for each type of sludge at a solids concentration that represented the discrete settling range (concentration lower than TOF). The biomass mixture had to have a TSS concentration below the TOF value to ensure discrete settling. The following critical settling velocities were applied to classify the intrinsic particles: 0.6, 1.5, 3, 9 m h^{-1}. A total of five batches were made with a known granular percentage (using Seed sludge) by weight of 0%, 10%, 20%, 40%, and 100% to evaluate the sensitivity of this test.

4.6.3.2 Threshold of Flocculation (TOF) Determination

Serial TSS dilutions were made ranging from as low as 50 mg TSS L^{-1} to as high as 10 g TSS L^{-1} for six different sludge types. Each TSS batch underwent a CSV test at 1.5 m h^{-1} and the normalized TSS in the supernatant (%) was determined. TOF was quantified as the deflection point (min, differential of 10%) of the curve plotting the normalized supernatant TSS vs the initial TSS concentration (Figure 4.1).

4.6.3.3 Shear Matrix

The Secondary and BNR sludge types listed in Table 4.1 underwent shear experiments. Shear experiments were split into two parts; (i) impact of shear on classes of floc formed and (ii) impact of shear at varied solids concentration. Shear experiments included additional steps to the standard CSV test described previously. After samples were introduced into the settling column, an IKA® EUROSTAR 200 control mixer with twin propeller was placed inside the settling column to provide a specific shear for ten minutes (supplementary Figure B.1). After the allotted time, the mixer was removed and the remaining steps of the CSV test were followed. Similar to Furukawa et al., (2012), the power consumption calculation was based on physical parameters of the wastewater matrix, geometry of propellers, geometry of settling column, and number of propellers. Shear from the mixer was calculated from the power consumption (Nopens, 2002). After exposure to shear, each batch underwent CSV class measurements depending on the shear experiment.

For the first part, a 30 L biomass mixture was prepared at 20% above the TOF value. This solids concentration was chosen so that the sludge matrix was not limited by the solids concentration inducing collisions for the minimum time period. Also the solids concentration was low enough to not result in a high collision number that would diminish any collision efficiency effects. Six 4 L batches were taken from the 30 L biomass mixture, and underwent specific shears at G; 20, 30, 40, 60, and 90 s^{-1}. At each shear, samples were collected at three CSV's; 0.6, 1.5, and 3 m h^{-1}.

The second part of the shear experiment entailed creating 15 L serial TSS dilutions that ranged from 50 mg TSS L^{-1} to 1000 mg TSS L^{-1}. Each activated sludge mixture underwent four different shears. They were commenced at low ($20s^{-1}$), medium ($30s^{-1}$), high ($60s^{-1}$) and very high ($90s^{-1}$) shear rates. At each shear a sample was collected at a CSV of 1.5 m h^{-1}.

4.7 RESULTS AND DISCUSSION

4.7.1 Conventional Settling Characterization

The $sSVI_{30}$, ISV, and LOSS values for sludge systems are listed in Table 4.1. Secondary sludge settling performance was poorest with a higher $sSVI_{30}$ and lower ISV values. The results from LOSS concentration corresponded with $sSVI_{30}$ and ISV. The BNR LOSS concentration was 200 and 400 mg TSS L^{-1} higher than that of good and poor performance Secondary sludge, respectively, confirming that BNR compared to Secondary had a larger solids fraction at high settling velocity (Table 4.2). The latter correlated well with the ISV values of these activated sludge types. LOSS was shown to be a more accurate tool when compared to ISV and $sSVI_{30}$ when characterizing hindered settling rates (Mancell-Egala et al., 2016). The Seed had the best settling characteristics with superior $sSVI_{30}$, ISV, and LOSS when compared to the other 5 sludge types. BNR and Sidestream sludge types, had comparable $sSVI_{30}$, however, the latter displayed a higher ISV and LOSS. This indicates that BNR settles slower but compresses more than Sidestream sludge. This is also indicative of Mainstream sludge as well. Mainstream sludge hindered profile settles 0.4 m h^{-1} faster than BNR sludge, but has a higher $sSVI_{30}$ recorded at 121 mL g^{-1} TSS compared to 88 mL g^{-1} TSS. Furthermore, only Secondary poor performance had an $sSVI_{30}$ higher than the Mainstream sludge. Without the contradictory values from ISV and LOSS, the Mainstream sludge would be depicted as preforming subpar to almost all other sludge types tested.

4.7.2 Intrinsic Characteristics

The methodology of classifying sludge characteristics over a discrete settling range was first evaluated using artificial mixtures of a fully granular sludge (Seed sludge) and fully flocculant sludge (Secondary sludge). Artificial granular fractions of 0, 10, 20, 40 and 100% on a mass basis were prepared. The measurements on those mixtures are shown in Figure 4.2A. It can be seen that the ISC determination was accurate within 2% of the known weighted fraction, confirming the vast differences between granules and flocs. The 100% flocculant and 100% granular sludge were completely different in this test. The artificial batches fell between the two extremes and were ranked according to their percentage of each extreme.

Figure 4.2B shows that for CAS, a majority of solids (>92%) were unable to settle at a CSV of 1.5 m h^{-1}, and required flocculation or coagulation to settle. The latter is in accordance with the accepted definition of CAS as always needing flocculation to settle (Ekama et al., 1997). Furthermore, the ISC was able to distinguish differences in CSV fractions between flocculant systems, in this case BNR and Secondary sludge. The BNR sludge had a significantly larger fraction (~10%) that settled out above 0.6 m h^{-1} compared to the Secondary sludge. Additionally, the ISC results for hybrid systems was in accordance with the operational definitions of each system. The Seed sludge had the largest combined fraction that settled above 1.5 m h^{-1} (96%). This was in agreement with the literature Wett et al., (2010) confirmed that a cyclone selects a sludge fraction based on density, therefore, the Seed sludge would have a higher density and thus require little to no bio-flocculation or coagulation to settle out (de Kreuk et al., 2004).

Similarities can be seen between the hybrid sludge types and artificial mixtures on a macro level (Figure 4.2A & B). Both the Mainstream and 20% artificial sludge mixture had a similar weighted fraction at a CSV of 1.5 m h^{-1}. However, on a micro-scale, fractions between CSV of 0.6 – 1.5 m h^{-1} and 3 – 9 m h^{-1} show that the sludge types are dissimilar. The same can be observed for Sidestream sludge and the 40% artificial mixture. The ability to see changes in narrower CSV classes could be an excellent method to monitor physical selection strategies intended to increase the density of particulates in a system. The ISC indicates the fraction of particulates that need either flocculation or coagulation, and thus can be used to diagnose the cause of poor effluent quality in hybrid systems. Poor effluent quality can be caused not only by poor flocculation but by poor coagulation (Bratby, 2006). The effluent quality of highly granular sludge types has been shown to be a drawback (Wan et al., 2011) and sand filters or other post treatment steps may be needed to achieve low effluent TSS concentrations. In systems with a lower granular fraction, the latter coagulation limitation can be potentially resolved by more extensive flocculation.

4.7.3 Collision Efficiency Indicator: Threshold of Flocculation (TOF)

In Figure 4.3 the solids that remain in the upper layer of a volume of activated sludge that is allowed to settle in a container over a range of initial solids concentrations is shown. For the data in Figure 4.3, a settling time of 2 minutes was used and the choice of 2 minutes is explained below. The topmost layer is referred to as the supernatant layer and the solids that remain in the supernatant layer are a function of the flocculation properties, time of settling, and the initial solids concentration of the activated sludge. Activated sludge is comprised of solids/particulates with a density close to water and flocculation enables these solids to increase their mass. For solids to leave the supernatant layer they must first flocculate and then settle out. Figure 4.3 expresses this mechanism in reference to the total time activated sludge is left to settle out in a container (t_T). As mentioned previously, the time it takes solids to form a floc (t_F) is dependent on the collision efficiency and collision number (which is defined as the probability two flocs will collide). As the initial solids concentration of the activated sludge is decreased, the collision number decreases and the t_F increases. As t_F increases it reaches a threshold whereby the time it takes the flocs formed to escape the supernatant layer (t_S) is no longer possible for a given t_T ($t_T < t_F + t_S$). After this threshold is met there should be little to no settling and the percentage of solids in the supernatant should no longer vary as the initial solids concentration is decreased.

The minimum solids concentration at which significant settling could be measured (10% change), was defined as the Threshold of Flocculation (TOF) and characterizes the collision efficiency of the sludge. The larger the TOF, the more collisions need to take place for a significant floc size (or settling velocity) to be achieved under the batch settling time proposed. The batch settling time for the data shown in Figure 4.3 was set at 2 min which was directly linked to a critical settling velocity of 1.5 m h^{-1}. The latter was chosen based on the study of Daigger (1995) which determined 1.7 m h^{-1} to be the surface overflow rate (SOR) at which clarifier failure occurs. The slightly lower critical settling velocity used in this study allowed for a safety factor of 15% to account for measurement variability. TOF was thus quantified by the deflection point of the S-curve shown in supplementary Figure 4.3 with a minimum differential of 10% for a critical settling velocity of 1.5 m h^{-1}, representing flocs that would normally be retained in conventional clarifiers. Additionally, no shear or mixing was applied during this test to avoid ortho-kinetic flocculation. Gravitational flocculation is weaker than ortho-kinetic flocculation so that the number of collisions is not artificially increased at lower solids concentrations, so the collision efficiency is characterized only by the sludge properties. Therefore, the TOF was hypothesized to occur at lower concentrations only for systems with sludge properties that allow for high collision efficiency.

4.7.4 Collision Efficiency: Threshold of Flocculation (TOF)

High rate activated sludge systems have been shown to have a higher effluent TSS concentration than medium and low rate, most probably due to coagulation and flocculation limitations (Liao et al., 2006). According to Liao high-rate systems suffer from deteriorated effluent quality due to an unstable microbial community, resulting in poor biomass flocculation and separation. This is in contrast to long SRT sludge types, which fully hydrolyze influent particulates while producing more EPS, creating more potential for sweeping smaller lighter particulates into the flocs. Limitations are therefore moved more towards flocculation and settling. It was hypothesized that TOF could capture the difference in collision efficiency of sludge types, thereby distinguishing coagulation/flocculation limitation. Indeed, good and poor performance Secondary sludge types hade TOF values 1.6 and 2.8 times higher than BNR sludge, respectively. The latter confirmed that high rate sludge suffers from greater coagulation/flocculation limitations than low rate sludge, and that TOF can capture this. TOF results are also in agreement with effluent quality from operational data and conventional metrics such as $sSVI_{30}$ and ISV (Table 4.1).

For hybrid systems, TOF was found to be lower. A TOF of 155 mg TSS L^{-1} was observed for the Mainstream deammonification pilot sludge (Figure 4.4B). However, no TOF could be detected for the Sidestream deammonification and Seed sludge types. Even at a low TSS concentration (50 mg TSS L^{-1}), significant TSS capture was achieved (Figure 4.4B). The lack of a defined TOF for systems with a high granule fraction can be explained by the fact that granules do not need bio-flocculation to settle (de Kreuk et al., 2004) and therefore significantly decrease the supernatant TSS in the test, even at very low TSS concentrations. The floc fraction, however, will need to reach a certain threshold concentration before it can settle and the flocculation behavior will determine effluent quality. Although the TOF value can still provide an indication of the minimum MLSS needed to be fed to the clarifier to have significant settling, it does not give significant information about the flocculation affinity (collision efficiency) when the floc fraction is low. Therefore, more in-depth analysis focusing on a complete matrix of TSS concentrations and CSV was performed (see Figure 4.5) to find an alternative parameter that can describe collision efficiency even when a significant fraction of granules is present. From Figure 4.5, it can be seen that when looking at the 1.5 m h^{-1} CSV curve, steeper transitions into faster settling classes were obtained when moving from poor settling Secondary to good settling Secondary sludge and further into BNR sludge (Figure 4.5A - C). Especially for the poor settling Secondary sludge, a longer lag time in transition to faster settling classes was observed, indicating that the collision efficiency was lower. Moreover, for the flocculant systems, BNR sludge formed faster settling flocs (> 3 m

h^{-1}) compared to the Secondary system (Figure 4.5A - C). For the hybrid systems (Figure 4.5E, F), this full matrix analysis allowed one to observe that Sidestream sludge formed fast settling flocs compared to the Mainstream sludge. However, the rate of change, when 1.5 m h^{-1} was again used as benchmark, was smaller for the Sidestream sludge compared to Mainstream sludge.

As this matrix test highlighted the dynamics of flocculation more closely, an alternative quantification for flocculation affinity (collision efficiency) was proposed that could potentially eliminate the interference of the granular fraction. For the systems with granules, there seems to be a trend between slopes of the lines and the quantity of granules (Figure 4.5D - F). For each CSV applied, the solids removal rate was calculated as the maximum slope of each curve is presented in Figure 4.4. Subsequently the calculated solids removal rates were plotted against their respective CSV (Figure 4.5A - F) and a good exponential curve fit (R^2 above 95%) was found. The parameters of the latter exponential fit are shown in Table 4.2. The curvature of the exponent was governed by the α value, with high α resulting in a high curvature, which corresponded to sludge types with flocculation limitation and thus longer flocculation time. BNR systems had the lowest α value (0.805) in contrast to Secondary sludge such that a Poor Performance was representation of a poorly flocculating system (α values of 1.575, see Table 4.2). This also correlated well with the previously described TOF values (Table 4.2). Moreover, the exponential curve fitting also allowed for a differentiation in flocculation behavior in hybrid and granular systems (Table 4.2). Granular sludge had the lowest α value of 0.07, indicating it has no flocculation limitation, but could still suffer from coagulation limitation. Wan et al., (2011) reported that there are always suspended solids in the supernatant for granular sludge systems and that further clarification steps are needed to provide an acceptable effluent quality. A hybrid system encompasses the strength of both granular and flocculant systems without their weaknesses, as it provides sweeping flocs to remove suspended solids, and granules to provide ballasting, resulting in faster settling velocities. Unlike TOF that was affected by discrete settling properties (granular content), the α value is a direct quantification of flocculation limitation and shows how the collision number (solids concentration) relates to floc formation (CSV fractions). However, from a practical standpoint, the k-value determination was labor intensive and might only be worth it for hybrid systems as the conclusions based on TOF for flocculant systems remained the same (Table 4.2). Overall the methods (both TOF and k) were able to quantify differences in collision efficiency. It should be noted, however, that the CSV method, used as the basis for all parameters is not sensitive enough to allow for the parameters to be used to differentiate between coagulation and flocculation limitation and only describes the combined effect. Combining these collision efficiency parameters with process data such as effluent quality might allow for more detailed understanding of the underlying limitation. This will be investigated in future studies.

4.7.5 Floc Strength

There is a clear distinction between operational shear and transport shear. Operational shear is the shear present in the reactor and this shear influences the sludge matrix, i.e. higher shear stresses the microbiological community thereby increasing Extracellular Polymeric Substances (EPS) production (Spicer, 1998). Transport shear impacts flocs formed by the sludge matrix. Floc strength of sludge revolves around the hypothesis that flocculation occurs in the feed channel. If that is the case predicting transport shear's impact on flocculation is paramount in estimating floc strength and clarifier performance. Wastewater treatment plants are designed to minimize shear forces that lead to floc breakage. However, in reality that is not always the case, with regions of high shear present in reactors and channels (McCurdy et al., 2004). This may include regions around weirs, ledges and baffles. Flocs must resist these high shear points and avoid breaking into smaller particulates before entry into a clarifier. This is an important operational condition, as smaller particles of the same density generally settler slower and have lower removal rates (Boller and Blaser, 1998). According to Serra et al., (1997) increasing the shear gradient increased collisions, but decreases the efficiency of collisions. Therefore, by characterizing shear it should be possible to better characterize collision efficiency and define a new property (floc strength), by measuring its variance with shear. Using shear to characterize floc strength is a variation of a method that has been used in several studies (Leentvaar and Rebhun, 1983; Francois, 1987; Fitzpatrick et al., 2003). The relationship of floc strength with TOF was investigated in this study.

4.7.6 Floc Strength: Shear Studies

Figure 4.6A shows the impact of different shear conditions during a 10 minute ortho-kinetic flocculation test on the formation of different floc settling fractions for Secondary sludge under good performance and at 500 mg TSS L-1 (20% higher than TOF). A significant effect of shear was observed for this sludge type, with larger flocs being formed under low shear conditions and breakup of flocs under high shear conditions (Figure 4.6A). This was in accordance with the literature (Serra et al., 1997). When looking at the CSV cut-off of 1.5 m h^{-1}, the floc strength could be investigated in detail by applying different shear conditions under a range of sludge concentrations (Figure 4.6B - D) and observing the changes at that CSV class at different shears. For the Secondary sludge at good performance, a high impact of shear is seen in Figure 4.6A, as indicated by a wide spread of the curves (Figure 4.6B) and thus larger difference in supernatant TSS. In contrast, both the BNR sludge as well as the Secondary sludge at poor performance had only a minimal impact of shear on the flocculation efficiency, with an 18±5 and 16 ± 5% supernatant TSS change, respectively (Figure 4.6C-D). In terms of floc strength, the BNR sludge and the Secondary sludge at poor performance were thus example of stronger flocs although the collision efficiency (see 4.7.4) was different. This is a demonstration that collision efficiency and floc strength need to be considered as two separate features and do not always correlate with each other.

Studies on floc strength elucidated a notable trend; smaller flocs are stronger (Jarvis et al., 2005). A mechanistic relationship for this trend has not been discovered. However, common belief is that floc compaction and internal bonds are the factors influencing floc strength (Jarvis et al., 2005). It is widely accepted that flocs are examples of fractal structures (Bushell et al., 2002). Fractional dimension analysis has shown flocs to break of at their narrowest or weakest point, resulting in smaller more compact structures. Fractional dimension analysis can indicate the compactness of a floc's structure, and flocs with more compact structures are stronger (Spicer et al., 1998). Additionally, Spicer et al., (1998) found that flocs in high shear environments were more compact. Furthermore, SRT has been linked to compactness and stability of floc structure (Liao et., 2006). Long SRT systems (low rate) produce more stable compact flocs, whereas short SRT systems (high rate) have flocs that are less stable and irregularly shaped.

Similarly, collision efficiency is an important factor in determining floc structure and depends on surface properties, hydrodynamic effects, and interaction forces within particulates (Jeldres et al., 2015). Collision efficiency is a complex function, which makes determination of it difficult. Some studies have used an adjustable parameter (Biggs et al., 2002), or a constant value (Zhang et al., 2003). More mechanistic tools involve mathematical models developed heuristically; they integrate the physical characteristics of the process and allow for a better understanding of a system. Trajectory analysis (Adler et al., 1981) has been used successfully to develop collision efficiency models. Collision efficiency dependence on shear rate has also been determined an increase in shear decreases collision efficiency (Serra et al., 1997). However, Aunins and Wang (1990) also found that increasing the concentration of a flocculant polymer diminishes the shear effect. The latter is contradictory to studies done on floc strength. Leentvaar and Rebhun (1983) found that a range of polymers (anionic, cationic and non-ionic) decreased biological floc strength. This further strengthens the argument that both collision efficiency and floc strength are separate parameters and their engineered responses are different.

4.7.7 Conventional vs. Novel Settling Parameters

Measurements collected from this study can be used to determine treatment for each specific activated sludge type to mitigate problems that arise in flocculation. The CSV method, unlike the other flocculation measurements, can be used to measure both collision efficiency and floc strength. Current methods to determine floc strength use turbidity measurements (Cheng et al., 2011). Turbidity measurements have shown to correlate well with floc sizes (r^2 = 0.95), but the turbidimeter must be calibrated for different wastewater matrices or coagulants/flocculants used (Cheng et al., 2011). In this regard, the CSV method is superior as it does not need to be calibrated for different samples. Furthermore, collision efficiency models require particle size information. This can be collected via the use of small angle light scattering instruments (Francois, 1987; Spicer et al., 1998), video cameras with image analysis (Wu et al., 2003), or photometric dispersion analyzer (Fitzpatrick et al., 2003). All other methods referenced to acquire collision efficiency or floc strength require specialized instrumentation that can be expensive and are most likely unavailable to municipalities. Material for the CSV test are inexpensive and can be found at most municipalities. Moreover, ISC is the only method available to quantify by weight the different classes of particles, although light scattering properties of particulates have been used to measure distribution of particles, Farrow and Warren (1993) conclude that light/laser scattering and transmission techniques are good for showing qualitative (rather than absolute) changes in particle sizes in activated sludge systems. ISC allows for quick determination of discrete particle distributions, thus changes in distribution for a particular system can be monitored. The application of this specific metric can be applied to external selectors, whereby operational strategies can be optimized based on the changes in particle settling classes.

TOF and LOSS measurements take into consideration the solids concentration, and measure the transitional concentration. Transitional concentrations were first used successfully as a hindered settling metric and have proven to be more accurate than ISV (Mancell-Egala et al., 2016). By accounting for the differences in stokesian and non-stokesian dynamics, hindered settling measurements were less skewed. Furthermore, all the different sludge volume index measurements provide no information on flocculation dynamics. TOF is novel as it is the first time flocculation dynamics can be monitored with the same level of complexity as the sludge volume index.

4.9 CONCLUSION

Flocculation metrics have been developed previously to better understand activated sludge flocculation. This is the first time discrete particle distribution, collision efficiency, and floc strength have been characterized without the need of specialized instrumentation. This paper developed three new parameters. ISC quantifies discrete particle distribution, allowing for monitoring and optimization of hybrid systems. TOF and k-value quantified flocculation properties in CAS and hybrid systems, respectively. Knowledge of the limiting factor can allow for an appropriate engineered response; addition of polymer/coagulant or installation of physical separators to address flocculation/coagulation or settling limitation, respectively.

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4.11 TABLES

Table 4.1: Source of sludge used for settling studies and their corresponding operational data (values are based on monthly averages).

Sludge Type	Description		Effluent TSS mg TSS L^{-1}	SOR $m^3\ m^{-2}\ h^{-1}$	SLR Kg $m^{-2}\ d^{-1}$
Secondary	Blue Plains secondary activated sludge	Poor Performance	34 ± 10	0.9 ± 0.10	67 ± 13
		Good Performance	17 ± 9	1.2 ± 0.14	111 ± 17
BNR	Blue Plains nitrification /denitrification sludge		7 ± 4	0.9 ± 0.11	56 ± 21
Mainstream	Blue Plains mainstream deammonification pilot		4 ± 0.4	0.033	N/A
Sidestream	Blue Plains sidestream deammonification pilot		12 ± 1.3	0.076	N/A
Seed	Strass WWTP Cyclone sidestream underflow		N/A	N/A	N/A

With SOR = Surface Overflow Rate, SLR= Solids Loading Rate

Table 4.2: Conventional and proposed settling metrics of sludge types used in the study. The parameters include ISV, $sSVI_{30}$, Limit of Stokesian Setting (LOSS), Intrinsic Aggregates (solid fraction above CSV of 1.5 m h^{-1}), Threshold of Flocculation (TOF), and α values (Exponential fit parameters of settling rates).

Sludge Type	**Conventional metrics**		**Proposed metrics**			
	$sSVI_{30}$ (mL g^{-1} TSS)	**ISV (m h^{-1})**	**LOSS (mg TSS L^{-1})**	**Intrinsic Aggregates (%)**	**TOF (mg TSS L^{-1})**	**α value**
Secondary: Poor Performance	283	0.4	780	3	700	-1.50
Secondary: Good Performance	116	3.3	1000	3	400	-1.00
BNR	88	4.0	1200	7	250	-0.81
Mainstream	121	4.4	1100	19	150	-0.37
Sidestream	81	4.8	1500	40	N/A	-0.22
Seed	21	4.5	5000	96	N/A	-0.07

4.12 FIGURES

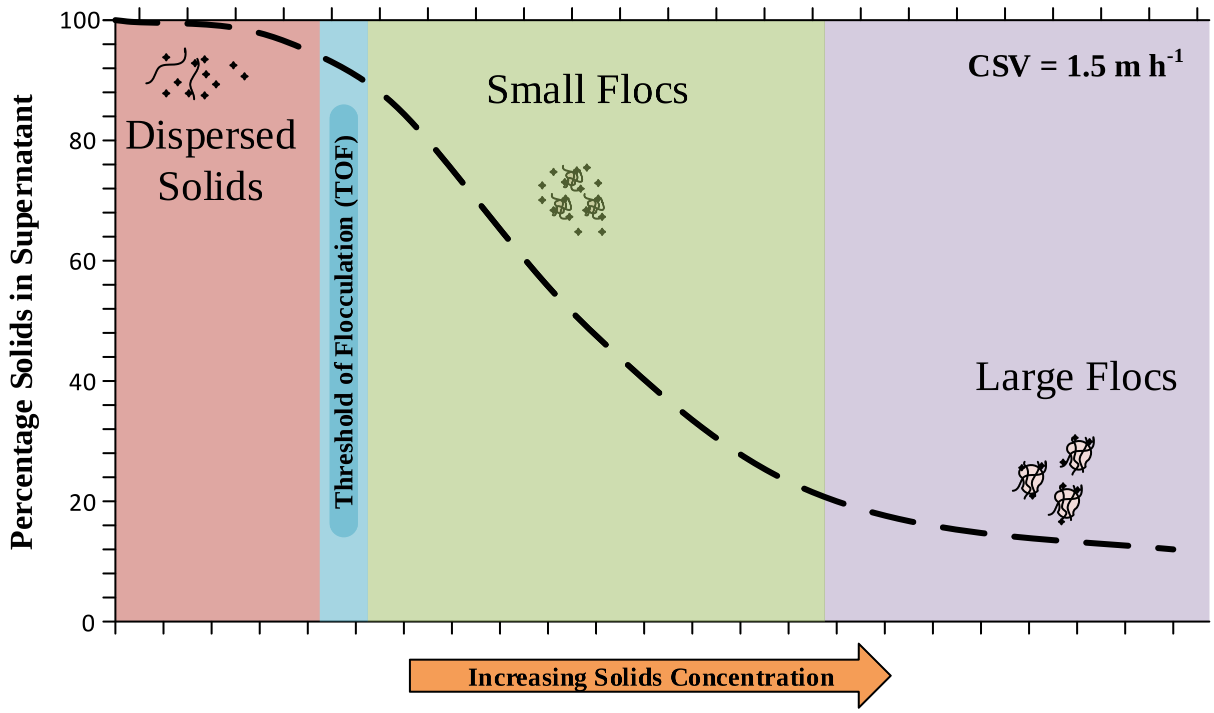

Figure 4.1: Concept diagram of threshold of flocculation (TOF) that can occur when varying solids concentration at a critical settling velocity of 1.5 m h^{-1}.

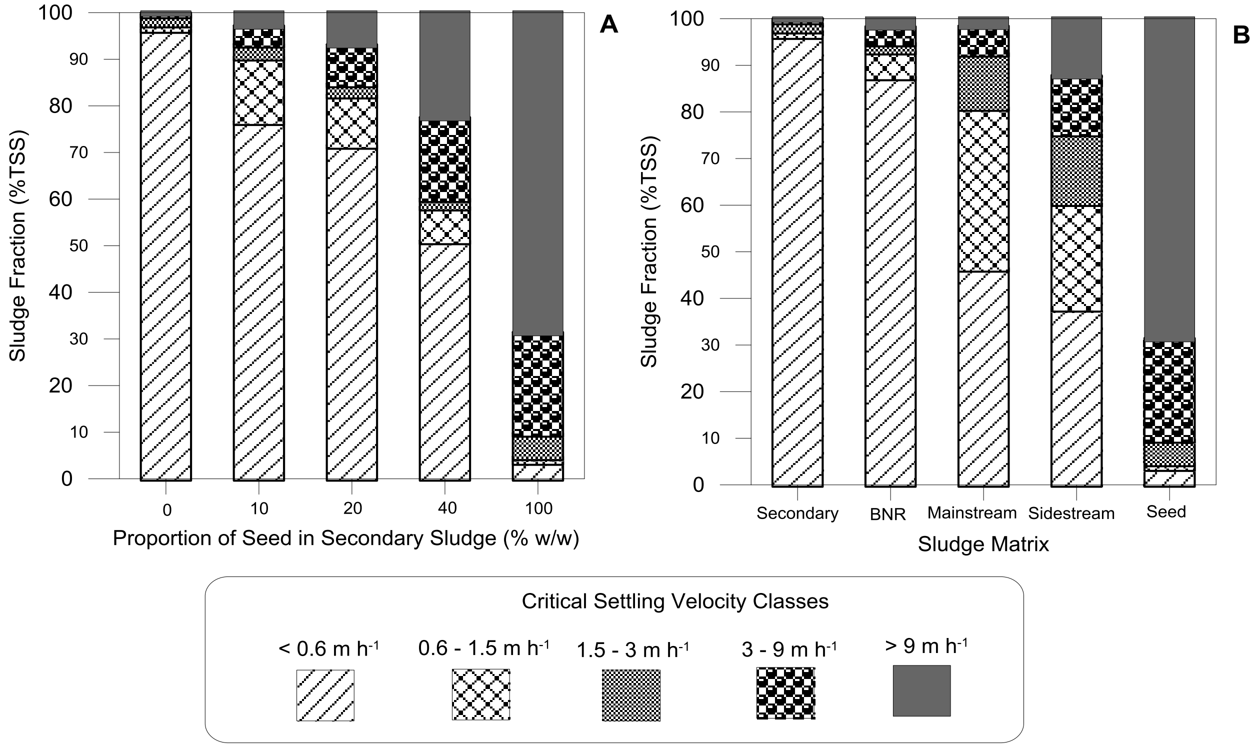

Figure 4.2: (A) Intrinsic settling classes (ISC) for artificial mixtures of secondary good performance sludge (flocculant) with different seed sludge (granular) percentages and (B) Stacked bar graphs showing ISC for five model types of sludge obtained under discrete settling conditions.

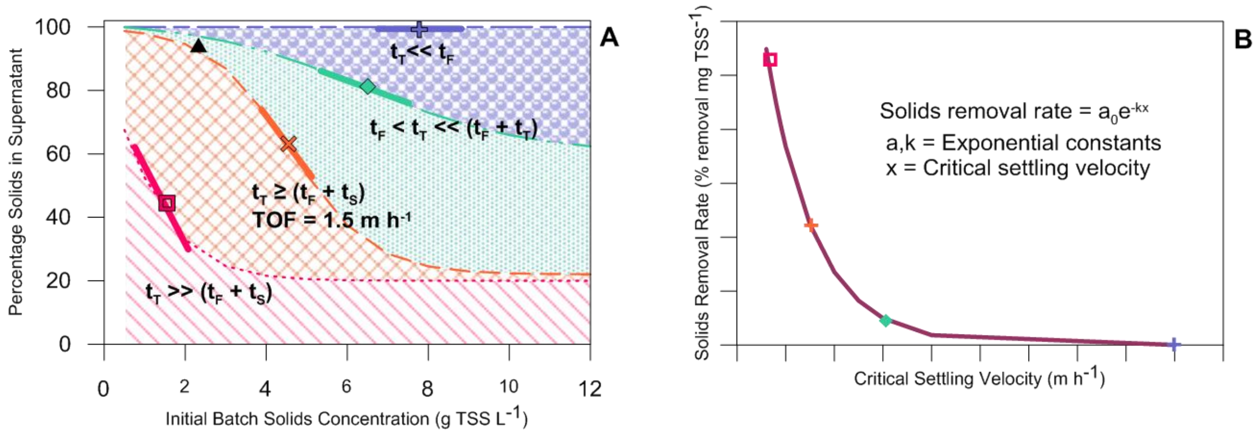

Figure 4.3: (A) Concept of solids that remain in supernatant at varied initial solids concentrations. Varying settling time (tT) for a particular type of flocculant sludge results in four possible scenarios (represented as dotted lines). Triangle highlights Threshold of Flocculation (TOF) and the shaded region represents the solid fraction for a corresponding settling time. (B) Solids removal rate curve is created from the slopes of the different scenarios which are represented by solid lines (A) and plotted against critical settling velocity.

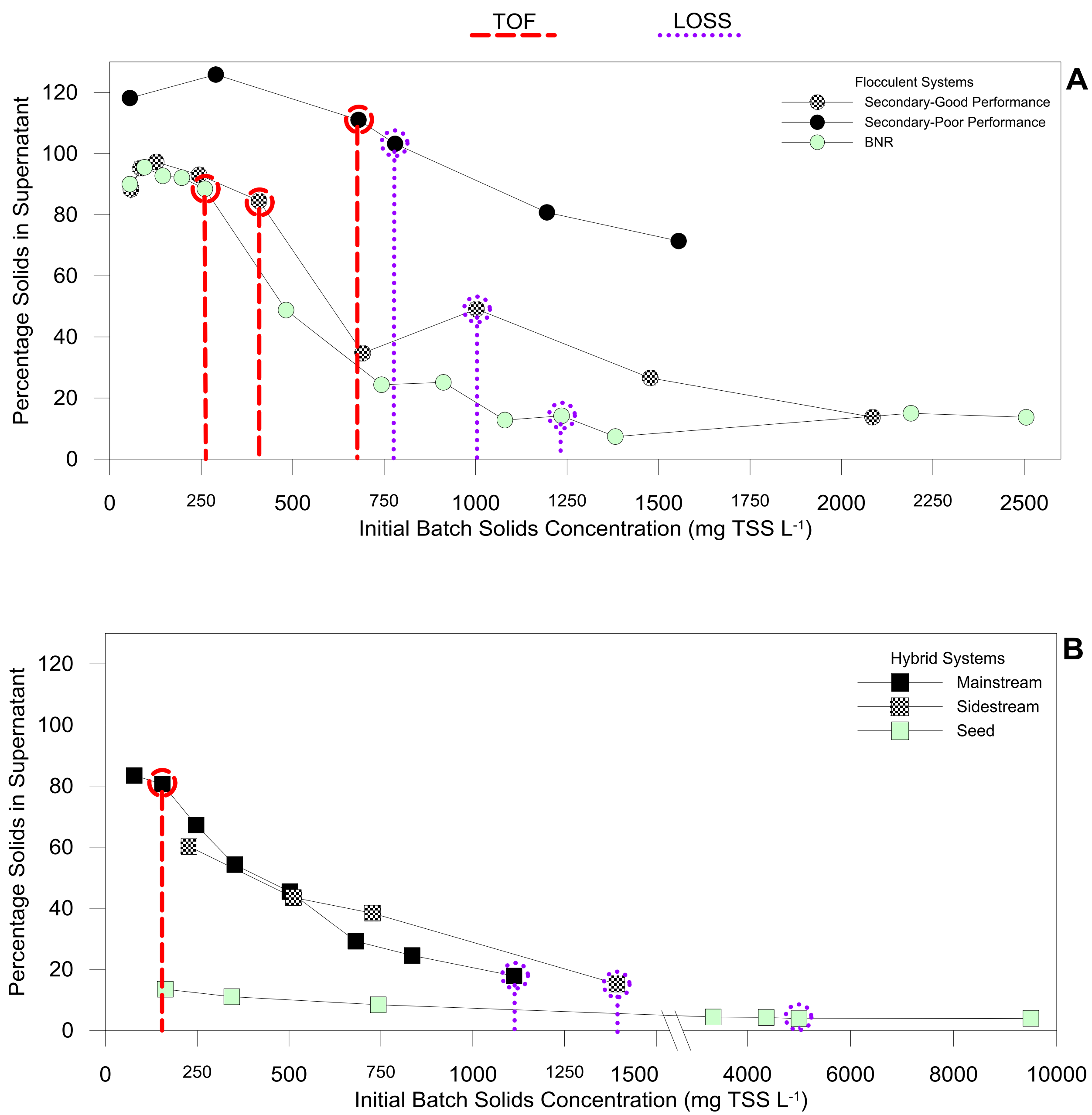

Figure 4.4: Solids remaining in the supernatant (% of initial TSS) at CSV of 1.5 m h^{-1} was plotted against concentration in all six different types of sludge. The difference in TOF (orange dotted line), and LOSS (purple dotted line) highlighted for (a) flocculant systems, and (b) hybrid systems.

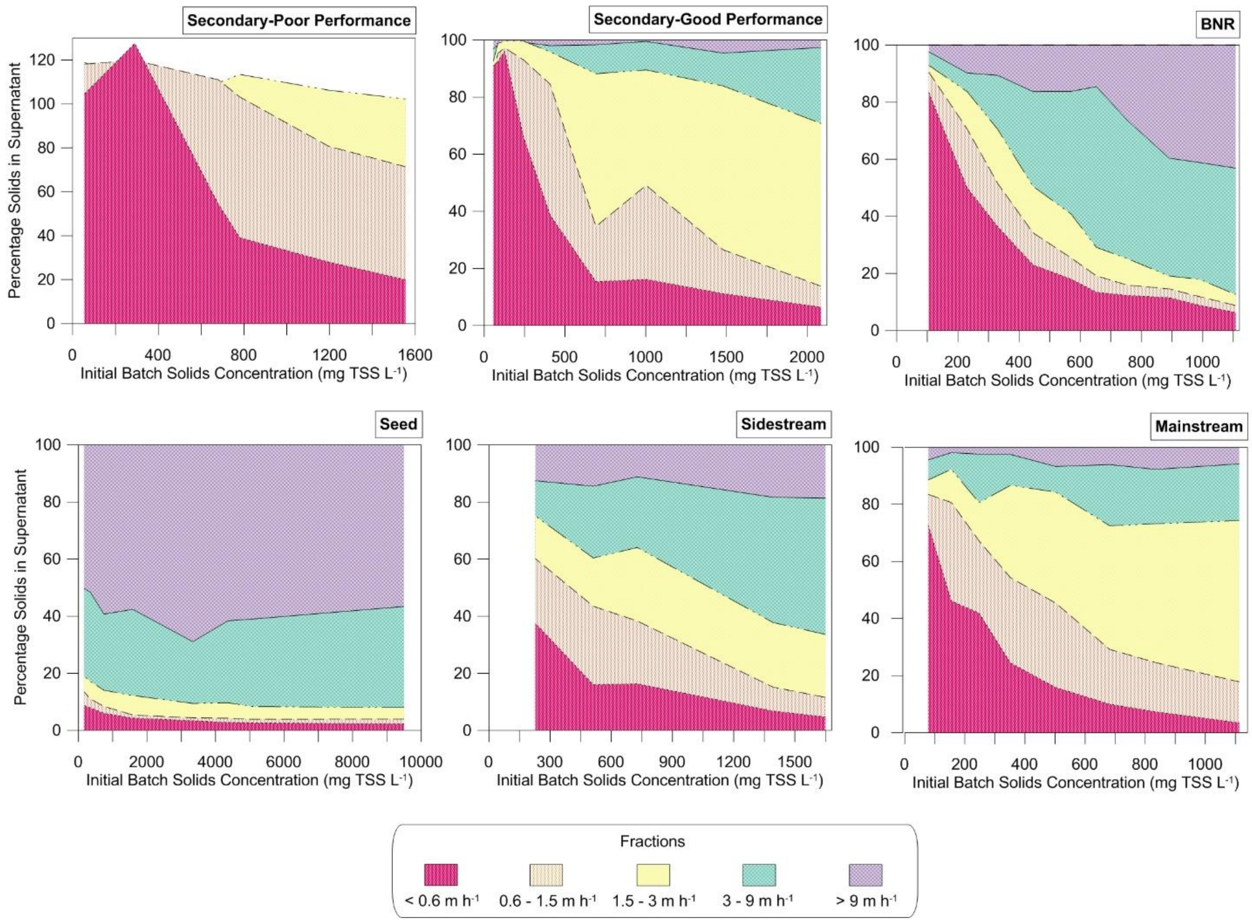

Figure 4.5: Solids remaining in the supernatant at a varying CSV was plotted against solids concentration for six types of model sludge; (A) Secondary poor performance, (B) Secondary good performance, (C) BNR, (D) Seed, (E) Sidestream, (F) Mainstream. (Bulking or flocs floating upwards will result in a supernatant solids above 100%)

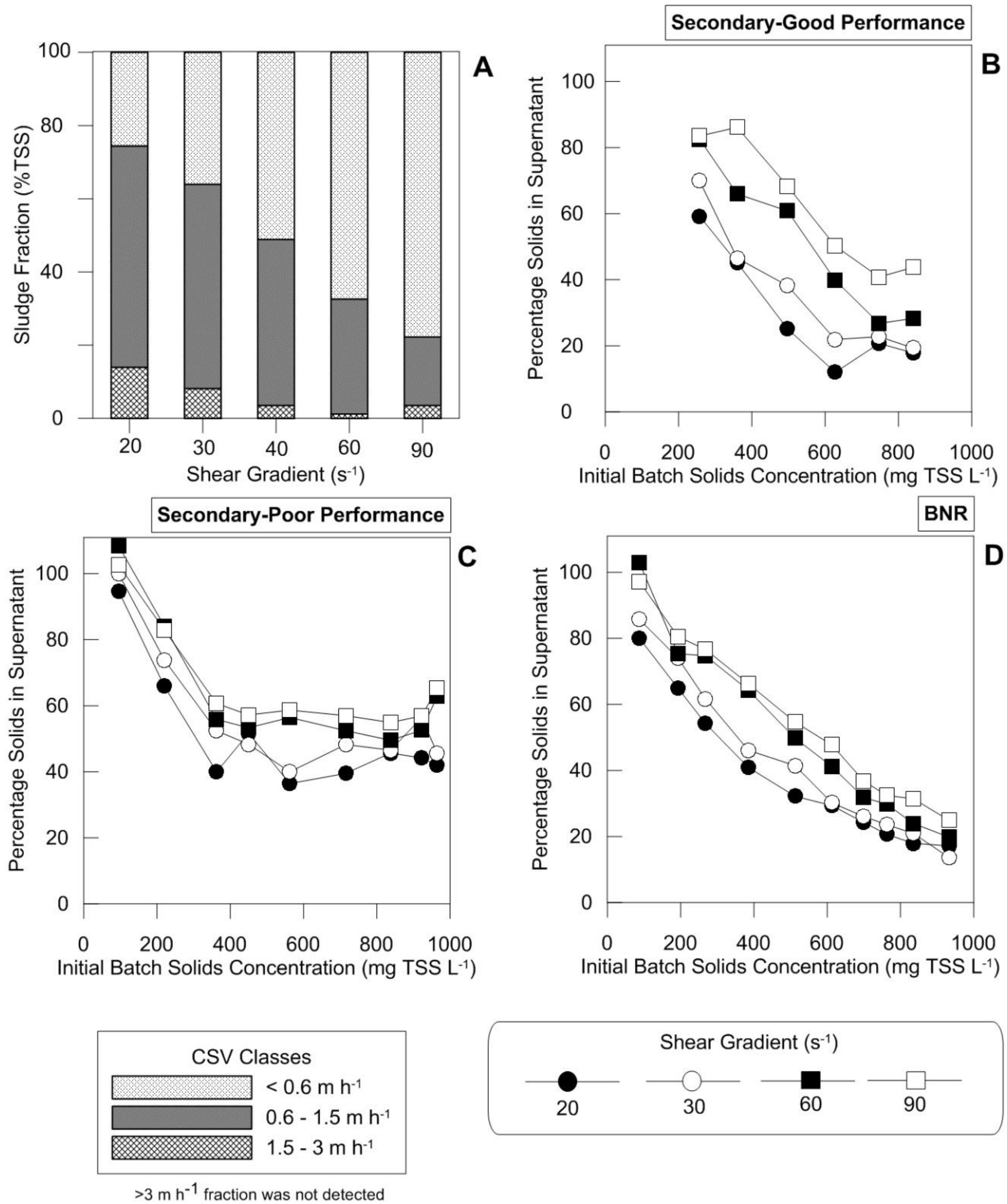

Figure 4.6: (A) Impact of shear on the floc settling classes formed after flocculation (500 mg TSS L^{-1}) for secondary good performance sludge. (B-C) Impact of shear on floc structure for (B) secondary: good performance, (C) secondary: poor performance, (C) BNR.

CHAPTER FIVE

SETTLING REGIMEN TRANSITIONS QUANTIFY SOLID SEPARATION LIMITATIONS THROUGH CORRELATION WITH FLOC SIZE AND SHAPE.

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5.2 ABSTRACT

This study monitored three different activated sludge systems from the Blue Plains AWWTP for a 1 year period to explore the relationship between effluent suspended solids (ESS) and activated sludge settling and flocculation behavior. Hindered settling rates (ISV) and sludge volume index (SVI) measurements were collected weekly. Novel metrics based on the solids concentration at which the transition between settling regimens occurred were also collected weekly. The transitional metrics were Threshold of Flocculation (TOF), and Limit of Stokesian Settling (LOSS). They marked the transition from discreet to flocculant settling, and from flocculant to hindered settling, respectively. A pilot clarifier and settling column were run and filmed to determine floc morphological properties. SVI was found to lose sensitivity ($r < 0.20$) when characterizing ISV above a hindered settling rate of 3 m h^{-1}. ISV and LOSS had a strong correlation ($r = 0.71$), but ISV was subject to change, depending on the solids concentration. Two sludge matrix limitations influencing ESS were characterized by transition concentrations; pinpoint floc formation, and loose floc formation. Pinpoint flocs had TOF values above 400 mg TSS L-1; loose floc formation sludge had TOF and LOSS values below 400 mg TSS L^{-1} and 900 mg TSS L^{-1}, respectively. TOF was found to correlate with the particle size distribution while LOSS correlated to the settling velocity distribution. The use of both TOF and LOSS is a quick and effective way to characterize limitations affecting ESS.

5.3 KEYWORDS

Final Clarifier, activated sludge, particle size distribution, settling velocity distribution

5.4 NOMENCLATURE

AWWTP	Advanced wastewater treatment plant
BNR	Biological nutrient removal
$cBOD_5$	5 day biological oxygen demand from colloids
CAS	Conventional activated sludge
d_{30}	Volumetric diameter
DAF	Dissolved air flotation
dHRT	Dynamic sludge retention time
ESS	Effluent suspended solids
F_p	Sludge produced in the activated sludge process
HRT	Hydraulic residence time
ISV	Initial settling velocity
LOSS	Limit of stokesian settling
M	Mass of solids in a system
n	Number of units in a subgroup of the sample under study
N	Total number of units in the sample
PCA	Principal component analysis
r_h	Parameter associated with settling behavior in hindered settling zone
SLR	Solids loading rate
SOR	Surface overflow rate
SRT	Sludge retention time
$sSVI_{30}$	Stirred sludge volume index at thirty minutes
TSS	Total suspended solids
VSS	Volatile suspended solids
v_F	Average floc settling velocity
v_s	Settling velocity constant
WLR	Waste liquor return

5.5 INTRODUCTION

The removal of suspended matter from water (or clarification) is one of the major goals of wastewater treatment. The conventional activated sludge (CAS) treatment process is the most common in use at wastewater treatment plants, however, the clarification step has been the bottleneck (Ekama et al., 2002). Flux theory has been the dominating influence in the clarification process, and this theory assumes that flocculated solids settle as a blanket, leaving a clarified overflow layer above. According to this theory, the overflow liquid (effluent) is either of low effluent suspended solids (ESS), or the blanket rises and overflows, resulting in a high solids overflow. The theory focuses on the thickening and storage of sludge in settling tanks, and neglects the dynamics of the ESS. Models based on flux theory focus on predicting sludge blanket height while assuming a fixed ESS under steady state-conditions, and only predict changes in ESS when failure occurs from a rising blanket (Takacs et al., 1991; Ekama et al 1997; 2002). Under standard operation changes in effluent quality occurs under stokesian settling, and conventional metrics (based non-stokesian) lack the capability to characterize ESS dynamics.

Settlers are designed to remove particles above a certain mass based on the surface overflow rate (SOR) (Metclaf and Eddy, 2004). The biomass in CAS has densities close to water (Shuler and Jang, 2007), therefore aggregation (bio-flocculation) of the biomass to form a floc is necessary to increase particle size and thus settling rate. Depending on the environment and sludge matrix the solids stick together and transition from a micro-floc (barely visible to the eye) to a macro-floc (easily visible to the eye). Smaller flocs (or particles) tend to have low mass and low removal efficiencies (Boller and Blaser, 1998). Moreover, flocs that are less compact tend to have low strength (Boller and Blaser, 1998). Flocs with low strength break up into smaller particles in regions of high stress present in channels and clarifiers (McCurdy et al., 2004). Current knowledge of floc formation for activated sludge identifies four underlying mechanisms responsible for size and compactness; double layer compression, charge neutralization, bridging, and colloid entrapment (Bratby et al 2006). Limitations in any of these mechanisms can be corrected by the appropriate engineered response (e.g. more salt addition to increase double layer compression). Knowledge of the type of limitations of the bio-flocculation process for a specific CAS system, can help operators correct for them and decrease ESS.

Current (or conventional) settling characteristic metrics used at municipalities are the sludge volume index (SVI) and initial settling velocity (ISV). The sludge volume index can be measured in several ways. Bye and Dold (1998) found sludge volume index done at a solids concentration above 3.5 g TSS to be most popular in use in the UK because of minimal variance with increasing solids concentration. Furthermore, using a continuous moving rake to diminish wall effects can further improve the precision of the method and is known as stirred sludge volume index ($sSVI_{30}$). ISV is collected at most municipalities and it is done at the reactors solids concentration. This practice makes comparing ISVs difficult, because ISV changes with solids concentration. From a previous year-long study done at Blue Plains AWWTP both $sSVI_{30}$ and ISV were found to have no correlation to ESS when done at a fixed solids concentration (Mancell-Egala et al., 2016). However, they have been proven to be good indicators for the thickening and compression of sludge (Takacs et al., 1991; Chancelier et al 1996; Metclaf and Eddy, 2004; Bürger et al 2007; David et al., 2009).

Studies have looked at developing settling characteristic metrics based on the transition between settling regimens (Mancell-Egala et al., 2016; in preparation). There are four settling regimens; discrete, flocculant, hindered, and compression (Ekama et al., 1997). Metrics based on transitional concentrations, unlike conventional metrics, factor in the variance in settling at different solids concentration. The use of transitional settling metrics instead of conventional metrics is more practical for monitoring a system with short sludge retention time (SRT) also referred to as a high-rate systems. Initial studies into the transition between discrete to flocculant settling was suggested as a means to characterized collision efficiency and can be defined as the threshold of flocculation (TOF) (Mancell-Egala et al., in preparation). Collision efficiency is a measure of the success of two particles sticking together after a collision. This mechanism is directly related to the energy barrier concept of the Derjaguin, Landau, Verwey, and Overbeek (DVLO) theory (Elimelech, 1991). If the energy barrier of a colloid is high, fewer colloids have enough kinetic energy for a successful collision. This limitation affects floc size and can be characterized by a TOF value. The transition from flocculant to hindered settling, defined as limit of stokesian settling (LOSS), characterizes the settling velocity of a floc (Mancell-Egala et al., 2016). According to stokes law, size and shape of a floc determines settling velocity. More compact flocs are more spherical according to fractal analysis (Bushell et al., 2002), and more spherical objects have less drag and settle faster according to stokes law. Therefore, using both TOF and LOSS in tandem can help characterize the compactness of a floc.

In this research, two high-rate secondary treatment systems and one nitrification/denitrification treatment system at Blue Plains AWWTP were monitored for a year. The nitrification/denitrification treatment systems' ESS has been historically low and stable. However, the high-rate secondary systems' ESS has historically been unstable and frequently high, the causes might be attributed to either floc size, floc structure, or both. The goal of this paper was to monitor changes in ESS and assess settling regimen transitional metrics ability to characterize floc size and structure. To accomplish this goal the study undertook four tasks: (1) Conventional settling metrics and settling regimen transitional metrics were collected for a 1 year period on all three systems; (2) Operational data was collected and analyzed to determine specific parameters that might influenced ESS; (3) Image analysis was conducted to characterize the size and shape of the flocs; (4) A pilot clarifier was run to explore 1-dimensional solids separation for different sludge types and evaluate solid-liquid separation specific to sludge type and the floc's resistance to hydrodynamic shear.

5.6 MATERIALS AND METHOD

5.6.1 Sample location, collection, preparation

Blue Plains AWWTP is the largest advanced wastewater treatment plant in the world. The plant treats on average 375 million gallons of raw sewage per day, originating from Washington DC, and neighboring counties in Maryland and Virginia, USA. Blue Plains was chosen to test our settling characteristic metrics due to the diverse systems that are incorporated into the facilities wastewater treatment processes. There are high rate (1-2 day SRT) secondary systems with large changes in sludge quality over a short period of time as a consequence of short SRT systems due to aggressive wasting strategies. This allows for the sensitivity and variance testing of all settling characteristic methods. There are also low rate systems that are more stable in terms of sludge quality, making it possible to assess the reproducibility of the proposed methodology. Samples were collected from three separate treatment systems located at the Blue Plains facility;

- The West system: a high-rate secondary treatment process for the removal of organic carbon and phosphorous. The system consists of two plug flow reactors of similar size/dimensions. The SRT is kept between 1 – 2 days.
- The East system: a high-rate secondary treatment process for the removal of organic carbon and phosphorous. The system consists of a total of four reactors; two plug flow reactors of similar size/dimensions and two smaller reactors of similar size/dimensions. The SRT is kept between 1 – 2 days.
- The Biological Nutrient Removal (BNR) system: is the nitrification and denitrification treatment of the effluent from the West and East systems. The system contains 12 plug flow nitrifying reactors and 8 plug flow denitrifying reactors with a post aeration before the clarification step.

The clarifiers dedicated for the West and East system are identical in total size and dimension. BNR clarifiers had a similar surface area but were 30% deeper than the East and West system. The West and East system differed in influent composition as well as reactor size. BNR wasted activated sludge was being fed to the West system during the first 5 months of monitoring before being switched to the East system. Additionally, waste liquor return (WLR) was being fed to the West systems exclusively for the first 5 months, then around 40% of the WLR stream was diverted to the East system for the remaining 7 months of monitoring. The WLR comprised of reject water from the digestion process and dissolved air flotation (DAF) effluent. Samples were collected at the end of the reactor passes, at a channel before being fed to the clarifiers. Sampling was done using a bucket instead of a pump to avoid shearing of the floc. Samples were collected at the same time every day to avoid variability due to diurnal flow for 1 year. Sixty liters of samples from each system were collected during individual trials. Samples were collected every week, excluding wet weather days, to avoid anomalous operational conditions. Water used for dilutions was from the final effluent from the Blue Plains plant and was dechlorinated to avoid changing the water chemistry and deterioration of flocculation.

5.6.2 Operation data collection and preliminary analysis

Operational data from the Blue Plains AWWTP was collected from the process control system and supervisory control and data acquisition system. The flow and solids concentration data was used to construct mass balances for the different systems. Flow rates were collected from several probes while solids concentrations were determined at the Blue Plains research lab. The operational data was also used for dynamic sludge retention time (dSRT) calculations based on Takacs et al., (2008) formula;

$$SRT_t = SRT_{t-1} + 1 - \frac{SRT_{t-1} \cdot F_p}{Mx_t} \qquad (1)$$

Where Mx is the mass of solids in the system and F_p is the sludge produced in the activated sludge process. At steady state condition, F_p is the waste sludge. F_p can be estimated by summing the observed sludge production and mass lost by endogenous decay. Endogenous decay can be estimated assuming a volatile suspended solids (VSS) decay coefficient of 0.05 to 0.06 per day times the mass of mixed liquor's VSS. The observed sludge production can be predicted by the yield concept by developing a linear relationship between the observed sludge production and the influent $cBOD_5$.

5.6.3 Transitional concentration metrics

There were two transition concentration metrics used to characterize floc behavior; transition from discrete to flocculant settling and transition from flocculant to hindered settling. They are known as threshold of flocculation (TOF) and limit of stokesian settling (LOSS), respectively. Both methodologies require the same equipment and the detailed methodologies with the equipment used are presented in previous studies (Mancell – Egala et al., 2016; in preparation)

For the TOF measurement serial TSS dilutions were made ranging from 0.1 g TSS L^{-1} to 1 g TSS L^{-1} for each activated sludge type, with a maximum gap between dilutions of 0.1 g TSS L^{-1}. Each TSS batch underwent a CSV test at 1.5 m h^{-1} and the percentage of the remaining TSS in the supernatant (%) was calculated. TOF was quantified as the deflection point (min, differential of 10%) of the curve plotting the TSS that remained in the supernatant over the initial TSS concentration.

In order to generate the LOSS, a series of dilutions were made from mixed liquor samples. The dilutions were produced between the ranges 0.5 g TSS L^{-1} to 2 g TSS L^{-1} with a maximum gap between dilutions of 0.1 g TSS L^{-1}. Two liters of each batch was placed in a clear jar. The content of the jar was then mixed without a vortex forming and settling was observed visually. To detect LOSS, one must start with a high solids concentration mixture, ensuring an interface which is easy to detect visually. One must then make serial dilutions using the same sludge. LOSS is the highest solids concentration dilution with no detectable interface.

5.6.4 Flux theory testing procedure

A settling column was fabricated in accordance with the Clarifier Research Technical Committee (CRTC) of the American Society of Civil Engineers and is identical to that used by Wahlberg et al. (1995). Initial settling velocity (ISV) and Stirred sludge volume Index (sSVI30) procedures were conducted in this column, as well as a series of ISVs to obtain a solids flux curve (Wahlberg and Foundation, 2001). The TSS for ISVs and $sSVI_{30}$ were kept consistent at 3.5 g to provide. For the generation of a flux curve five ISVs were collected at different solids concentration. The solids concentration ranged from 900 mg TSS/L to 5000 mg TSS/L. De-chlorinated process water was used for all dilutions.

5.6.5 Continuous pilot clarifier procedure

A pilot clarifier was built in accordance to Kinnear's (2002) dissertation. The pilot clarifier is an acrylic column with an inner diameter of 20 cm and total height of 305 cm. Samples are fed through two opposing valves situated at the midpoint of the column (152 cm). The pilot clarifier was operated under an open system, with recurrent 300 L batch collection of secondary mixed liquor fed continuously to the clarifier, while overflow and underflow are disposed down the drain. Masterflex® peristaltic pumps were used for feed and underflow pumps. Sample ports were located at 15 cm intervals such that undisturbed samples could be collected throughout the column depth. A more detailed schematic is shown in supplementary Figure E.1A

The pilot clarifier was run under an operational baseline scenario for all three sludge types (Table 5.1). The operational baseline was based on Blue Plains' secondary system's six week operational hydraulic surface flow rate average. The scenario was run until steady-state conditions could be assumed (4 HRTs). Samples were collected at three intervals; 2nd, 3rd, and 4th HRT. Two samples were collected from each sample location, sample locations included effluent, underflow, feed, and clarifier column. The clarifier column had samples collected every 0.30 m (1 ft) starting 0.15

m below the water surface from the top of the column to 0.15 m from the bottom of the column. The goal of this scenario was to monitor solids concentration profile for different sludge types and evaluate settling characteristic metrics prediction of the actual solids separation process.

5.6.6 Stokesian settling column procedure

A settling column was built in accordance to Ramalingam et al., (21012) study. The apparatus consisted of two parts a 0.93 m acrylic column and a 0.3 m Imhoff cone, combined to give a total volume of volume of 6.5 L as shown in supplementary Figure E.1B. The procedure consisted of first filling the column with dechlorinated plant final effluent. The bottomless sliding well on the top of the column was then filled with 10 mL of mixed liquor sample concentrated to 4 g TSS L^{-1}. Samples were concentrated to ensure there was no limitation to flocculation. The sample was introduced to the column when the well was slides on top of the column.

5.6.7 Image analytics setup and procedure

Both the stokesian settling column and the pilot clarifier column were filmed by a Cannon REBEL T3i camera with a Cannon EF-S 60 mm f/2.8 Macro USM lens. The camera was positioned on motorized dolly setup by Dynamic Perception, LLC. The dolly used was a Stage Zero digital system with AT2 real-time joystick motion controller. The camera was positioned 0.25 m from the column or the clarifier, a detailed schematic can be seen in supplementary Figure E.1C. For the stokesian settling column the recording took place 0.50 m from the top of the column. The pilot clarifier had 3 fixed filming positions situated at, 0.15, 0.60, and 1.05 m from the bottom of the clarifier. All filming positions for the pilot clarifier were below the feed point. This was done because a majority of the solids that were above the feed were below the cameras detection (particle size < 0.5 mm).

All videos filmed were analyzed by MediaCybernetics® Image-Pro Premier 9.1 software. Tracking of the floc was achieved via a color intensity threshold. Once the flocs were identified and tracked specific properties were recorded; velocity, trajectory, ferret diameter, surface area, and roundness. Each flocculant settling column trial had a sludge type filmed for 10 minutes to ensure a majority of solids passed the camera's lens. The pilot clarifier had 3 minute videos taken of each fixed position.

The data that corresponded for each floc were then further analyzed (average surface are, ferret diameter, average settling velocity, and roundness). A volumetric diameter (d_{30}) was calculated and represent the characteristic particle-size distribution (PSD). Flocs in this study were assumed to form spheres and according to Sokovnin and Zagoskina (2002) a volumetric diameter is ideal for representing isometric particles (spheres, regular polyhedral) whose three dimensions are similar. Settling velocity distributions (SVD) and PSD were also constructed. Each bracket of particle-size or settling velocity sludge fractions comprised of the summation of the volume of the total flocs that met that specification. Floc volume was calculated from the surface area of the floc (assuming a sphere). After distribution plots were constructed a log-normal model fit was implemented using the curve fitting toolbox within MathWorks® MATLAB® R2014a. Usually a normal distribution is used to fit distributions when the response variable is nonnegative. However, a least square curve fitting can be used if the experimental errors were additive and can be considered as independent draws from a symmetric distribution, with constant variance. In this study, that is not the case as it is not possible to measure a negative floc diameter of settling velocity. Therefore, multiplicative errors was assumed, symmetric on the log scale, and a least squares method was used to fit the curve. The log-normal model had a strong fit ($r > 0.85$) with the data collected. The detailed steps to construct the PSD and SVD can be seen in supplementary Figure E.2.

5.6.8 Statistical analysis

Principal component analysis (PCA) was done using R x64 version 3.2.2 to analyze operational plant data and find correlations with ESS. A total of 10 variables were analyzed; ESS, SOR, SLR, MLSS, BNR addition, WLR, influent solids concentration, temperature, dSRT, and HRT. PCA is an ordination technique based on the reduction of variables by projecting the initial set of variables into new dimensions (PCs). Each new dimension is determined as an eigenvalue of the correlation (or covariance) matrix. The matrix is obtained from the multivariate matrix of the data set (i.e. 10 variables), so a limited number of dimensions extracts the highest possible variance. The PCs direction is based on eigenvectors associated with eigenvalues. The values that define the original data set is called a Score. The score graph made it possible to compare the relative similarities among variables. PCA's score graph was used to reveal hidden structures in the operational variables by reducing the dimensionality of the data, filter noise from the data, and identify how different variables work together to create the dynamics of the system (Garland and Mills 1991; Victorio et al., 1996). Additionally, a t-test was conducted using Microsoft Excel® to establish variance in settling characteristics and operational data collected. For trials done on the different weeks there is a biological difference in sludge therefore a directional hypothesis was assumed and a two-tail was assumed. Biological differences was assumed to be unequal variance.

5.7 RESULTS

5.7.1 Plant operations effect on the effluent suspended solids

Figure 5.1 shows a large variation in ESS over a narrow range in extrinsic parameters (i.e. SOR, and, SLR) for the West secondary, East secondary, and BNR systems for a 1 year period. The BNR system showed little variance in ESS, and extrinsic parameters. The West and East secondary system had a narrow SOR range that averaged around 0.85 ± 0.12 m h^{-1} and 1.23 ± 0.18 m h^{-1}, respectively. However, the ESS for the West and East secondary systems varied considerably and were recorded around 33 ± 16 mg TSS L^{-1}, and 25 ± 22 mg TSS L^{-1}, respectively. The SLR for both secondary systems varied more than the SOR. Yet, when the ESS variation is examined over a tighter range in SLR the ESS was still observed to vary considerably. When the average SLR ± 20% was examined, the West and East secondary system's ESS was 33 ± 19 mg TSS L^{-1} and 24 ± 16 mg TSS L^{-1}, respectively. It can be concluded that the variations in ESS were not a result of the extrinsic parameters, but might be a result of the intrinsic parameters (changes in sludge and wastewater matrix).

5.7.1 Monitoring Periods

Figures 5.2 and 5.3 show operational parameters and settling characteristics measurements, respectively, for the West, East, and BNR systems. The yearlong monitoring was split into four periods. The periods were selected based on certain key factors. First Period (November – March): BNR sludge and WLR only supplied to the West system. Second Period (April – June): WLR and BNR sludge is introduced to the East system and there is a cessation of BNR sludge to the West system. Additionally SLR is tripled for the East system but decreased by a factor of 1.5 for the West system. Third Period (July – September): A stable period were ESS, SOR, SLR, BNR sludge and WLR addition are less erratic. Fourth Period (October): A short period where ESS increases and WLR increases for the West and East systems.

5.7.2 Operational and settling performance

The BNR systems continued its historically good effluent quality throughout all the four periods with an average ESS of 6.95 ± 4 mg TSS L^{-1}(Figure 5.2A). The SOR for the BNR system averaged around 0.92 ± 0.1 $m^3 m^2 h^{-1}$, and the SLR averaged at 50.13 ± 13 kg m^{-2} d^{-1}. The dSRT remained consistently at 17.9 ± 3 with a brief period in March when the dSRT was at 28 days (Figure 5.2E). Throughout the four periods, the BNR system had a stable TOF of 240 ± 48 mg TSS L^{-1} and LOSS was also stable for a majority of the year averaging at 1300 ± 208 mg TSS L^{-1} (Figure 5.3A-B). The ISV and $sSVI_{30}$ averaged 2.00 ± 0.44 m h^{-1} and 98 ± 14 mL g TSS^{-1} (Figure 5.3C-D), respectively. There was a slight but significant ($p < 0.05$) improvement in all settling characteristics metrics collected in the last two periods compared to the first two periods. This could be attributed to warmer weather, PCA data for the four periods are displayed in supplementary Figure E.3. All settling characteristics measurements over the four periods demonstrated that the BNR sludge had good settling.

The West system suffered from the poorest effluent quality for a majority of the year (Figure 5.2A). During the first period ESS averaged at 24.0 ± 6 mg TSS L^{-1}. Furthermore, SLR and ESS had three peaks that occurred over the same time interval. PCA (see supplementary Figure E.4) showed that both SLR and ESS correlated on dimension 1 (r = 0.60, 0.55, respectively) and dimension 2 (r = 0.75, 0.50, respectively), accounting for 71% of the total variance for that period. WLR addition was reduced by 75% over the course of period 1, however, the ratio of BNR sludge to WLR remained below 1 for a majority of the first period. During the second period ESS increased up to 87 mg TSS L^{-1}. As a result, SOR and SLR were reduced to compensate for the high ESS. The ratio of the total WLR was shifted more towards the East system in an effort to reduce SLR and ESS (Figure 5.2D). All operational parameters stabilized during the 3rd period and ESS averaged at 26.1 ± 4 mg TSS L^{-1}. During the fourth period ESS tripled, although the SLR showed a decreasing trend. SOR remained constant during this period, only WLR increased consistently during the 4th period. The West system's settling characteristics were not all in agreement during the year. The $sSVI_{30}$, ISV, and LOSS portrayed excellent settling while TOF showed the opposite (Figure 5.3A-

D). TOF was consistently the highest for the West system. The 3rd period recorded TOF at its lowest and most stable, averaging at 450 ± 90 mg TSS L^{-1}. In contrast, the West system's $sSVI_{30}$, ISV, and LOSS were the best compared to the other systems.

The East secondary system started the first four months with a stable ESS (at 14.3 ± 3 mg TSS L^{-1}), SOR, and SLR (Figure 5.2A). The fifth and last month in period 1 was characterized by a doubling in ESS and SOR while dSRT was the highest (3.7 days) recorded. In the 2nd period SLR tripled, and SOR increased by 60%. Furthermore, BNR sludge and WLR were introduced into the system during the 2nd period and all these factors might have contributed to highly variable ESS (30.1 ± 10 mg TSS L^{-1}) in the East secondary system. Due to the numerous operational changes, PCA (Figure E.5) was unable to detect any significant correlation ($p > 0.05$) with any of the other variables ($r < 0.25$). During the 3rd period a return to a more stable operation was established. The SOR and SLR dropped down to previous levels, and the ESS remained at its lowest and most stable value recoded for that system (10.5 ± 2 mg TSS L^{-1}). A notable difference between BNR sludge additions to the East compared to the West system was that the ratio of BNR sludge to WLR was above 1 in almost all cases while it was below 1 for the West system. During the 4th period ESS rose sharply and peaked at around 60 mg TSS L^{-1}. This went in hand with a decrease of the BNR sludge to WLR ratio below 1. PCA (Figure E.5) attributed the sharp rise in ESS to WLR with a significant ($p < 0.05$) strong ($r > 0.85$) positive correlation of the two parameters that accounted for 54% of the total variance. The East systems' TOF unlike the West system did not fluctuate much, averaging at 280 ± 80 mg TSS L^{-1} (Figure 5.3). Unlike for TOF, East's $sSVI_{30}$, ISV, and LOSS all were in agreement that introduction of BNR sludge improved settling. All metrics showed significant ($p < 0.05$) improvements for $sSVI_{30}$, LOSS, and ISV by 50%, 200%, and 300%, respectively.

5.7.3 Settling metrics evaluation.

Figure 5.4 shows the comparisons of the settling metrics over the year-long monitoring of the West, East, and BNR systems. In Figure 5.4A we can see a non-linear relationship between ISV and $sSVI_{30}$. As the hindered settling rates increase above 3 m h^{-1} the $sSVI_{30}$ metric becomes less responsive and plateaus ($r = 0.21$). However, when ISV rates are below 3 m h^{-1}, the $sSVI_{30}$ metric has a strong negative linear relationship ($r = 0.80$) with hindered settling rates. Alternatively, LOSS has a strong positive correlation with ISV ($r = 0.77$) and remained linear throughout the ISV range (ISV and LOSS had a r^2 of 0.70 and 0.68 below an ISV of 1.5 m h^{-1} and above an ISV of 4 m h^{-1}, respectively). Figure 5.4C – D show TOF relationship to ESS; For the West system there is a positive correlation ($r = 0.71$), but there is no correlation for the East or BNR system ($r < 0.1$).Furthermore, there was no correlation ($r < 0.2$) to any of the other settling characteristics metrics or operational parameters (Figure E.6).

5.7.4 Continuous pilot clarifier study

Results from the continuous clarifier study are shown in Table 5.1 and Figure 5.5. East was shown to perform better under flocculent conditions or low solids concentration with the highest settling velocity of 24.5 m h^{-1}. The East system also had the highest average flocculent velocity and largest average floc size recorded at 27.6 m h^{-1}, and 4.8 mm, respectively. However, at elevated solids concentration or at a hindered settling regimen the East sludge performed the worst, indicated by the sharpest drop in hindered settling velocity with increasing solids concentration ($r_h = 0.74$). The East sludge had the worst $sSVI_{30}$, ISV, and LOSS at 104 mL g TSS^{-1}, 2.45 m h^{-1}, and 800 mg TSS L^{-1},respectively. In contrast, the West sludge hindered settling rates were the least affected by increasing solids concentration indicated by the lowest drop in hindered settling velocity with increasing solids concentration ($r_h = 0.25$). West sludge had the best $sSVI_{30}$, ISV, and LOSS at 47 mL g TSS^{-1}, 6.04 m h^{-1}, and 1600 mg TSS L^{-1}. However, West sludge performed the worst at low solids concentration or at a flocculant settling regimen with the smallest average floc and lowest average floc velocity recorded at 2.7 mm and 17.4 m h^{-1}, respectively. The TOF metric was in agreement with the image analysis data with the West sludge having the highest value of 800 mg TSS L^{-1}. BNR sludge did not out perform any of the other sludge types in the settling metrics. However, the BNR had similar (within 15%) $sSVI_{30}$ and ISV to East, but its hindered settling was 45% less affected by increasing solids concentration compared to East ($r_h = 0.39$).

The solids concentration profile for the three systems are distinctive (Figure 5.5A – C). BNR sludge took 2 HRT(s) to reach steady-state above and below the feed layer, followed by the East secondary system's sludge at 3 HRT(s). The West secondary system reached equilibrium below the feed layer after 3 HRT(s) but failed to do so above the feed layer. The West secondary sludge ESS continued to rise throughout the entire clarifier run, reaching 120 mg TSS L after 8 hours.

ESS stabilized for the BNR and East secondary sludge at 15 ± 3 mg TSS L^{-1} and 60 ± 5 mg TSS L^{-1}, respectively. Below the feed layer, the solids concentration profile remained constant, a low sludge blanket formed below the lowest sample port. Solids concentration below the feed layer for the three sludge systems ranked from highest to lowest was 450, 400, and 350 mg TSS L^{-1} for West, East, and BNR, respectively.

5.7.5 Stokesian settling study

Results from the stokesian settling column are shown in Table 5.2 and Figure 5.6 and 5.7. The West secondary sludge system went through the largest fluctuation of TOF compared to the other two types of sludge (Figure 5.3). Figure 5.6 A – B show the effects of TOF on the PSD and SVD; an increase in a TOF value resulted in small flocs and shifted the SVD towards the left. This can be seen in Table 5.2, when TOF was tripled the average floc sized decreased by a factor of 5. However, smaller average floc size increased hindered settling velocity with ISV and $sSVI_{30}$ improving by 40% and 25%, respectively. The East secondary system had a more consistent TOF, but LOSS varied over the course of the study (Figure 5.2). Figure 5.6C – D show the effect of LOSS on the SVD and PSD; an increase in LOSS resulted in faster flocs and the distribution became broader, the parameter “a” decreased by 20% with a 75% increase in LOSS. LOSS seemed to have no effect on the size of the flocs but it affected the shape; a higher LOSS produced rounder (about 25% rounder) more compact flocs (Table 5.2). Moreover, more compact flocs settled in hindered manner better, with ISV and $sSVI_{30}$ improving by 12% and 25%, respectively.

5.8 DISCUSSION

5.8.1 Sludge matrix variations

Figure 5.1, 5.2 and 5.3 show that the sludge produced from each of the three systems monitored were vastly different in terms of settling characteristics measurements and solids separation efficiency. Wilen et al (2003) found out that there are very large variations in activated sludge (floc size, morphology, chemical composition) from different systems caused by differences in design and operational conditions. However, within one system there were fewer variations, as seen in the BNR system (Figure 5.1, 5.2, and 5.3). The SRT played a critical role in sludge type; shorter SRT systems have a less diverse community (Duan et al., 2006; Basaran et al., 2013). Furthermore, in a wastewater treatment plant shorter SRT systems are more prone to changes in microbial community because there is a continuous inoculum of microorganisms. The microbial community is associated to bio-flocculation (Faust et al., 2015) and therefore settling characteristics. This can be seen in Figure 5.3 as BNR (long SRT) does not vary in settling characteristics as compared to the East and West systems (short SRT). The results from this study suggests that each sludge type investigated, irrespective of how it varied, had a limiting factor affecting effluent quality, and this limitation can be pinpointed using a combination of stokesian and non-stokesian settling metrics. These limitations affected either or both size and compactness of a floc, an example is the West secondary system that suffered from small flocs (Table 5.2, Figure 5.5), and was identified with the highest TOF values (Figure 5.3).

5.8.2 Floc size

The West secondary system had the most variation in TOF of the three systems monitored (Figure 5.2). Since higher TOF was linked to smaller floc size (Figure 5.6), it can be stated that the West system's ESS limitation was in the formation of macro-flocs as smaller particles generally have lower removal efficiencies (Boller and Blaser, 1998). This is further supported by the West secondary system's positive correlation of TOF with ESS (Figure 5.4C). The East and BNR system's TOF had little to no variance (Figure 5.3), excluding macro floc formation as a limitation. Figure 5.4C and D shows 400 mg TSS L^{-1} as the TOF cutoff for macro floc formation being the leading limitation for elevated ESS. Below 400 mg TSS L^{-1} changes in ESS are no longer caused by the inability to produce large flocs, another limitation was likely responsible.

The East secondary system suffered from the worst metrics associated with non-stokesian settling, while the West system had the best non-stokesian settling values. According to fractal analysis, smaller flocs had a more compact structure than larger flocs, as smaller structures were less likely to have weak points randomly located anywhere across their cross-section (Bushell et al., 2002). Compact floc structures were more spherical and were more likely to retain their shape in a hindered regime, therefore limiting compression and enmeshment. This phenomena has been documented in granules, which are very compact structures that don't compress at all (van Loosdrecht et al., 2013). The West secondary system's flocs were not only the smallest but the most spherical with the lowest roundness number (Table 5.2). The ability of compact floc structures to resist enmeshing when in a hindered settling regimen minimizes the reduction in porosity of the sludge. Kinnear (2002), successfully modelled compression of sludge using the carmen-kozeny equation, and discovered that the porosity of a sludge blanket was the major factor in determining how fast hindered settling occurred. This further supports why the West secondary system had great hindered settling and why the East secondary and BNR system suffered from poor hindered settling.

5.8.3 Floc shape

The compactness/roundness of the floc had a positive relationship with hindered setting rates and compression; more compact particles had faster settling rates than less compact structures of the same mass. This was supported by stokes-law as less spherical objects of the same mass have a higher drag effect and slower settling velocity. BNR's compactness did not vary much (< 5%) and was more compact than the East secondary system on its best and worst days by 10% and 30%, respectively. Additionally, more compact structures are considered to have higher floc strength due to an increase in the number of bonds holding the aggregate together (Jarvis et al., 2005). This was supported by a previous study (Mancell-Egala et al., in preparation) that found the West secondary and BNR systems more resistant to shear than the East secondary system by 15% and 20%, respectively. It can be therefore suggested that the East secondary system had larger loosely formed flocs resulting in low floc strength.

Floc strength is an important operational parameter in the clarification process, Low strength flocs easily break up in regions of high stress around the reactor or clarifier (McCurdy et al., 2004). The shear-flow formula illustrates that increases in flows in the channels leading to a clarifier or SOR would increase the shear exposure to particles. A pattern of increases in SOR resulting in increases in ESS would be prevalent in a system that has a floc strength limitation, and this pattern was noticed in the East secondary system before the introduction of BNR sludge. PCA (supplementary Figure 5.4) showed both ESS and SOR had a strong ($r > 0.85$) correlation in dimension 1 accounting for 69% of the total variance in the East sludge before BNR sludge addition. Furthermore, before the introduction of BNR sludge to the East secondary system LOSS, $sSVI_{30}$ and LOSS were significantly ($p< 0.05$) worse. It appears that a low TOF (large average floc size) and low LOSS (low compactness) characterizes a floc strength limitation. The LOSS for the East secondary system averaged 560 ± 45 mg TSS L^{-1} and 1050 ± 155 mg TSS L^{-1} before and after

BNR addition, respectively. The BNR system can be used as a template for a sludge with had no major limitations in floc strength or floc size, both shown from image analysis (Table 5.2). Furthermore, the BNR system had a LOSS averaging at 1300 ± 250 mg TSS L^{-1} throughout the year and the East secondary system only reached in improved floc strength when LOSS increased up to 1000 mg TSS L^{-1}. Combining these two observation a cutoff of 900 mg TSS L^{-1} for the LOSS value can be used to characterize a floc strength limitation.

5.8.4. Bio-augmentation

The introduction of the BNR sludge to the West and East secondary systems had positive effects on the settling characteristics as well as improved nitrification potential under short SRT. Sludge with BNR addition still displayed about 25% of the original BNR aerobic ammonia oxidizing bacteria activity (Zhang et al., (2015). The addition of BNR sludge can be defined as bio-augmentation as it falls into the bio-augmenter category by improving catabolism of specific compounds (i.e. ammonia) and enhancing removal efficiencies (Herrero and Stuckey, 2015). BNR sludge was a useful bio-augmenter; its effects can be clearly seen in the West sludge's yearly performance (Figure 5.2). The West secondary system ESS and TOF went up three-folds after the termination of the bio-augmentation. Furthermore, SOR and SLR were decreased to reduce the spike in ESS. The termination of bio-augmentation impacted floc size, the West secondary sludge lost the ability to produce large flocs which resulted in small compact flocs (Table 5.2). This was a clear demonstration of how the West secondary systems clarifiers lost capacity when bio-augmentation was dropped. The East secondary system did benefit from bio-augmentation, but its effect could not be observed from operational data. As soon as bio-augmentation began on the East system, SOR and SLR increased. Additionally WLR was added to the system. All these variable changes made detection of bio-augmentations impact difficult. However, all the settling characteristics metrics improved significantly ($p < 0.05$) after bio-augmentation. The most notable of these improvements was the effect on floc morphology. Bio-augmentation improved compactness of the east floc (Table 5.2), making it settle faster in a hindered and flocculent manner.

The improvement BNR sludge had on the West and East systems flocculation can be linked to its SRT. Studies have shown the development of different bacterial population at different SRTs

(Basaran et al., 2013; Faust et al., 2015). Pala-Ozkok et al., (2013) found differences in bacterial population dynamics in acetate utilizing batch reactors at SRTs of 2 and 10 days. The shorter SRT of 2 days, had a bacterial community with faster growth rates than the community at the longer SRT of 10 days. This shows that even though the same substrate was used, different bacterial communities developed as a result. Furthermore, bacterial communities at longer SRTs were characterized to hold a range of genes involved in EPS and alginate production (Albertsen et al., 2013; Faust et al., 2015). Alignate was found by van den Brink et al., (2009) to assist in floc formation by producing a gel-like network when in contact with multivalent ions present in wastewater. This supported findings by Liao et al., 2006 that found out longer SRT sludge had better floc formation. Therefore, introducing long SRT BNR sludge (18 ± 3 days) to short SRT West and East sludge (1.5 ± 0.3 days and 2.0 ± 0.5 days, respectively) will introduce bacterial communities that potentially aide in floc formation.

5.8.5 Colloidal adsorption

The introduction of WLR to both West and East secondary systems had a negative impact on ESS (Figure 5.2). WLR is comprised of the reject water from the digestion process and the DAF effluent. Characterization of the reject water showed a total COD of 3.9 ± 0.07 g COD/L and solids of 0.23 ± 0.003 g TSS/L. DAF flows showed a high total COD of 12.6 ± 0.08 g COD/L and solids concentration of 8.4 ± 0.02 g TSS/L. The WLR samples had no measurable TOF (particulates were unable to form aggregates that could settle out within the 1.5 m h^{-1} critical settling velocity cutoff). Furthermore, a critical settling velocity test showed that 51% of the total WLR TSS had a settling velocity less than 0.6 m h^{-1}. Characterization of the WLR suggested that a majority of the particulates were colloidal and would not settle without being adsorbed onto flocs. Moreover, DAF was defined as the source of the majority (70%) of the particulates in WLR. Inlet streams to DAF processes have been found to be high (51% w/w) in particulate matter below 100 um (Miranda et al., 2015) and extensive polymer dosages are required to efficiently remove these small sized particulates. Therefore, the DAF process at the Blue Plains AWWTP might benefit from an adjustment in polymer dosage in the DAF treatment process.

Period 4 (Figure 5.2 and 5.3) showed clearly that the ESS increased with increasing WLR, while settling characteristics remained the same. Since floc morphology or settling rates were not causing increases in ESS, it was suggested that the adsorption sites on the floc were saturated from the increases in WLR. Adsorption of colloids onto flocs is a well-studied mechanism for reducing ESS and the primary contribution of flocculant aides (Spalla and Cabane, 1993; Chaplain et al., 1995). This can explain why increases in WLR had a strong positive correlation ($r>0.85$) with ESS. Even though ESS increased in both West and East secondary systems during period 4, two different operational strategies were conducted to mitigate the deterioration of effluent quality. The West secondary system reduced the SLR causing a reduction in ESS. However, this was brief and the ESS increased as WLR addition continued to increase. The East systems ESS control strategy had better results as the ESS declined sharply when BNR sludge addition increased and SLR decreased.

Period 4 suggested that saturation of adsorption sites was a third limitation that can result in elevated ESS. However, the ability to characterize this limitation is beyond the scope of this study. It should be possible to characterize the potential adsorption ability of a floc when given size and shape. There is a potential for TOF and LOSS to be used in this manner. However, to calibrate the metrics for adsorption there will need to be a strict characterization of the colloidal material entering and leaving the system. A new experiment would need to be designed in a controlled setting (without the random variations in wastewater supplied) that looked at how adsorption sites change with different floc sizes and compactions.

5.8.6 Implications of TOF and LOSS

The implications of transitional settling concentration metrics evaluated in this study have been shown to be broad and powerful for characterizing solids separation performance under varying settings. Furthermore, ISV and $sSVI_{30}$ have been proven to be less reliable in characterizing non-stokesian settling due to variance in different solids concentration (Mancell-Egala et al., 2016). The pilot clarifier experiment (Figure 5.5) showed how the settling characteristics metrics TOF and LOSS were in agreement with the solids concentration profile along the depth of the clarifier. Since TOF has been associated with particle size and LOSS with compaction of the particle it reasons that;

- A clarifier with West secondary sludge without bio-augmentation (High TOF and High LOSS) had very small compact flocs, low recovery efficiencies, and took longer to reach steady state due to solids accumulation. This can be seen as the feed layer high turbulence and currents prevents solids from settling.

- A clarifier with East secondary sludge with bio-augmentation (Low TOF and Low LOSS) had large loosely formed floc and no solids accumulation. However, floc breakage resulted in high ESS. This can be seen with higher solids concentration near the feed due to high shear from turbulence.
- A clarifier with BNR sludge (Low TOF and High LOSS) had large compact flocs, no solids accumulation issues, and no floc breakage resulting in low ESS. This can be seen with negligent increase in solids concentration near the feed where shear is high from turbulence.

Knowledge of the limitations of a sludge type opens up pathways for effective engineered responses. Ekama et al (1997) characterized sludge systems like the West secondary system with issues forming macro floc to have "pinpoint" flocculation. Mitigating pinpoint flocculation can be achieved by reduction of the energy barrier between particles by double layer compression or charge neutralization. This can be done via coagulants and the West system compared to the East and BNR had the lowest optimal organic coagulant polydiallyldimethylammonium (polyDADMAC) dose (0.04 kg/t) that also resulted in the largest decrease in ESS (47%). The East secondary system was characterized to have floc compaction limitation, this can be mitigated by improving the bridging mechanism via branched polymer addition (Murthy et al., 1998).

5.9 CONCLUSION

Two limitations that cause deterioration of effluent quality were characterized with transitional settling concentrations. TOF was found to characterize floc size and values above 400 mg TSS L^{-1} were found to relate to a floc size limitation. A strong correlation (r = 0.71) with ESS was observed for sludge displaying TOF values greater than 400 mg TSS L^{-1}. LOSS was found to characterize floc settling velocity and a strong correlation (r = 0.77) with hindered settling rates at 3.5 g TSS L^{-1} TSS was found. SVI was shown to be less responsive for fast hindered settling rate sludge (>3 m h^{-1}). LOSS was found to be an indicator of compactness of a floc and sludge displaying LOSS values below 900 mg TSS L^{-1} had a floc compaction limitation. More compact flocs settle faster and were more resistant to shear. BNR sludge was found to be a good bio-augmenter and improved both floc size and strength. The BNR systems long SRT was found to be responsible for producing a diverse microbial community that aides in the flocculation process. WLR was found to increase the colloidal material addition to the systems, and was the cause of a third limitation linked to adsorption. However, this last limitation would require additional experiments to be characterized. Moreover, TOF and LOSS proved to be invaluable metrics, the information they provided can help diagnose and treat effluent quality issues in any activated sludge system.

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5.11 TABLES

Table 5.1: Operational parameters and settling characteristics of West, East and BNR sludge that was used in the pilot clarifier on October 20th.

`	Feed	SOR	SLR	$sSVI_{30}$	ISV	V_S	r_h	TOF	LOSS
	g TSS L^{-1}	$m^3 m^{-2} h^{-1}$	Kg m^{-2} d^{-1}	mL g TSS^{-1}	m h^{-1}	m h^{-1}	-	mg TSS L^{-1}	mg TSS L^{-1}
West	2.06±0.1	1.0	74	47	6.04	14.0	0.25	800	1600
East	1.97±0.1	1.0	71	104	3.00	24.5	0.74	200	800
BNR	2.17±0.4	1.0	78	95	2.45	12.8	0.39	200	1400

Table 5.2: Settling characteristics and image analytical data of West and East that was obtained from the stokesian settling column before and after BNR addition.

Sample	Date	TOF	LOSS	ISV	$sSVI_{30}$	d_{30}	V_F	Roundness
		mg TSS L^{-1}	**mg TSS L^{-1}**	**m h^{-1}**	**mL g TSS^{-1}**	**mm**	**m h^{-1}**	**-**
West: No Bio-Aug	Jun 30th	1500	2200	4.04	49	1.11	13.74	0.49±0.4
West: Bio-Aug	Mar 2nd	450	1900	2.81	65	5.40	27.30	1.08±0.5
East: No Bio-Aug	Feb 16th	300	800	1.43	120	6.31	20.3	1.59±0.9
East: Bio-Aug	Jul 15th	300	1400	1.60	91	6.28	26.0	1.19±0.6

5.12 FIGURES

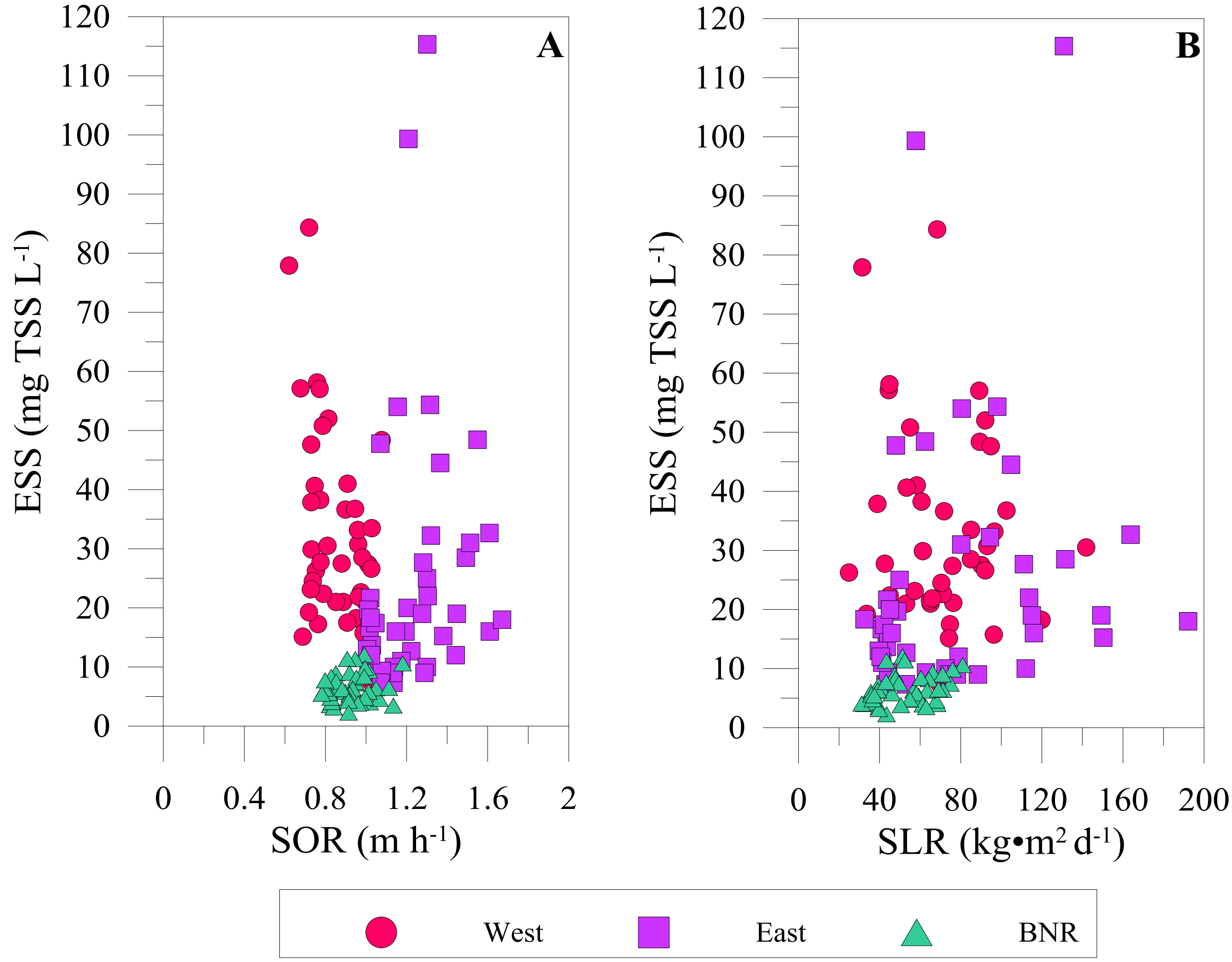

Figure 5.1: Surface overflow rate (SOR) and solids loading rate (SLR) versus effluent suspended solids (ESS) over 1 year of monitoring for West, East, and BNR systems.

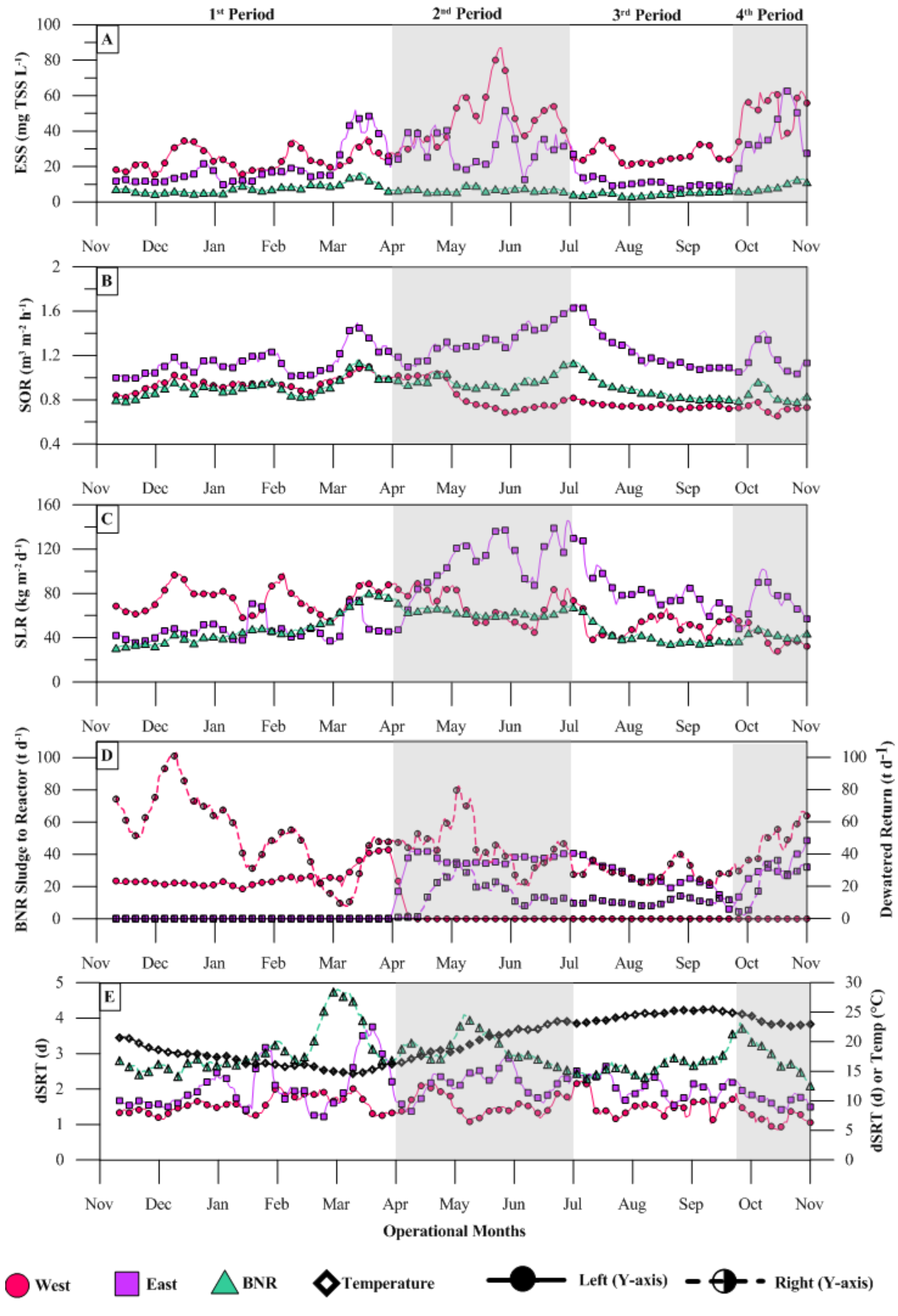

Figure 5.2: 10 day moving average of operational data of the West, East and BNR systems for the 1 year (2014/2015) of observations. The year's data has been split into four periods that have been shaded. Operational data collected were; (A) effluent suspended solids (ESS), (B) surface overflow rate (SOR), (C) solids loading rate (SLR), (D) BNR and dewatered return additions to the West and East systems, (E) dynamic sludge retention time (dSRT) and Temperature (Temp).

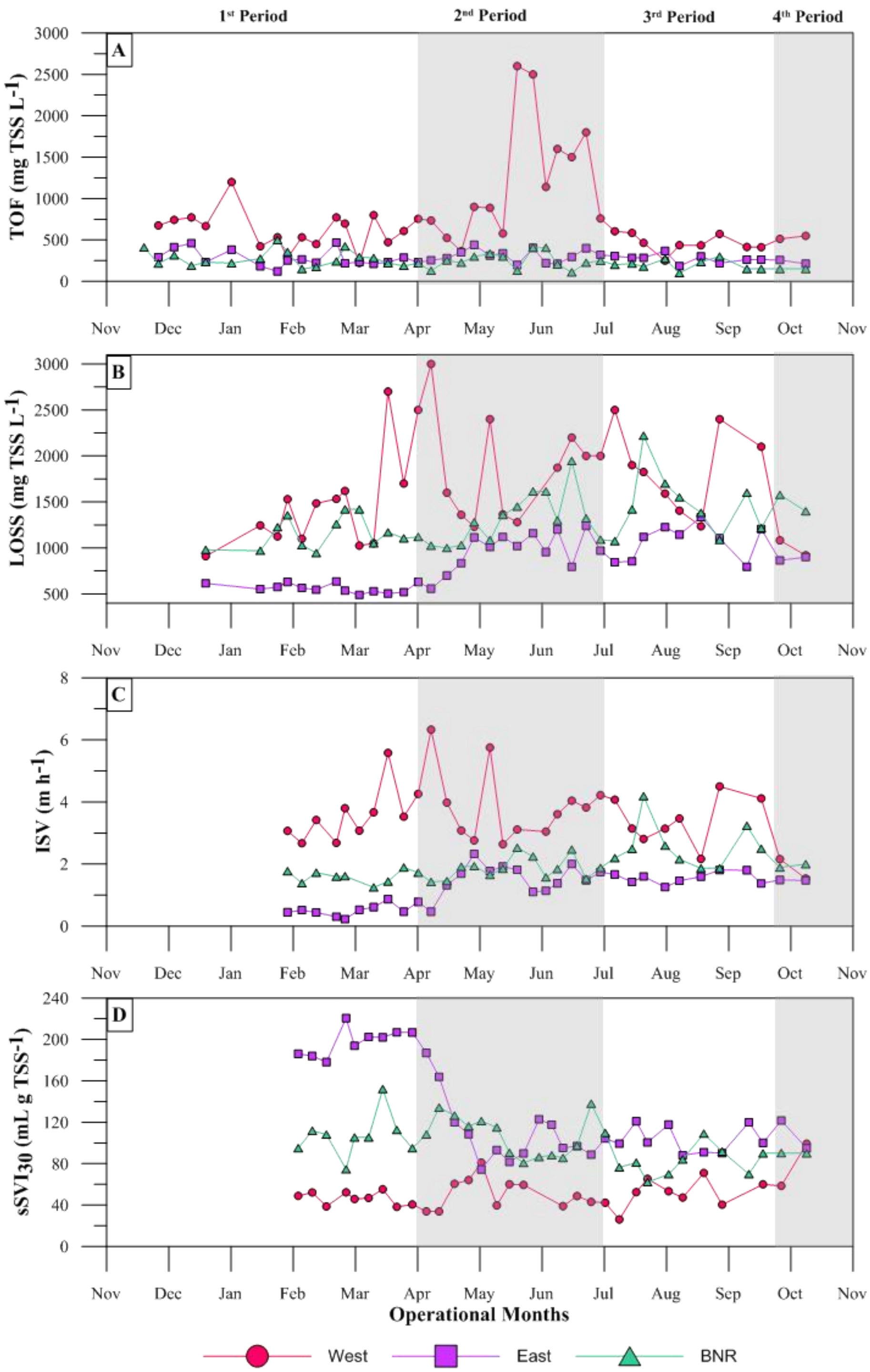

Figure 5.3: Settling characteristics of the West, East and BNR systems for the 1 year (2014/2015) of observations. The year's data has been split into four periods that have been shaded. Settling characteristics collected were; (A) threshold of flocculation (TOF), (B) limits of stokesian settling (LOSS), (C) initial settling velocity (ISV), and (D)stirred sludge volume index ($sSVI_{30}$).

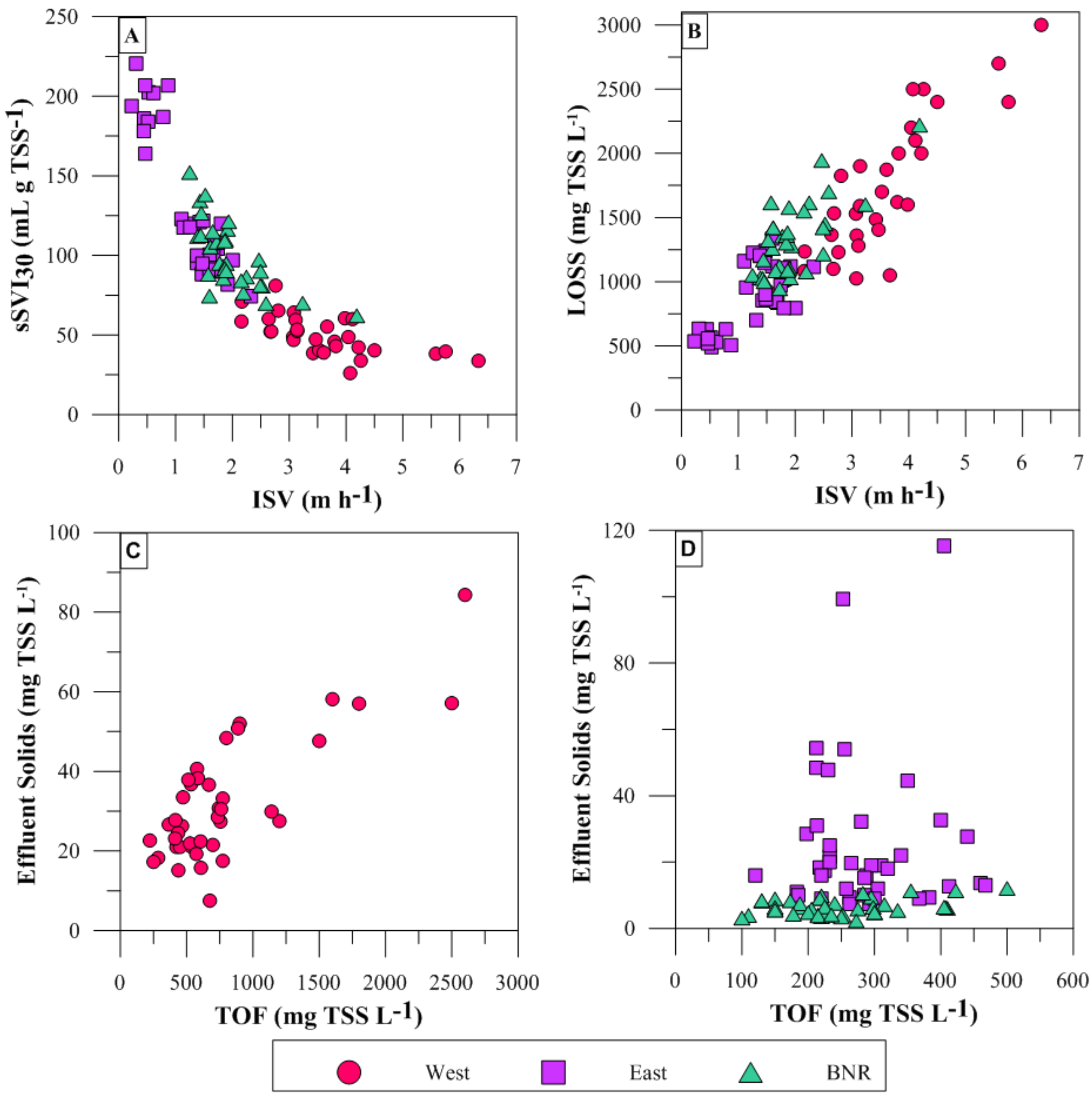

Figure 5.4: Initial settling velocity (ISV) versus (A) stirred sludge volume index (sSVI30), (B) limits of stokesian settling (LOSS) for the West, East, and BNR systems. TOF versus effluent for the (C) West, (D) East and BNR systems.

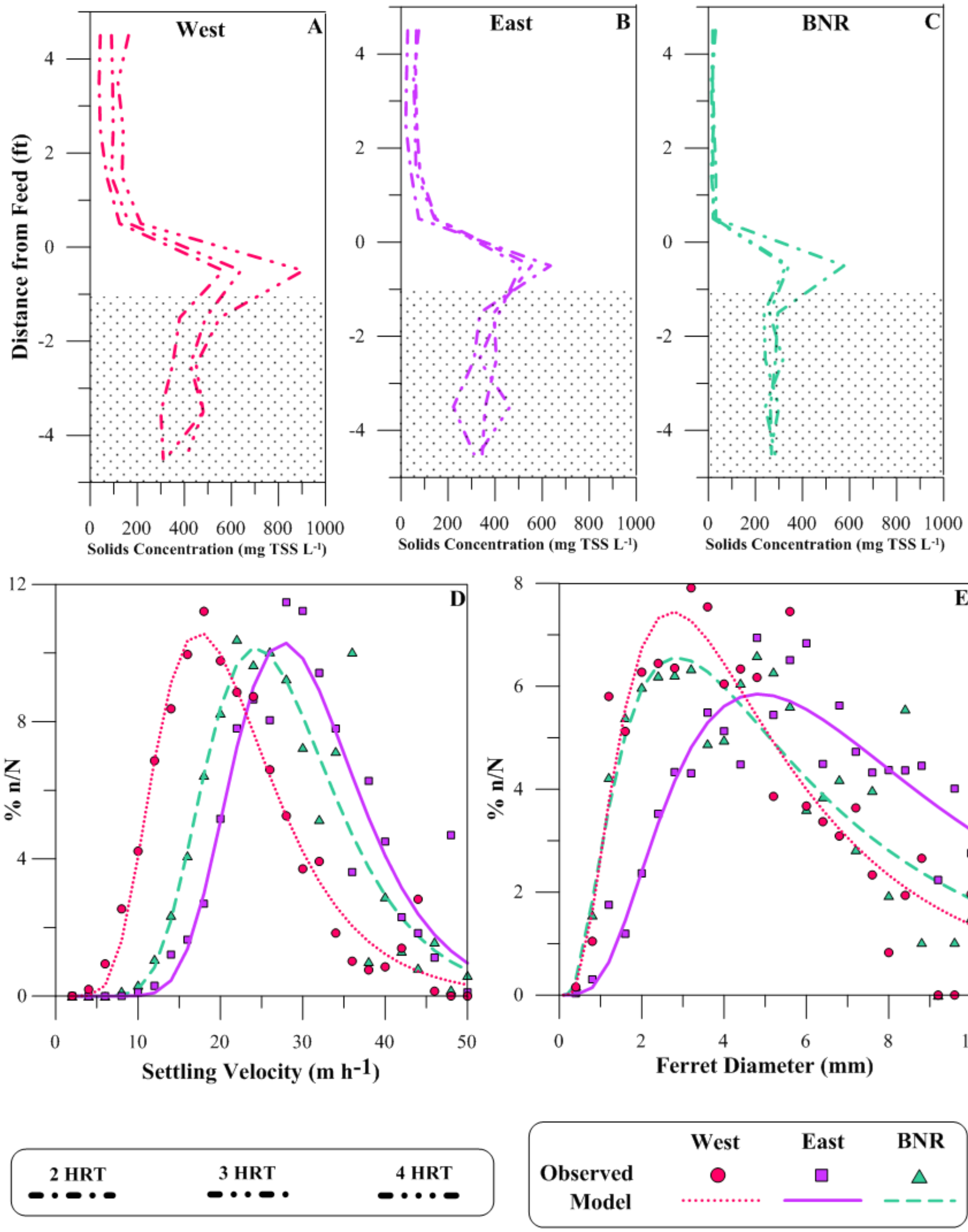

Figure 5.5: Solids concentration profile for the pilot clarifier for the (A) West, (B) East, and (C) BNR sludge. Log-normal fits for the (D) Settling velocity and (E) Particle size distributions for the West, East, and BNR sludge during the pilot clarifier experiment. The solids concentration profile for the pilot clarifier was collected at 2, 3, and 4, HRTs.

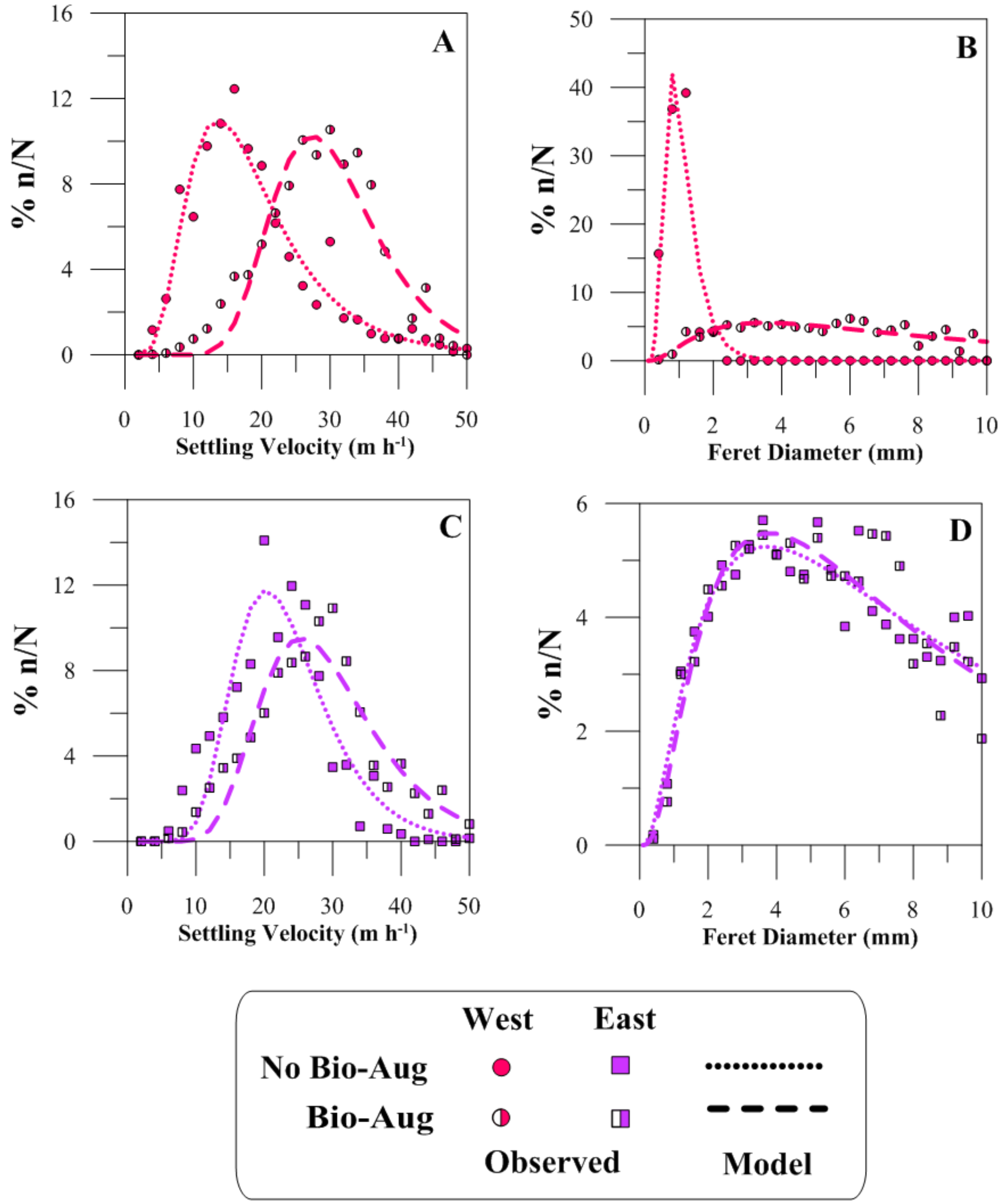

Figure 5.6: The flocculant settling column image analytical data, log-normal fits for the West sludge (A) settling velocity and (B) particle size distributions and for the East sludge (C) settling velocity and (D) particle size distribution. Analysis was done on the periods before BNR addition (No Bio-Aug) and after BNR addition (Bio-Aug).

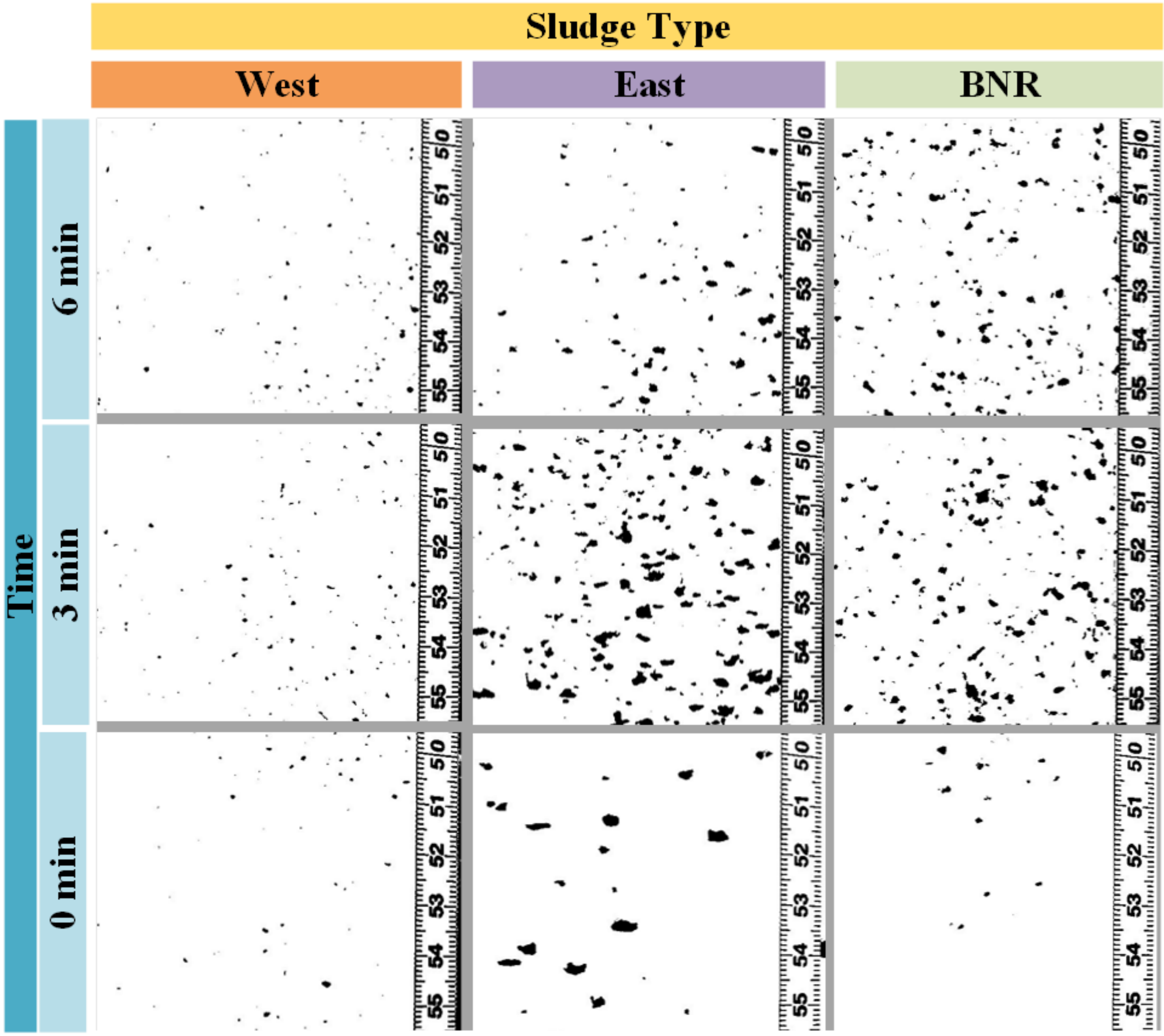

Figure 5.7: The binary picture matrix of the different sludge types in the flocculant settling column. The matrix column represent the different sludge types (West, East, and BNR), and the row represent the flocs distribution at different times during the batch experiment. The last row represents time zero when the first flocs is in the cameras view.

CHAPTER SIX

CONCLUDING REMARKS AND ENGINEERING SIGNIFICANCE

Efficient solid-liquid separation is at the core of sustainable wastewater treatment. Successes in identifying limitations to solid-liquid separation of activated sludge systems is necessary to achieve various ends: *effective carbon capture, increased wastewater treatment capacity, smaller clarifier footprint at treatment plants, and decreased costs of settling additives*. This dissertation was aimed at developing novel settling characteristic metrics based on the current understanding of the fundamental mechanisms that control coagulation, flocculation, and settling. These new metrics are crucial to optimizing design and capacity of current solid-separation processes at the classical limits of operability. This research examined different sludge types that differed in granular fraction, hindered settling rates, and effluent quality at similar clarifier operation. Flocculation mechanics are an old and, in many ways, well understood process. However, there is a lack of settling metrics utilized at wastewater treatment plants that are based on these principals. Current parameter estimation procedures based on flocculation mechanics require equipment that are not readily available to municipalities, due to their expense or sensitivity to the environment. This work explored alternate methods that require simple tools to achieve these parameter estimations based on the current understanding of flocculation. Specific contributions in this area by this research include the following:

Determining granulation and intrinsic aggregate size distribution by critical settling velocity. Quantifying the granule and flocculent fractions that are not possible with other rudimentary conventional methods is now possible. The ability of having a more refined metric for that can distinguish sludge quality at a higher degree helps in computing the ballasting function from the granular fraction and clarification function of a sludge from the flocculant fraction that until now has not been possible. This makes integration of both flocculent and granular systems using the current infrastructure possible. This method allows physical separation processes and operational control strategies to be monitored and optimized so that they can aggressively select and waste granules as desired.

Identifying settling characteristic parameter linked to thickening failure in clarifiers. The limit of stokesian settling (LOSS) concentration was shown to increase for better settling sludge types and decrease for poorly settling sludge types. Gravity flux curves, unlike LOSS exaggerate good settling sludge types and change dramatically for small incremental changes in settling characteristics. LOSS was able to identify and quantify good and bad settling sludge types. Moreover the LOSS test takes half an hour whereas generating a gravity flux curve takes over four hours. Sludge Volume Index (SVI) was a good measure for drastic changes in sludge characteristics, but a more poor method to quantify settling characteristics. A pilot clarifier study validated two hypothesis; there is no hindered settling in a functioning clarifier and LOSS is the maximum feed layer concentration before clarifier failure occurs. This allows operators to monitor the feed layer and have a means of predicting clarifier failure based on the solids loading rate.

Determining collision efficiency and floc strength parameters by using the critical settling velocity. Flocculation metrics such as flocculation collision efficiency, and floc strength have been developed previously to better understand activated sludge. However, this is the first time the latter metrics have been characterized without the need of specialized instrumentation. This research developed two new parameters. Threshold of Flocculation (TOF) and α-value quantified flocculation properties in conventional activated sludge systems and hybrid systems, respectively. Knowledge of the limiting factor can allow for an appropriate engineered response; addition of polymer/coagulant or installation of physical separators to address flocculation/coagulation or settling limitation, respectively.

Linking novel settling characteristic metrics to both floc size limitation and floc compaction limitation that affect effluent suspended solids concentration. Two limitations that cause deterioration of effluent quality were characterized with transitional settling concentrations. TOF was found to characterize floc size and to relate to a floc size limitation. LOSS was found to characterize the floc settling velocity. LOSS had a strong correlation with hindered settling rates. SVI was shown to be less responsive for a fast hindered settling rate sludge. LOSS was found to be an indicator on compactness of a floc. More compact floc settle faster and were more resistant to shear. A sludge matrix that did not have a floc size limitation but fell below the LOSS cutoff had a floc strength limitation. A long SRT sludge was found to be a good bio-augmenter and improved both floc size and strength. A long SRT was found to be responsible for producing a diverse microbial community that aides in the flocculation process. TOF and LOSS proved to be invaluable metrics, the information they provided can help diagnose and treat effluent quality issues in any activated sludge system.

APPENDIX A

SETTLING TRANSITION CONCENTRATION MEASUREMENT TO QUANTIFY SLUDGE SETTLING BEHAVIOR

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A.2 ABSTRACT

A new analytical technique classified as settling transition concentration (STC) was used to determine the total suspended solids (TSS) concentration at which mixed liquor settling characteristics transition from flocculent settling (Type II) to zone or hindered settling (Type II). STC involved producing a series of MLSS/secondary effluent dilutions at with different TSS concentrations in standard settling jar. The resulting settling process was viewed both with the naked eye as well as filmed using a High Definition camcorder. The recording was analyzed utilizing a MATLAB program that determined the settling classification of each mixture. The MATLAB program functions by incorporating the settling jar into a red green blue (RGB) matrix, the mixture is represented by varying color pixels. The MATLAB program analyzes these pixels via colors red, green, and blue with index values that range from 0 to 255. The program plotted the RGB values against the corresponding depth of the jar, and the plot was used to highlight the interface. STC data was analyzed for a period of six months in the Blue Plains Advanced Wastewater Treatment Plant, and it showed that the STC typically occurred between 600-700mg/L but drifted above 700mg/L for a good settling sludge, and below 600mg/L for a poor settling sludge. In addition, STC was collected for mixed liquor samples before and after the addition of Sodium ions, it was observed that STC was lowered by 100mg/L. STC proved to be a useful method in characterizing sludge settling characteristics.

A.3 KEYWORDS

RGB analysis, activated sludge, one-dimensional modeling, batch mixed-liquor settling,

A.4 NOMENCLATURE

AWWTP	Advanced wastewater treatment plant
CFD	Computational fluid dynamics
G	Gravitational solids flux
ISV	Initial settling velocity
MLSS	Mixed liquor suspended solids
RGB	Red green blue
r_h	Hindered settling constant
sSVI	Stirred sludge volume index
STC	Settling transition concentration
STCD	Settling transition concentration determination
TSS	Total suspended solids
V_B	Batch volume
V_T	Total volume
V_S	Sludge volume
v_0	Settling velocity constant
v_s	Settling velocity
X	Solids concentration
X_A	Actual solids concentration
X_T	Target solids concentration

A.5 INTRODUCTION

Secondary clarifiers produce low TSS supernatant as secondary effluent, and also serve as sludge thickeners which provide a constant underflow of thickened return activated sludge (RAS) to the aeration basin. They also provide sludge storage zones which safeguard the storage of sludge during wet-weather events (Ekama et al., 1997). The objective of these clarifiers is to facilitate the gravitational separation of the microbial mass and other particles that either get enmeshed in the mixed liquor or have the ability to settle out from the water. Clarifiers that do not successfully achieve any one of these objectives will eventually allow excessive solids to report to the effluent (supernatant) flow. Therefore, considerable attention has been given to methods to design, evaluate and optimize secondary clarifiers. Despite all these benefits and efforts to optimize these clarifiers, it remains challenging to predict when secondary clarifiers will fail; thus secondary clarification is a limiting factor in wastewater treatment plants (Cheremisinoff, 1995; Ekama et al., 1997; Jeppson, 1996; Lin, 2001).

Common methods employed to analyze clarifier operation include solids flux and state point analysis methods which are incorporated into computational fluid dynamics (CFD) models which simulate the flow field and sludge removing process in clarifiers. This approach was first used by Krebs (1991). This approach utilizes the Navier-Stokes equation to create the models, which are then combined with initial settling velocity (ISV) data collected from column testing, resulting in a computer model capable of predicting short-circuiting areas of clarifiers, sludge digestion, and mixing patterns (McCorquodale et al., 2004). It is important to know the MLSS concentration at which transition to hindered settling occurs for the application of this modeling approach. If this critical MLSS concentration is known, operators might better predict when hindered settling regions form in the upper regions of the clarifier and certain failure modes become likely.

Unfortunately, CFD models which incorporate solids flux and state point analysis methods often do not account for the different settling phenomena such as floc formation, different floc size characteristics, pinpoint flocculation among others which are present in clarifiers and its shortcomings have been raised by many authors (DeClercq, 2003; Ekama et al., 2002; Lakehal, 1999; Li, 1987), who have had concerns with the separation of solids predicted by current settling models. Ekama and Marais (2002) state that the prediction of the sludge settling behavior by these models remains poor; Lakehal et al. (1999) have stated that this approach underestimates the value of the settling velocities in the sludge blanket, which serves to illustrate that these models are not an accurate depiction of the settling activity occurring in a clarifier. These inadequacies in the use of CFD models for settling behavior are illustrated in Figure A.1 below, which depicts the Vesilind equation (used in CFD models) prediction of settling velocity as a function of concentration of the solids present in the clarifier, then contrasts this with actual measured settling velocities done by Kinnear (2002), and McCorquodale (2004).

You might want to keep the shading but change the colors to make this a little less bold?
Figure A.1 indicates that a settling velocity exhibits a sharp discontinuity when settling type changes from Stokesian to non-Stokesian. The labeled region E in Figure A.1 is the concentration where the settling type is changing, this is known as the settling transition concentration. These areas of discontinuity may be due to the different physics involved in each type of settling. Flocculent settling displays Stokesian behavior meaning models will depict a settling particle having three prevalent forces acting on it; gravity, buoyancy, and drag, yet these particles are not perfectly spherical and the porosity of these particles plays a larger role in predicting the settling velocity (Kinnear, 2002). Describing this behavior in modeling efforts remains challenging but has been attempted, whereas hindered and compressive settling exhibits non-Stokesian settling behavior this means an additional force is acting on the particles, which is the contact force of the other particles that reduces the settling velocity. The current model to characterize zonal settling is the Vesilind equation, which will determines the ISV) solely as a function of concentration. Approaches to non-Stokesian settling which begin with a fundamental force balance demonstrate that settling velocity is also a function of the concentration gradient. Flux and modeling approaches have attempted to incorporate this into clarifier design, yet a widely-applied, satisfying approach has yet to be developed.

One approach to developing an improved indicator of secondary clarifier fluid dynamics it to investigate the behavior of the Settling Transition Concentration (STC), which might assist in predicting one-dimensional settleability of MLSS. STC is the TSS concentration in which settling behavior does not clearly follow either the flocculent settling or hindered settling behavior but exhibits characteristics of both types of settling. Monitoring STC and its subsequent changes depends on physiology, biology, and chemical vectors, and should thus include variables that are not considered in traditional CFD approaches. As STC can only be measured after settling has begun to occur, it is important to get an accurate STC measurement, and quickly relate that measurement to traditional settleability measurements.

In this study, the concept of STC is introduced, and its relevance in predicting one dimensional settling behavior of activated sludge is investigated. Direct visual and video (RGB analysis) methods were employed to measure STC and determine if this parameter varies with settleability. RGB analysis entails using a program written in MATLAB to analyze videos and link the TSS in the mixed liquor to color to quantify the type of settling occurring. Additional experiments were also conducted to find an optimum methodology for the direct measurement of STC, and to confirm that this variable varies with settleability. Finally, STC was altered chemically with the addition of Sodium ions in order to validate that STC is linked to settleability.

A.6 DESIGN APPROACH

A.6.1 Settling Column

The settling column was fabricated in accordance with the Clarifier Research Technical Committee of the American Society of Civil Engineers and is identical to that used by Wahlberg and Keinath (1988, 1995). A schematic of the column is shown in Figure A.2. Three sump pumps were utilized: one to serve as a water bath (¼ HP Flotec), and the other two to supply mixed liquor to the columns (Flotec ½ HP). Standard measuring cylinders were used to measure supernatant and activated sludge samples.

A.6.2 TSS Batch Creation

Once mixed liquor is obtained with its estimated TSS value, an excel spreadsheet is then used to calculate the ratio of supernatant and pure mixed liquor to make a batch of mixed liquor with a desired TSS value (equations 1 and 2). Thickening or dilutions were then conducted as necessary, and the actual TSS value is then measured. The formula used in the excel spreadsheet is shown in Equation 3.1 and 3.2

$$Sample\ Addition = \frac{V_B}{X_A/X_T} \qquad Equation\ 1$$

$$Supernatant\ Additon = V - Sample \qquad Equation\ 1$$

Where:

$$V_B = Batch\ volume\ X_A = Actual\ solids\ conc.\ X_T = Target\ solids\ conc.$$

A.6.3 Column testing procedure

The column testing protocol utilized was obtained from the CRTC protocol (Wahlberg et al., 2001). Five mixed liquor batches were prepared with TSS values ranged from 1000mg/L to 2000mg/L with a 250mg/L gap between each batch. Moderate mixing was necessary in order to produce a homogenous mixture without breaking the floc in the sample. The 1 rpm rake and timer was immediately started as soon as the settling columns were filled. After the timer was started, the interface height was recorded as a function of time, and at least six data points were collected during the period of constant settling velocity. Once the settling velocity begins to slow down, the recording of the interface height was then paused, after 30 minutes had elapsed from the beginning of the experiment, a final interface height was recorded. After this, the settling column is discharged and another batch of mixed liquor is funneled into the settling columns and the same procedure is repeated. This test was carried out until all five mixed liquor batches were fed into the settling columns.

A.6.4 State point analysis

The region of constant velocity was visually determined from a plot of the interface height (or depth) as a function of time for each batch. Then, the linear region, defined as the settling velocity (v_s), can be determined. (Wahlberg et al., 2001). Values of v_s from each graph are then plotted as a function of the solids concentration; after which the v_0 and r_h from the curve (Equation 3) of best fit is determined using least squares arithmetic.

$$v_s = v_0 e^{-r_h X} \qquad Equation\ 3$$

Where:

$$v_s = Settling\ velocity \quad v_0 = Settling\ velocity\ constant$$

$$r_h = Hindered\ settling\ constant\ \ X = Solids\ conc.$$

At this juncture, one can then generate the settling flux curve by plotting Equation 4, allowing X to vary from 0 to approximately 10000mg/L

$$Gravitational\ Soolids\ Flux\ (G) = Xv_s \qquad Equation\ 4$$

The flux curves are computed over several trials, and compared in order to determine whether the settling in the clarifier is worse or better in terms of the TSS load the clarifier can operate at well before failing. A higher peak in the graph corresponds to better settling.

A.6.5 sSVI Procedure

The sSVI is determined by measuring the height of the settled sludge of an unaltered mixed liquor sample that has been pumped into the settling columns after thirty minutes has elapsed, and calculating the volume of sludge in the reactor (V_S) (Wahlberg et al., 2001). Equation 5 is used to calculate sSVI using the actual solids concentration (TSS) from lab analysis.

$$\frac{V_S/V_T}{X} \qquad Equation\ 5$$

A.6.6 STC Procedure

In order to generate the STC, a series of dilutions were made of mixed liquor collected from locations A and B (I missed it, where are locations A and B?). Some samples were spiked with 420mg/L of NaOH. (Why this concentration? How did you decide how much you needed to influence settling?) The dilutions were produced between the ranges of 1200mg/L and 300mg/L with a gap of 100mg/L and a volume of 2 liters. Each dilution is placed in a 2 liter jar, stirred, observed visually and filmed. Then the TSS is measured and graphed for each trial, illustrating the point at which the transition occurs in relation to TSS concentration. The videos are then analyzed with the commercial MATLAB program. STCD is then used to determine STC, which is plotted on a graph in order to show when the transition occurs in relation to the TSS.

A.6.7 MATLAB analysis and logic

The MATLAB code analyses the video based on RGB images analyzed by indexed green, red and blue into a matrix. The programmer then validates the data with the observed images as a function of the height of each jar in pixels. Figure A.3 illustrates this procedure. These images are then constructed into a movie that transposes the time that has elapsed in the video in a graph, and which can be analyzed by the programmer for the slope, if any.

A.7 SUMMARY AND CONCLUSION

This study explored a new MLSS settleability measurement potentially useful in determining secondary clarifier design and operating limitations. STC can be determined by making MLSS dilutions that are then analyzed by STCD in order to determine concentrations for which neither Type II nor Type II settling clearly occurs. This STC was observed to typically occur over a range of 100mg/L,

STC was determined to relate well to traditional measurements of sludge settling characteristics, even though it measures a different physical phenomena. Moreover, the hypothesis that the higher STC will provide better settling was evidenced by the addition of sodium based chemicals (NaOH) that are occasionally added to wastewater in plants for alkalinity and has been proven in high doses to cause an imbalance in the monovalent and divalent ion ratio which will produce bad settling. Figure A.5 shows that the NaOH addition decreased the STC and thus validated the theory that a high STC can be correlated to “good” settleability.

A correlation exists between flux curve parameters sSVI, and STC. The three methodologies corresponded to STC in terms of determining when settleability is “good” and this was further validated when the ISV measured by the plant also corresponded. However, sSVI did not compare with the other two methodologies or the ISV data measured by Blue Plains in terms of “poor” settleability, but flux curve parameters and STC as well as the ISV data was consistent in identifying the trial with the poorest settling. This test demonstrates that STC may provide useful information to support traditional settling parameters.

The RGB analysis of a video depicting settling was successful in accurately determining the STC in the jars since it reduced human error and provided consistent results. The STCD also proved useful in detecting the interface when the supernatant did not have a clear consistency which led the naked eye to make rough estimations about its location. Although traditional methods can be a time consuming process and do not always provide an accurate representation of the occurrences within a clarifier, State point analysis, depending on the number of settling column being operated, can take up to 4 hours. On the other hand, STC consumes much less time than either of these methods. For example, seven dilutions can be made in twenty minutes and the settling process of a two liter dilution takes an average of three minutes to elapse. Moreover, the equipment needed to conduct the experiment is also cheap compared to equipment for state point analysis since it only requires a digital camera, computer, and jars.

For the most part, STC has proven to be a viable pathway for the modeling of one dimensional settling, which could support existing techniques which rely on the Vesilind equation. This methodology could also shed light on the boundary layer conditions when placed into a two dimensional method. In addition, STC can provide further insight as to when clarifiers will fail since the STC dropping will result in hindered settling regions extending to flocculent settling causing higher TSS in the effluent.

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A.9 FIGURES

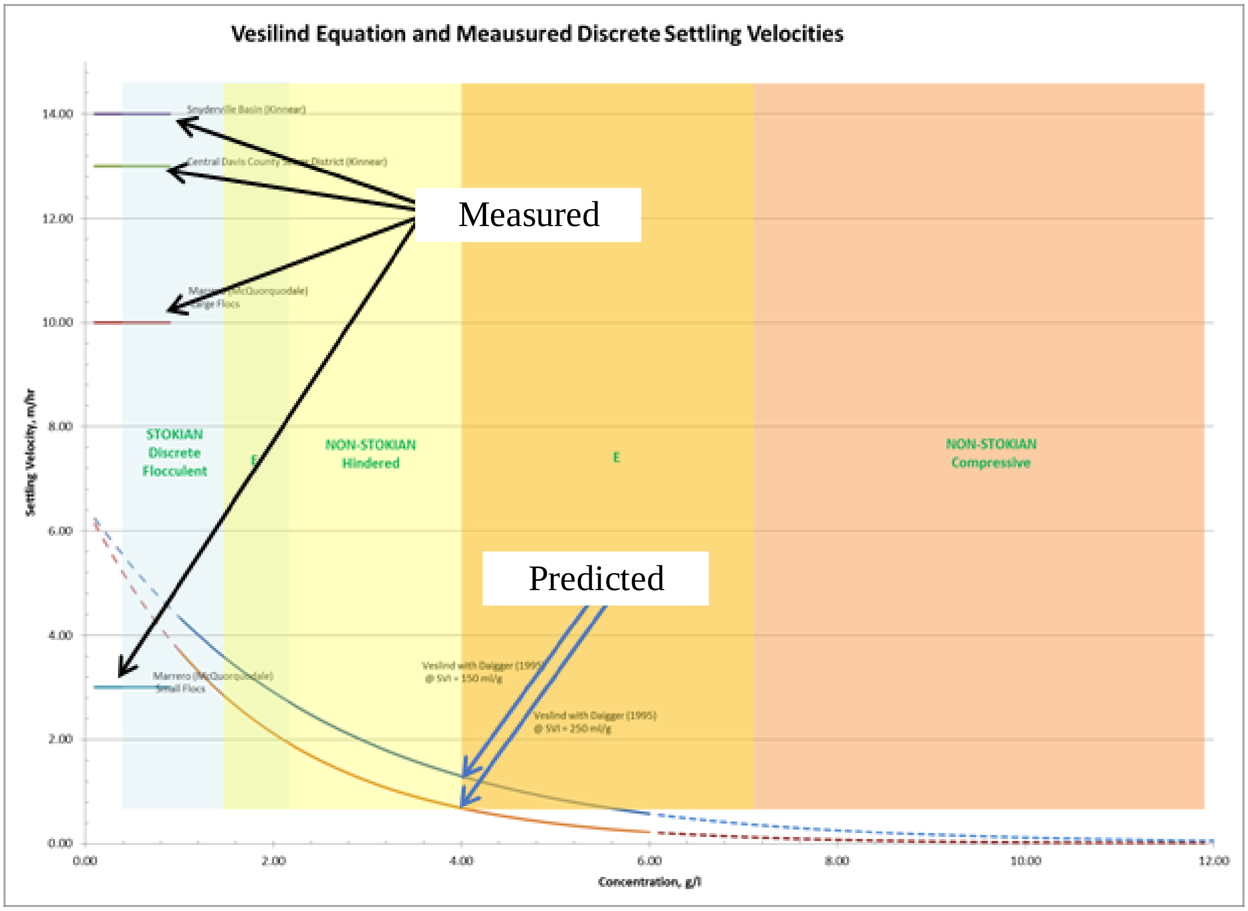


Figure A.1: Vesilind Equation and Measured Discrete Velocities depicted in this graph to illustrate the sharp discontinuity exhibited in settling velocities when settling type changes (after Kinnear 2010).

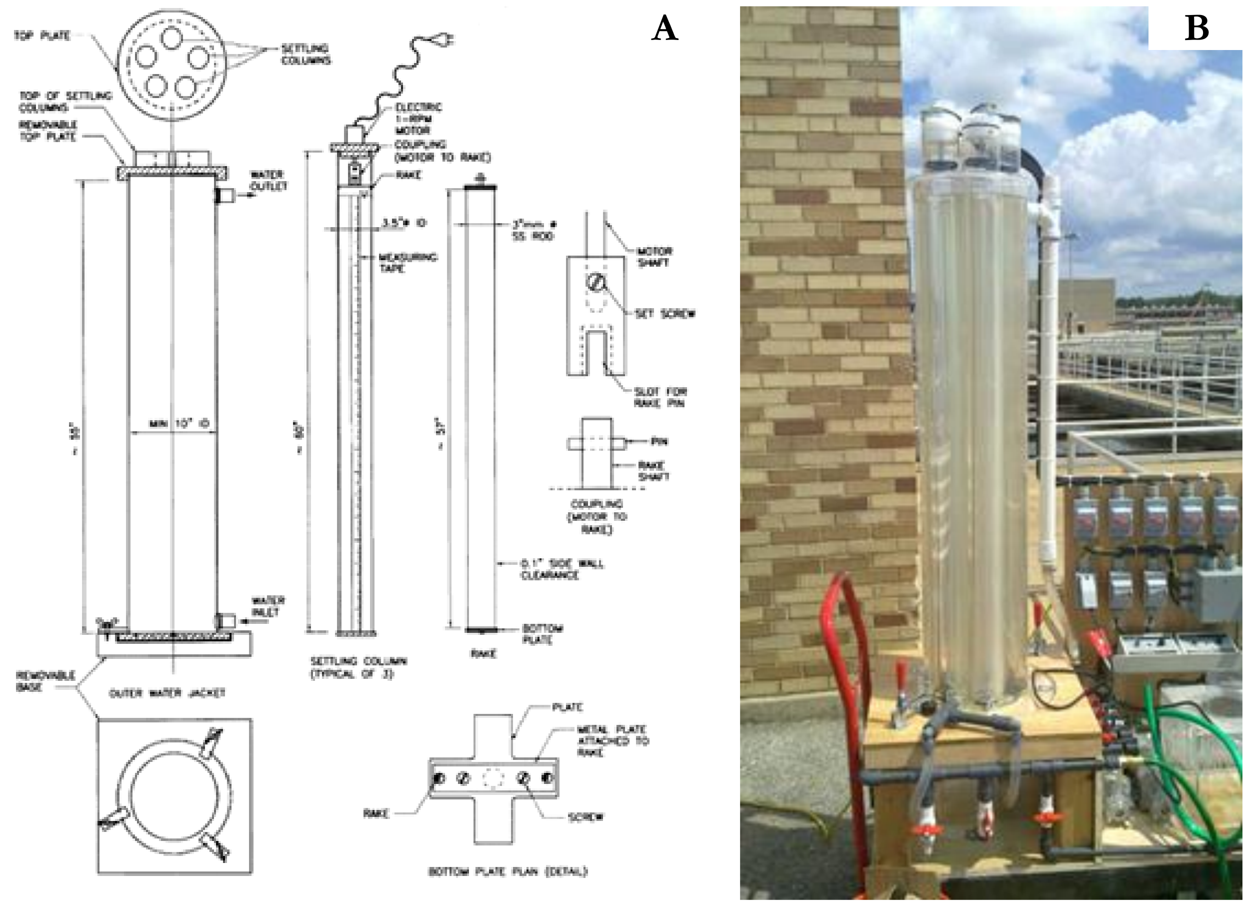


Figure A.2: Schematic of settling column fabricated by Blue Plains AWWTP (A) and picture of settling column that was fabricated by Blue Plains AWWTP (B).

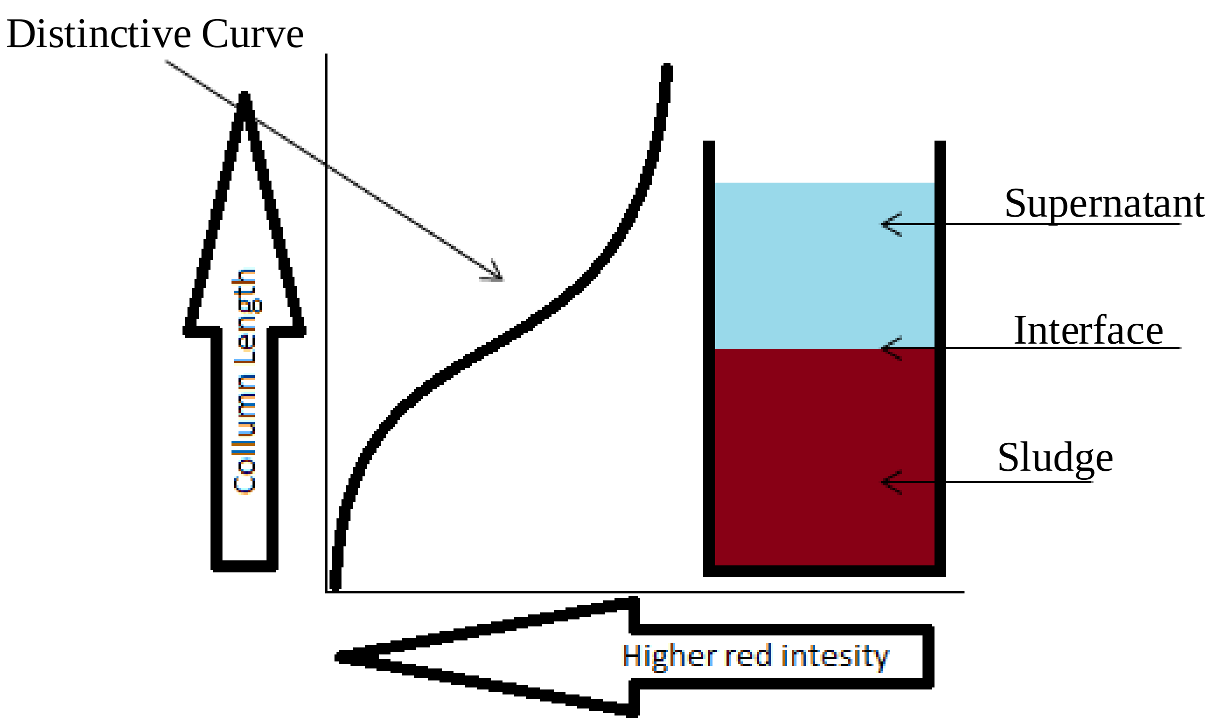


Figure A.3: STCD output curve for hindered settling, the arrows directs to increasing values

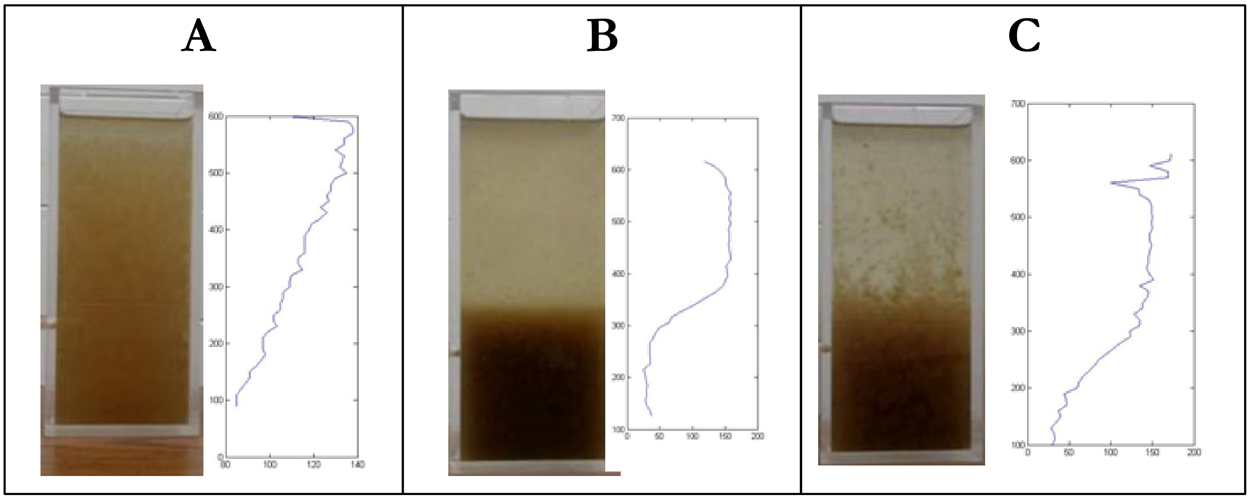


Figure A.4: STCD representation (A)flocculent settling, (B) hindered settling, and (C) transitional.

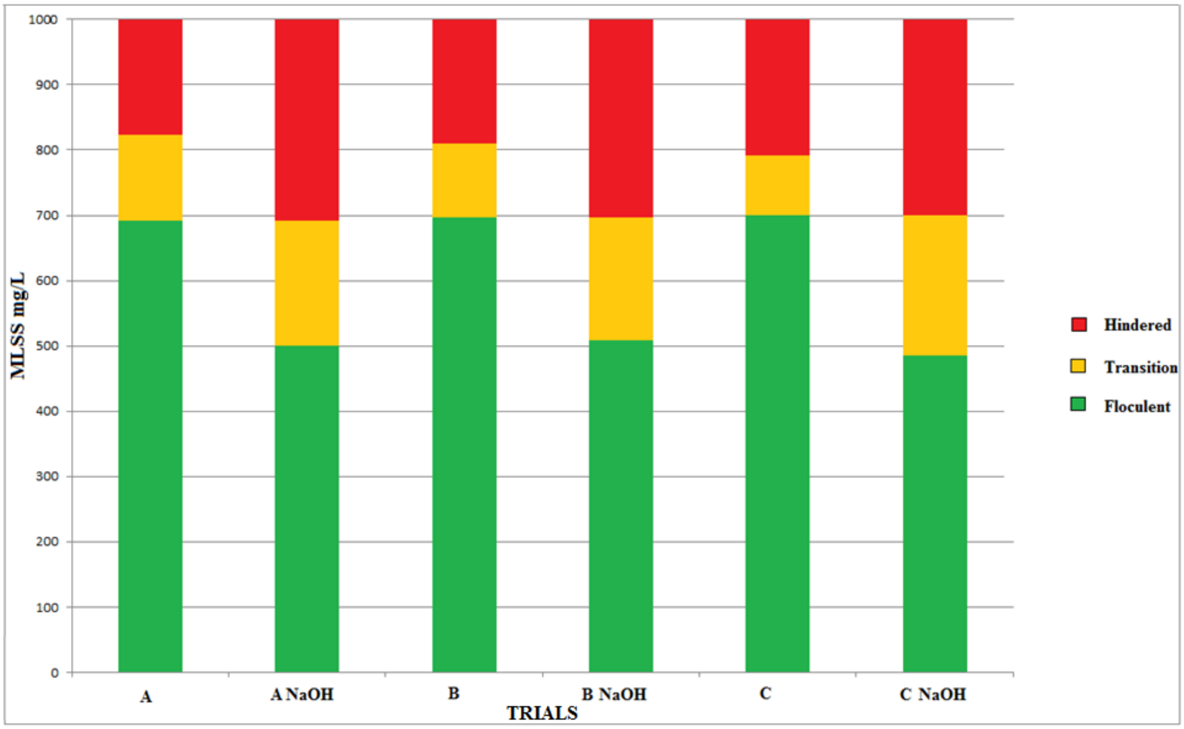
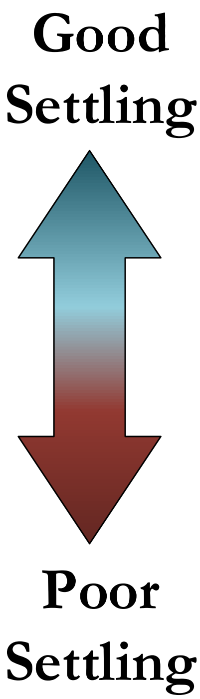


Figure A.5: Illustrates STCD values of the TSS range that transition occurs, which is displayed in yellow, trial numbers are labeled A-C, were NaOH addition has the NAOH suffix allocated to the specific trial.



Flux Curve	STC	sSVI
G	**G**	**G**
F	F	E
C	E	F
A	C	H
B	A	**D**
H	B	B
E	H	C
D	**D**	A

Figure A.6: Shows the correlation between Gravitational Flux curves, sSVI, and STC of all eight trials. The higher the trial is on the table the better settleabilty deemed by that test.

APPENDIX B

QUANTIFYING FLOCCULATION CAPACITY OF ACTIVATED SLUDGE

B.1 AUTHORS

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Published in Proceedings of the Water Environment Federation, WEFTEC 2012, New Orleans, Louisiana.

B.2 ABSTRACT

This study's focus was on quantifying the flocculation capacity of a sludge matrix. The Critical Settling Velocity (CSV) was used to classify the fractions of sludge that flocculated and settled. A test matrix was implemented whereby CSV and solids concentration were varied. Two sludge types were tested; secondary activated sludge from high rate activated reactors and nitrification/denitrification (BNR) sludge, both from Blue Plains advanced wastewater treatment plant. Two new settling characteristic measurements were obtained from the test matrix; Threshold of Flocculation (TOF), and 'α-values'. TOF is the transition concentration from discrete settling to flocculent settling, and was found to be directly related to flocculation capacity. Lower TOF corresponded to a high flocculation capacity, as flocs could be formed with fewer collisions. A solids removal rate was obtained from the test matrix, and followed an exponential trend. The curvature of the solids removal rate or 'α-value' also corresponded with flocculation. High curvature or α -value corresponded to high flocculation capacity and likewise for low α-values. BNR sludge had better operational performance with effluent quality of 7 mg TSS/L compared to 17 mg TSS/L for secondary systems. Comparison of BNR and secondary revealed lower TOF concentration of 250 and 400 mg TSS/L, and a higher α -value of 0.805 and 0.575, respectively. TOF and α-values were in agreement with operational performance. The shear tests were also performed, and showed the breakdown of well settling flocs into smaller fractions with increasing shear. Future work has to be done to relate these flocculation results to real-time full-scale flocculation behavior. This work will be used to develop effluent quality calibrations for settler models.

B.3 KEYWORDS

Flocculation, intrinsic properties, activated sludge,

B.4 NOMENCLATURE

BNR	Biological nutrient removal
CAS	Conventional activated sludge
CSV	Critical settling velocity
EPS	Extracellular polymeric substances
ISC	Intrinsic settling classes
ISV	Initial settling velocity
LOSS	Limit of stokesian settling
NSS	Non-settable solids
SOR	Surface overflow rate
SVD	Settling velocity distribution
SVI	Sludge volume index
t_F	Flocculation time
t_S	Settling time
t_T	Total time
TOF	Threshold of flocculation
α	Collision efficiency

B.5 INTRODUCTION

In wastewater, an optimally performing clarifier is required to meet the desired effluent solids concentration. Quantification of the maximal solids capacity, a crucial operational parameter, can assist in optimization of a clarifier. However, determination of secondary clarifiers' solids capacity has largely been ignored. This is in no part due to its significance, as clarification serves as the treatment limiting step in conventional activated sludge (CAS) systems (Ekama et al., 2002). Utilities design large clarifiers to increase the safety factor while disregarding the optimum and economical operational requirements. The presence of large clarifiers stemmed the need to understand fundamental mechanisms of solids separation in flocculent systems. Operators without tools of a reliable metric to gauge sludge settling performance rely on a reverse feedback loop to mitigate problems as they arise in secondary clarifier operations. Given the breath of limitation and understanding of CAS settling properties, we are often left with the following: The search for a reliable and simple metric for activated sludge quality based on fundamental understanding of flocculent settling properties.

A previous study showed that the Limits of Stokesian Settling (LOSS) of sludge correlate to settling velocity of sludge (Mancell-Egala et al., 2011). LOSS is the settling regime change occurring from stokesian to non-stokesian, with increasing LOSS concentrations corresponding to a larger percentage of the SVD in the high settling velocity range. Investigating stokesian settling and transitioning of stokesian settling types at varying solids concentration might indicate a relationship to the settling characteristics of activated sludge as well.

Intrinsic Settling Classes (ISC) studies (Mancell-Egala et al., 2014) was the next step, and characterized settling under purely discrete settling regime. Stokesian settling, which characterizes most of settling in a clarifier is separated into discrete and flocculent settling. Discrete settling for activated sludge systems occurs at very low MLSS concentrations (<400mg/L) when solids are limiting and macro flocs are not formed. ISC test determined solid classes based on critical settling velocity (CSV) in a discrete settling environment. The rational was that limiting solids concentration would retard bio-flocculation thereby increasing the disparity of settling velocities between granules and flocs. Three CSV classes were defined; granules, aggregates, and flocs. CSV of 10 m/h (Winkler et al., 2013) was determined as a characteristic for aerobic granules and the latter was for example used by Liu et al. (2005) to design reactors that select for granulation. In-between 10 and 1.5 m/h were classified as aggregates, these particles required no flocculation to settle out in an operational clarifier but lacked the superior settling properties of granules. Particles with a critical settling velocity below 1.5 m/h would not escape an operational clarifier and therefore were classified as flocs.

However, ISC alone cannot indicate how well the flocculant fraction will settle out and gives no prediction of the effluent quality. Therefore a new study was started to quantify flocculation. Flocculation is dependent on two functions: collision efficiency and number of collisions (Melik and Fogler, 1984). Collision efficiency is a successful attachment of two flocs through collision while the number of collisions is directly related to solids concentration and shear. According to current solid flux theory based on Kynch (1952) assumption, and bio-flocculation studies (Serra et al., 1996) there should be a solids concentration linked to the minimum collision rate to form larger aggregates. The transition concentration from discrete settling to flocculent settling might be directly related to collision efficiency. As the solids concentration decreases, it passes a certain threshold ensuing that flocculation time (tf) is equal to or greater than the settling time (ts). This threshold is defined as the Threshold of Flocculation (TOF) and characterizes the collision efficiency of the sludge.

Additionally, environment also plays critical role in floc formation. Conventional activated sludge (CAS) systems couple reactors and clarifiers. Sludge experiences different environments before it reaches the clarifier. Figure B.1 details this concept. In the reactor, mixing, dissolved oxygen among several variables determines intrinsic properties of solids in the system. Sludge is then transported within a channel at high shear environment from reactors to clarifiers that are a low shear environment. Feed well in clarifiers promote shear flocculation and enable a density current to direct solids to the bottom of the clarifier. Gravitational settling can occur when particles have cleared the feed well. Computational fluid dynamic studies on clarifiers have been deduced to underline settling mechanics at play in clarifiers. (Wahlberg et al., 2001). The goal of this study is to conduct testing matrixes and explore the mechanistic capabilities of the methods designed in previously (Mancell-Egala et al., 2014) to provide insight to flocculation capacity under varying environments and time frames. To explore the full scope of the environments flocs travel through, the study was divided into two subcategories; gravitational and shear studies.

B.6 MATERIALS AND METHODS

Two types of sludge with unique settling properties were selected to show varying degrees in flocculation. Secondary and BNR are two different types of purely flocculant systems. Secondary systems have a lower Surface Overflow Rate (SOR), but poorer effluent quality (Table 1). The latter suggests secondary sludge has either or both coagulation and flocculation limitation. Additionally it can be hypothesized that a high SOR selects for faster settling solids signifying less settling limitation for BNR compared to secondary sludge.

B.6.1 Gravitational Studies

The hypotheses for gravitational studies is as follows;

- Solids concentration directly correlates to generation of larger classes of solids.
- For two types of sludge with same solids concentration, number of solids collisions will remain the same whiles collision efficiency used to define Intrinsic Settling Classes (ISC) will differ.

With these two hypotheses, each sludge matrix undergoes LOSS determination to provide the range of stokian settling solids concentration. Depending on the LOSS of the sludge dilutions are made regressing from that specific value. Each dilution then undergoes four Critical Settling Velocities (CSV) executed at 0.6, 1, 1.5, and 3m/h. Higher CSVs were neglected as those refer to granules. This is done to highlight changes in CSV fractions as the level of flocculation changes. Protocols to achieve fractions of sludge that settles at different CSV followed previous work (Mancell-Egala et al., 2014). Also, solids removal rate can be explored under varying CSV. This rate is calculated by taking the slopes values after TOF at different CSVs and plotting it (figure B.2A &B).

The second hypothesis is the basis of TOF, TOF is quantified by the deflection point (triangle) of the curve in Figure B.2 with a minimum differential of 10%. Figure B.2 shows TOF being a function of batch settling time (t_T) by depicting four possible scenarios;

- **$t_T < t_f$:** all solids remain in suspension no matter the solids concentration due to a lack of time to flocculate
- **$t_f < t_T << (t_f + t_s)$:** solids have enough time to flocculate, but not enough time to settle resulting in elevated solids in suspension even at optimal solids concentration
- **$t_T \geq (t_f + t_s)$:** solids have enough time to flocculate and settle at optimum solids concentration resulting in discernment of TOF (triangle)
- **$t_T >> (t_f + t_s)$:** batch settling time is large enough for non-flocculating aggregates (or granules) to settle, resulting in an inability to locate TOF

Figure B.2 also shows the importance of tT in determining TOF. It is essential that the correct tT is used to localize flocculation limitation. For full scale settling tanks, tT represents hydraulic residence time (HRT). Operators convert clarifier HRT into SOR, similarly Perric et al., (2008) mathematically related tT to SOR. Failure of a secondary clarifier typically begins at SOR above 1.7 m/hr (1000 gpd/ft2) according to various stress test studies (Daigger, 1995; Ozinsky and Ekama, 1995). Therefore, a SOR of 1.5 m/h or total settling time of 2 minutes was used as a benchmark for discerning TOF as it illustrates the flocs that would usually settle in a clarifier under operational conditions. Also the slightly lower SOR allows a safety factor of 15% to account for human error.

Additionally, no shear is applied during this test to avoid ortho-kinetic flocculation. Gravitational flocculation is weaker than ortho-kinetic flocculation with shear as a driving force for flocculation. By using only gravity the number of collisions is not artificially increased for lower solids concentration, whereby leaving collision efficiency to be characterized by the sludge properties only. TOF is hypothesized to occur at lower concentrations for systems that are not flocculation limited. The rationale being; if flocculation still occurs in an environment with reduced collisions, collision efficiency must be high.

B.6.2 Shear Studies

The second study, involved investigating impact of shear on critical settling classes, classes changed for a particular sludge based on shear. Unlike the gravitational experiment that has only two variables; settling time, and solids concentrations, shear experiments have an additional variable; shear gradient. The study had three hypotheses;

- Increasing the shear gradient will increase collisions, but will decrease efficiency of collision
- Good compared to poor flocculant sludge will be affected less by shear
- Sludge matrices that formed faster settling flocs compared to sludge with slow flocs will reach their minimum solids in supernatant at lower initial solids concentration.

The shear studies had several additional steps. These steps involved placing a mixer and inducing shear for a specified time frame before determining CSV fractions. Shear is calculated based on revolutions per minute (rpm) of the propellers. Furukawa et al., (2012) calculated the power consumption based on physical parameters of the wastewater matrix, geometry of propellers, geometry of settling column, and number of propellers. Shear from the mixer can be calculated from the power consumption (Nopens, 2002). A detailed protocol can be found in Mancell-Egala et al., (in preparation).

B.7 RESULTS AND DISCUSSION

B.7.1 Gravitational Studies

Results from the gravitational studies can be found in Mancell-Egala et al., (in preparation). Notable findings from the gravitational studies was that ISC for both secondary and BNR indicated a floc fraction above 98%, therefore bio-flocculation was the main mechanism for both systems. The CSV method over varied solids concentration showed BNR compared to secondary sludge to have less TSS in supernatant at varying solids concentration and time. Additionally, BNR compared to secondary had a significantly lower TOF, reported at 250 and 400 mg TSS/L, respectively. Both these findings show that BNR has a higher collision efficiency and therefore a higher flocculation capacity.

The solids removal rate was calculated as the highest removal of solids per time and was plotted against CSV. The removal rates for both BNR and secondary sludge were a good (above 95%) fit for an exponential curve, and their corresponding parameters have been tabulated in table 2. The curvature of the exponent is governed by the “α” value, with high k resulting in a high curvature and likewise for low α. Results show that α value corresponds with flocculation. BNR systems had much higher α values when compared to secondary systems. BNR and secondary being ideal representations of a good flocculant and poor flocculant system had α values of 0.805 and 0.575, respectively. The α value appeared sensitive, enough to differentiate significantly (29%) the flocculation capacity of both types of sludge.

According to preliminary data from Mancell-Egala, 2015 Ph.D. proposal, TOF values are unobtainable at high levels of granulation. In this regard α-values can be used to characterize flocculation in hybrid systems. Figure B.4 illustrates two key findings, more detail is in Mancell-Egala., (in preparation). The concept graph plots three different systems, the first and second system is akin to BNR and secondary systems, respectively, with high curvature relating to high flocculation capacity. The third line shows no curvature, as there is no flocculation, but also shows the line starting and ending at higher points on the y-axis. The shift in the line along the y-axis correlates to settling velocity, with a shift in the positive direction on the y-axis correlating to higher settling velocity.

B.7.2 Shear Studies

Results from the shear studies can be found in Mancell-Egala et al., (in preparation). A notable finding in the study was that the lowest shear resulted in the least solids in supernatant both for secondary and BNR. There is a clear distinction between operation shear and transport shear. Operation shear is the shear present in the reactor, this shear influences the sludge matrix present, i.e. higher shear stresses the microbiological community thereby increasing EPS production (Spicer, 1978). Transport shear dictates flocs formed by the sludge matrix present. This shear experiment explores the latter shear, furthermore, literature states a maximum transfer shear of 60 s-1. This is in accordance with the results as only shears above 60s-1 had a NSS fraction greater than when no shear was present.

Moreover, BNR's supernatant quality was less affected by increases in shear with a total range of 12% compared to 52% for secondary. BNR on the whole was proven to be better than secondary at flocculating as shown by effluent quality, SVI, ISV, and gravitational experiments. With that knowledge we can conclude that sludge with better flocculation capacity is less affected by shear. The information provided by the shear experiment can be beneficial in modeling effluent quality during wet weather scenarios or other high flow events, when the transport shear increases.

B.8 CONCLUSION

Results from the study has shown that the test matrix provides insight into flocculation capacity of sludge which correlates well with the conclusions found using TOF. Moreover, the matrix work showed potential to quantify flocculation behavior of the flocculant fraction (α-values). The shear tests showed the breakdown of well settling flocs into smaller fractions with increasing shear. Future work has to be done to relate these flocculation results to real-time full-scale flocculation behavior. This work will be used to develop effluent quality calibrations for settler models.

B.9 REFERENCES

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B.10 TABLES

Table B.1: Location and description of both sludge types used in the experiment

Name	Location/description	ISV (m/h)	SVI (mL/g TSS)	Effluent TSS (mg TSS/L)	SOR (m/h)
Secondary	Secondary mixed liquor from Blue Plains AWWTP, a predominately flocculent system	3.0	116	17	1
BNR	Nitrification mixed liquor from Blue Plains AWWTP, a predominately flocculent system	4.0	88	7	1.1

Table B.2: Limit of Stokesian Setting, Threshold of Flocculation, and Exponential fit parameters of settling rates for Secondary and BNR sludge different types of sludge (Mancell-Egala et al., in preparation)

Sludge Type	LOSS (mg TSS/L)	TOF (mg TSS/L)	α value	Regression fit (r^2)
Secondary	1000	400	0.575	0.96
BNR	1200	250	0.805	0.99

B.11 FIGURES

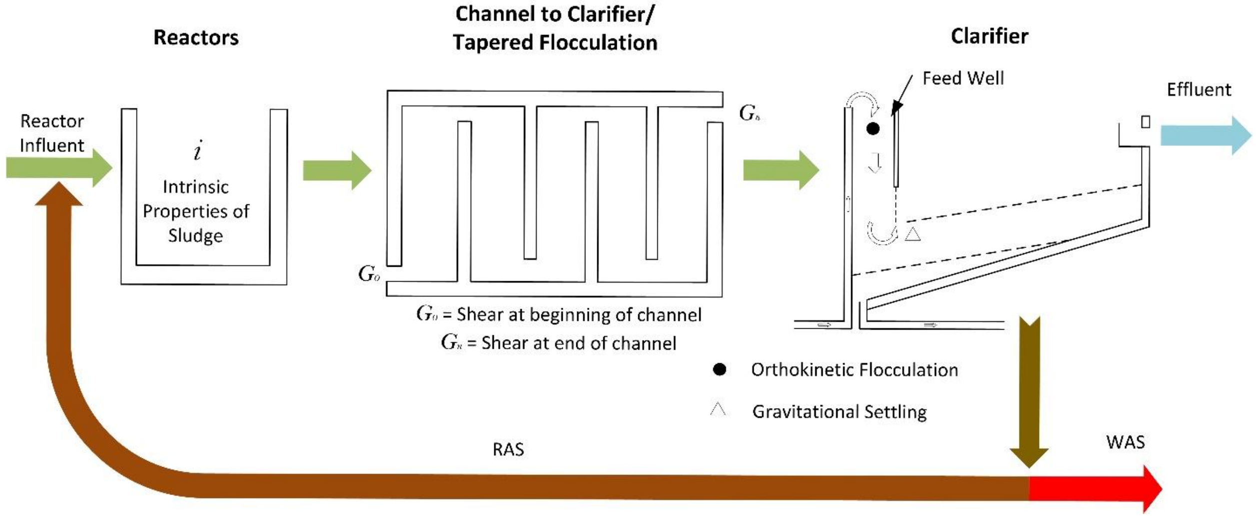


Figure B.1: Flocculation mechanics and location in secondary/advanced treatment with indications of different environment, sludge characteristics and settling mechanisms affecting flocculation.

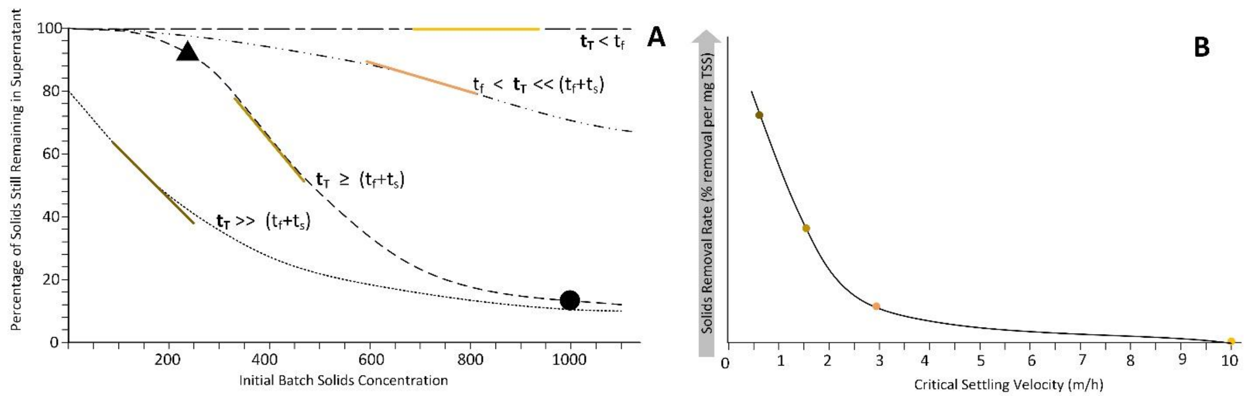


Figure B.2: (A) Concept of solids that remain in supernatant at varied initial solids concentration. Varying CSV or settling time (t_T) for a particular type of flocculent sludge results in four possible scenarios (represented as dotted lines). Triangle and circle highlights Threshold of Flocculation (TOF) and Limits of Stokesian Settling (LOSS), respectively. Solids removal rate can be calculated by taking the slope of solids removal at different settling times. (B) Concept of solids removal rate acquired from figure 2A.

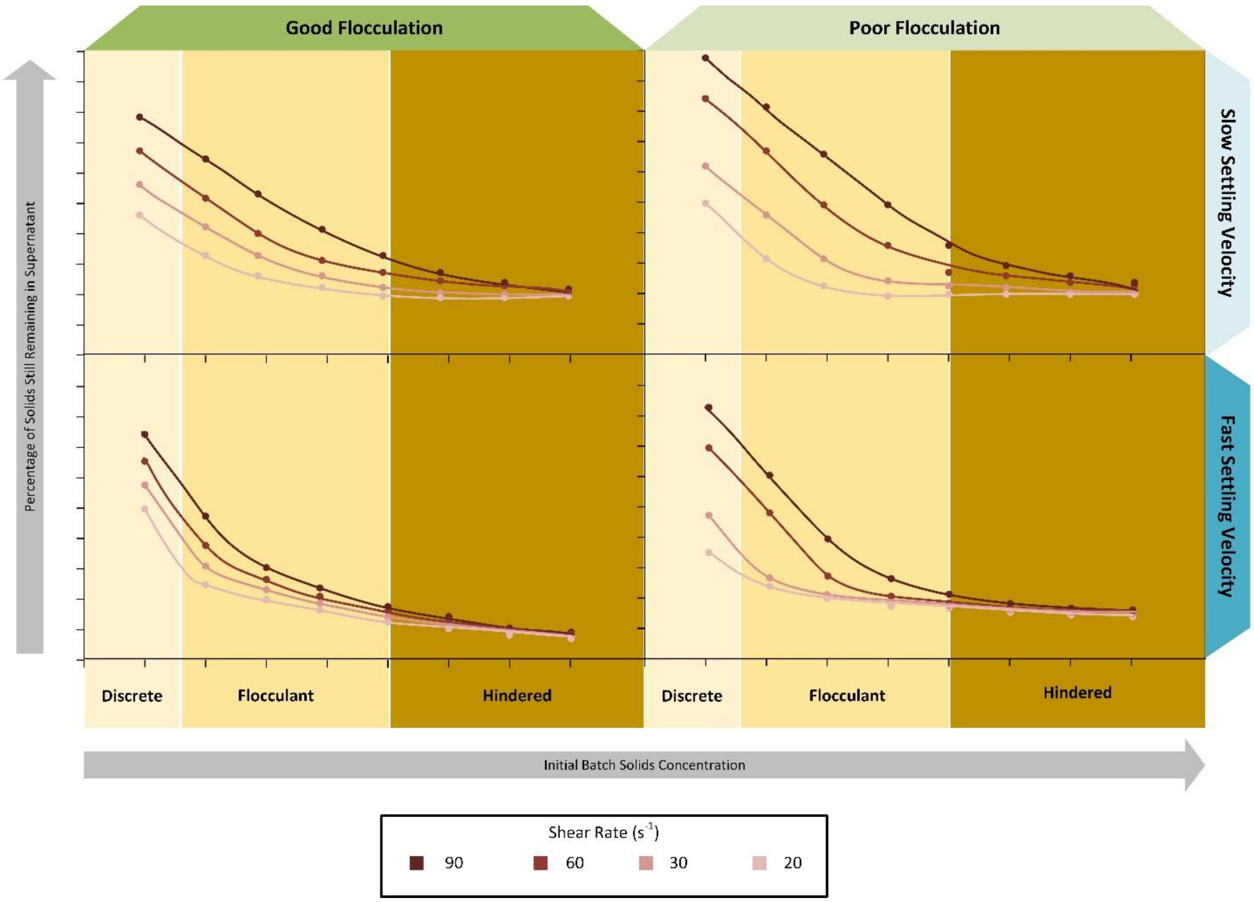


Figure B.3: Concept of how Critical Settling Velocity (CSV) overall – Intrinsic Settling Classes (ISC) just for discrete characterizes both flocculation and settling velocity.

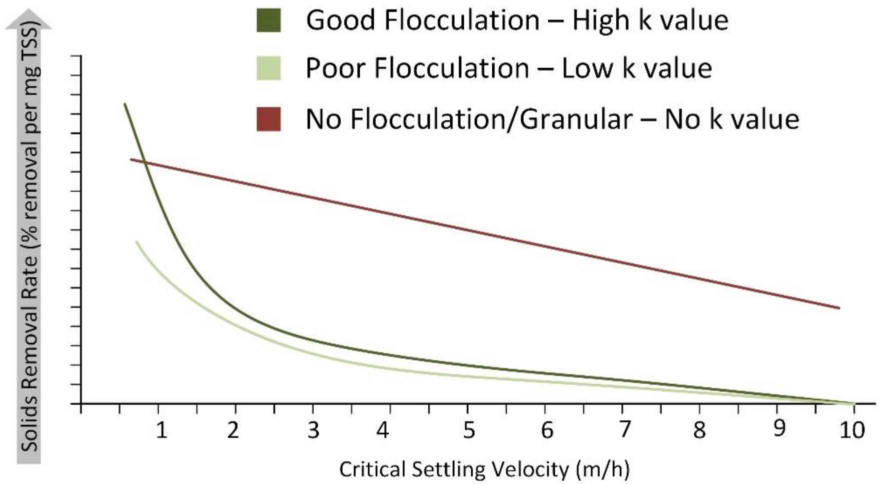


Figure B.4: Concept of curvature being correlated to flocculation capacity of a sludge type

APPENDIX C

SUPPLEMENTAL DATA FOR CHAPTER THREE

C.1 AUTHORS

William A.S.K. Mancell-Egala, David J. Kinnear, Kimberly L. Jones, Haydee De Clippeleir, Imre Takacs and Sudhir N. Murthy

C.2 TABLES

Table C.1: settling characteristic parameters from Vesilind model and Takács (1991) model. Vesilind parameters were used to calculate gravity curves in figure 6.

Operational	Vesilind model		Takács (1991) model		
Day	V_0 (m/hr)	r_h	V_o (m/hr)	r_h	r_p
2	11.597	0.732	-	-	-
4	16.954	0.804	31.5449	1.0338	2.0531
5	18.469	0.802	108.9655	1.2985	1.6088
15	11.748	0.699	-	-	-
17	14.758	0.972	95.1511	1.6520	2.0709
23	13.225	1.008	33.3445	1.5036	2.6086
32	10.594	0.898	10.6742	0.8207	2.1028
39	6.967	0.619	8.8131	0.7312	2.6943
44	9.378	0.742	32.2099	1.1424	1.7012
46	7.001	0.533	45.4862	1.2930	1.7200
51	15.822	1.198	38.0004	1.5484	2.5417
53	17.869	1.104	-	-	-
58	18.004	0.816	66.2489	1.1073	1.7238
60	22.260	0.740	101.7789	1.7833	2.1765

V_s: Maximum settling velocity (Vesilind)
k: Vesilind settling constant
V_o: Maximum theoretical settling velocity (Takács)
r_h: Settling parameter associated with hindered settling component of settling velocity equation
r_p: Settling parameter associated with the low concentration and slowly settling component of the suspension

C.3 FIGURES

Figure C.1: Pictures Pilot Runs form and Central Davis County Sewer District. From the picture discrete, flocculent, and compression zones are spotted clearly with no hindered zone.

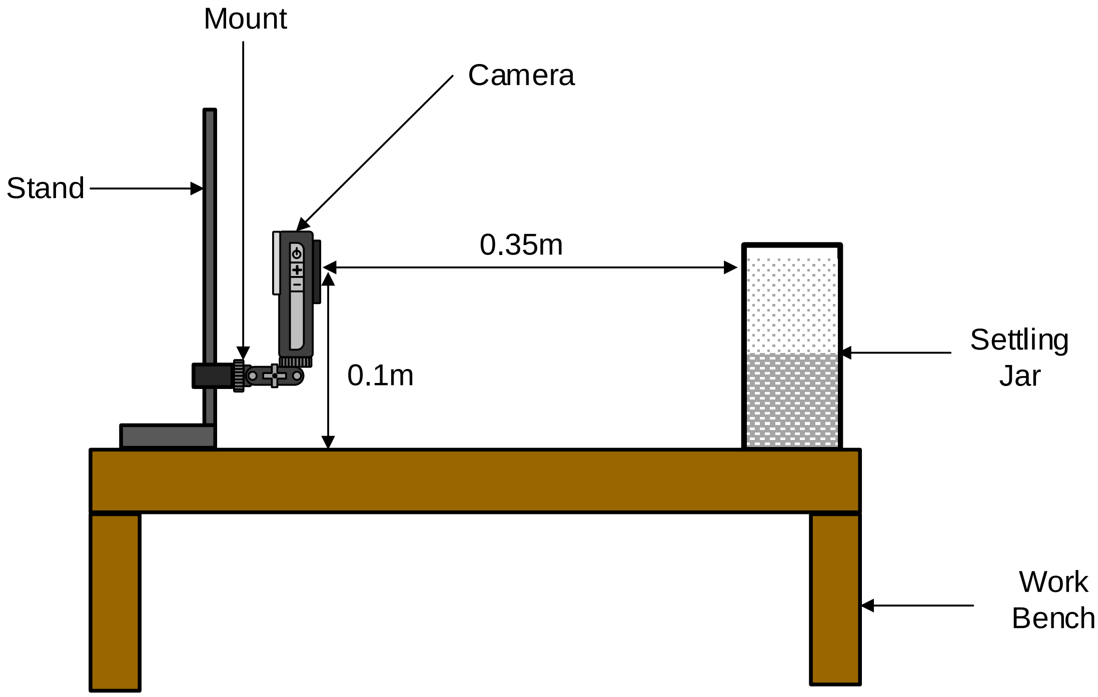


Figure C.2: Detailed schematic of image analytical setup

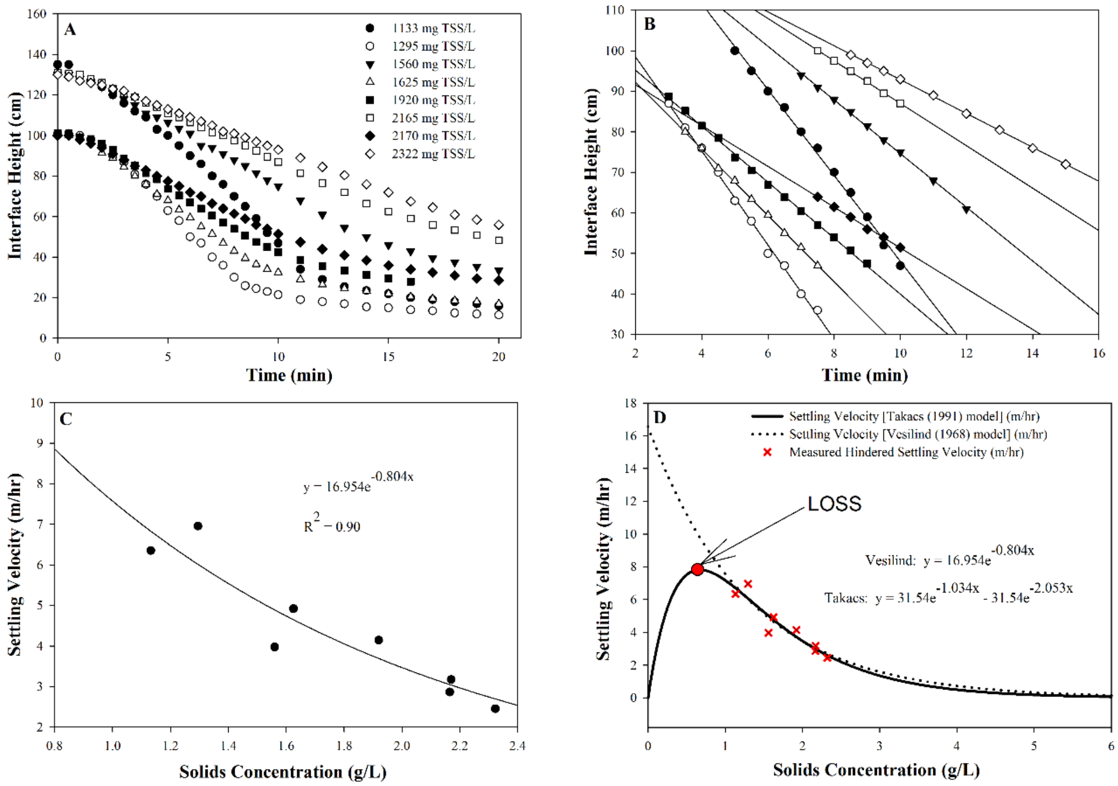


Figure C.3: (A) Column settling curves for different initial solids concentrations with secondary sludge from Blue Plains AWWTP, (B) Slopes of the linear part of the column settling curves, (C) Plots of linear slope vs initial solids concentrations and the exponential decay fit line with equation, (D) Vesilind (1968) and Takacs (1991) settling velocity model (settling velocity data from S2A were used to generate model) and LOSS value indication at maximum peak of Takacs model.

Figure C.4: Pictures of secondary channel where samples were taken, (A) shows sludge with large production of foam and (B) shows the same channel after 3 days of high wasting.

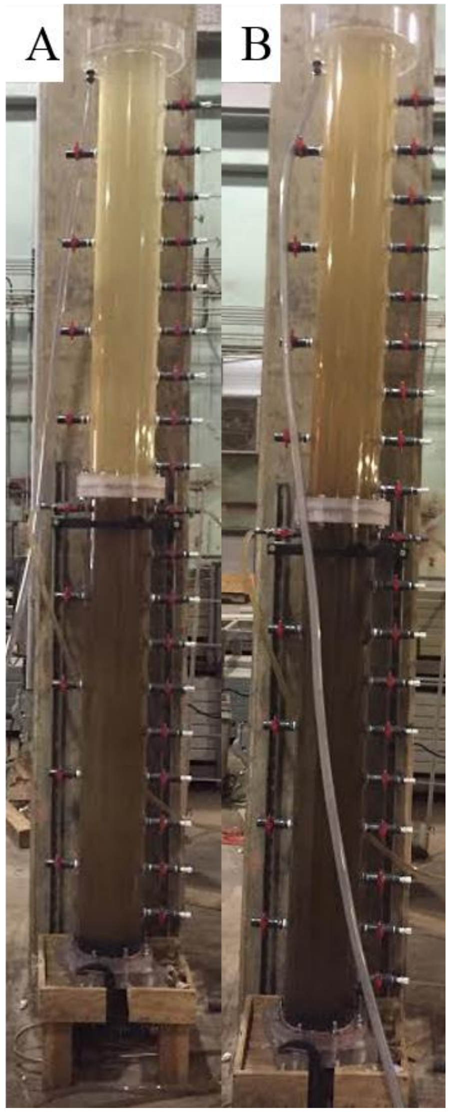


Figure C.5: Pictures of pilot clarifier when it reached steady state at (A) Scenario 1, and (B) Scenario 2

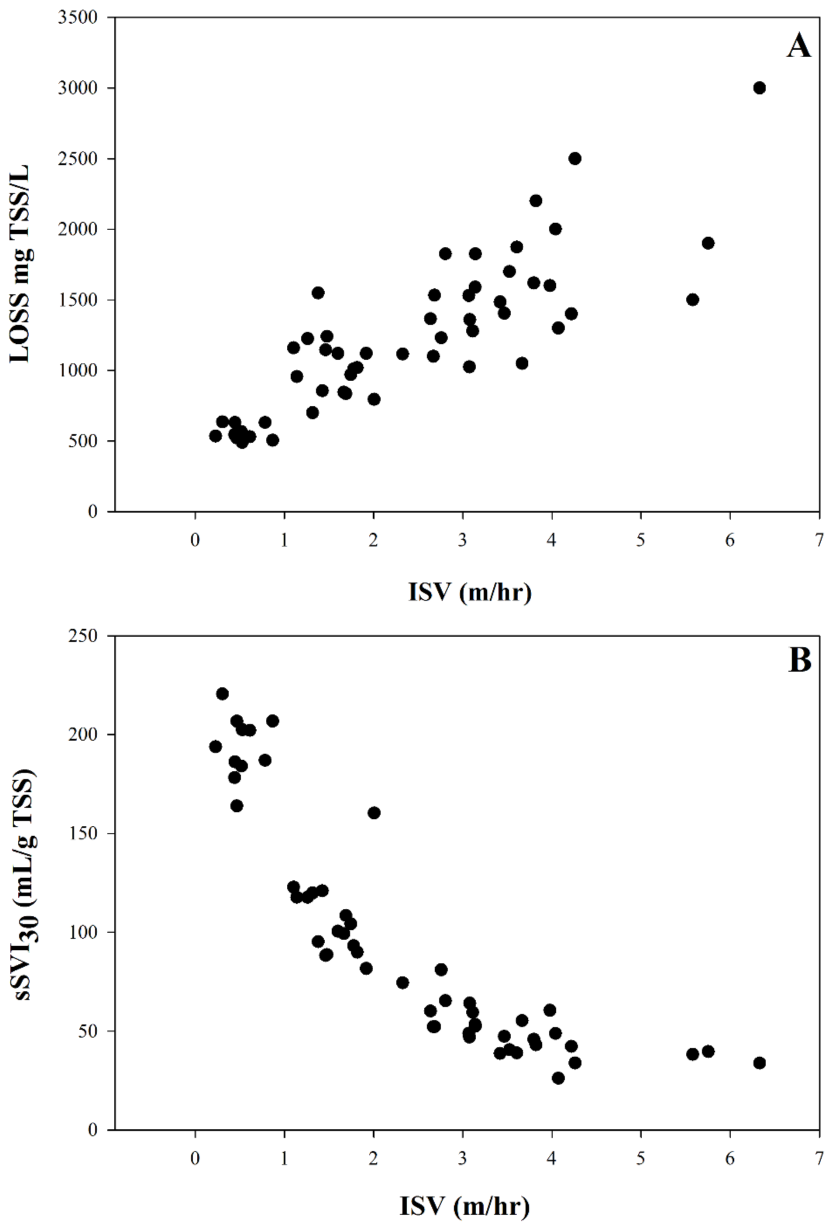


Figure C.6: 28 weeks' worth of raw data showing correlation between ISV and (A) LOSS and (B) LOSS

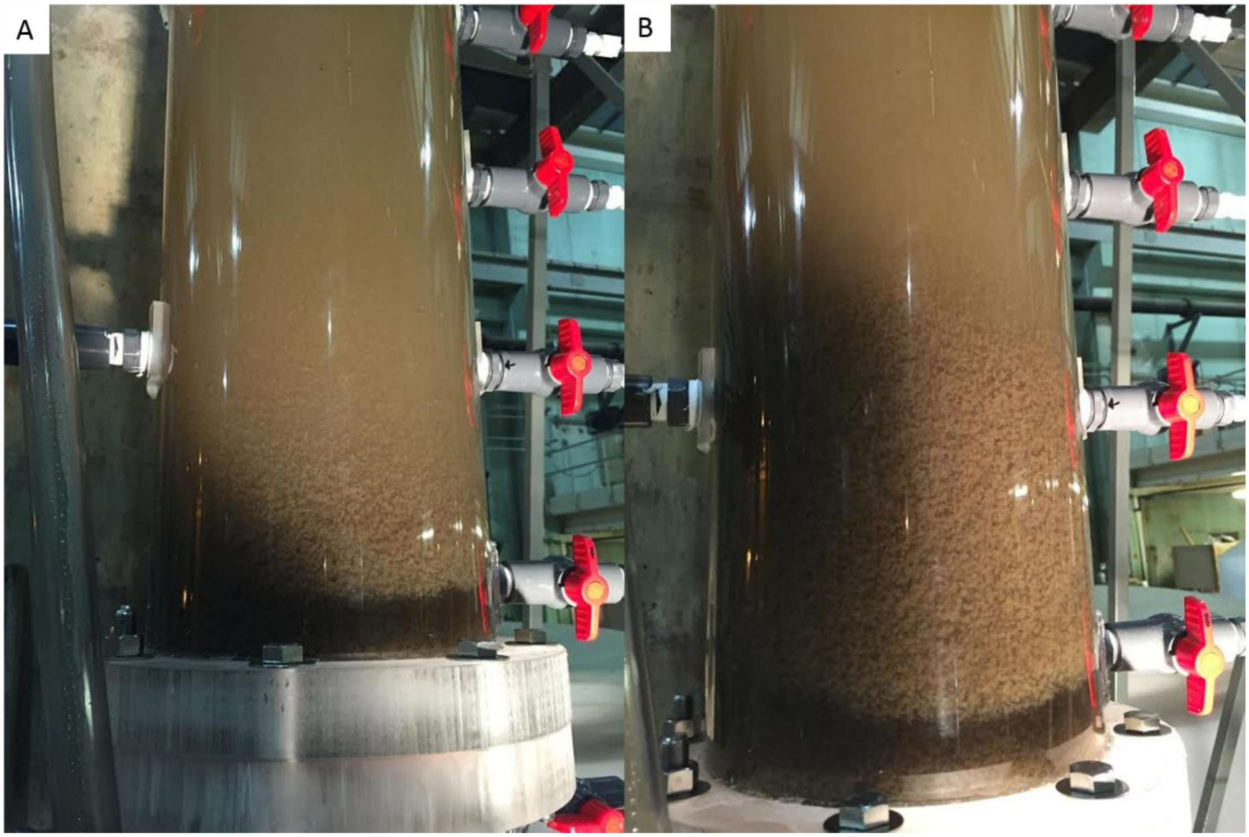


Figure C.7: Pictures of the pilot clarifier during the failure scenario, a hindered layer is seen rising above the feed layer at (A) 20 and (B) 25 minutes into the scenario.

APPENDIX D

SUPPLEMENTAL DATA FOR CHAPTER FOUR

D.1 AUTHORS

William A.S.K. Mancell-Egala, Haydee De Clippeleir, Chungyung Su, Imre Takacs, John T. Novak, and Sudhir N. Murthy

D.2 FIGURES

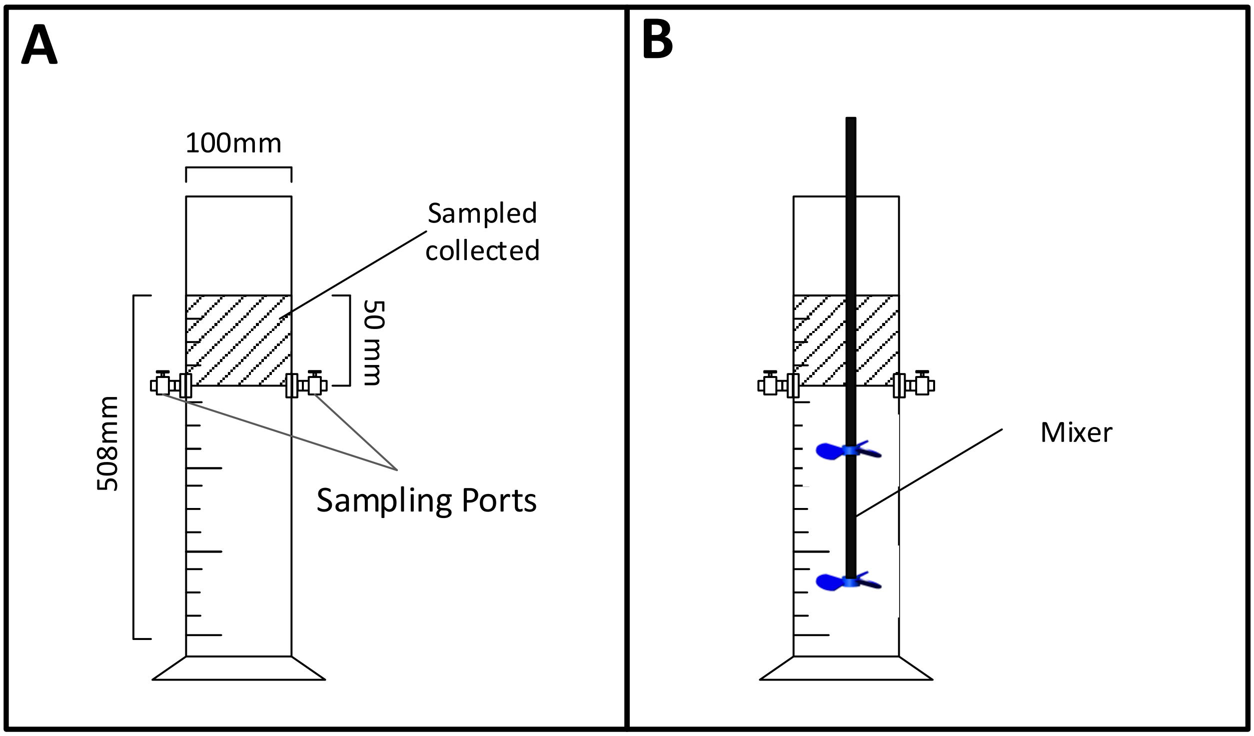


Figure D.1: Schematic of CSV apparatus used without (A) and with (B) shear addition. A supernatant layer sampled is shaded.

APPENDIX E

SUPPLEMENTAL DATA FOR CHAPTER FIVE

E.1 AUTHORS

William A.S.K. Mancell-Egala[a], Haydée De Clippeleir, Chungyung Su, Imre Takács, John T. Novak, David Kinnear, and Sudhir N. Murthy

E.2 FIGURES

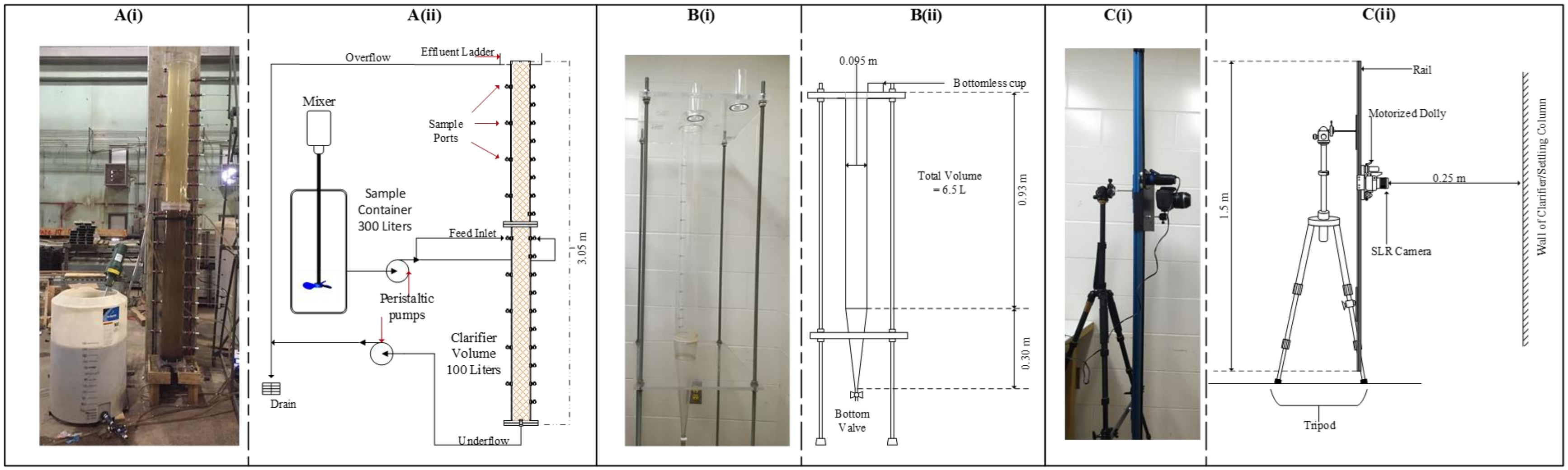
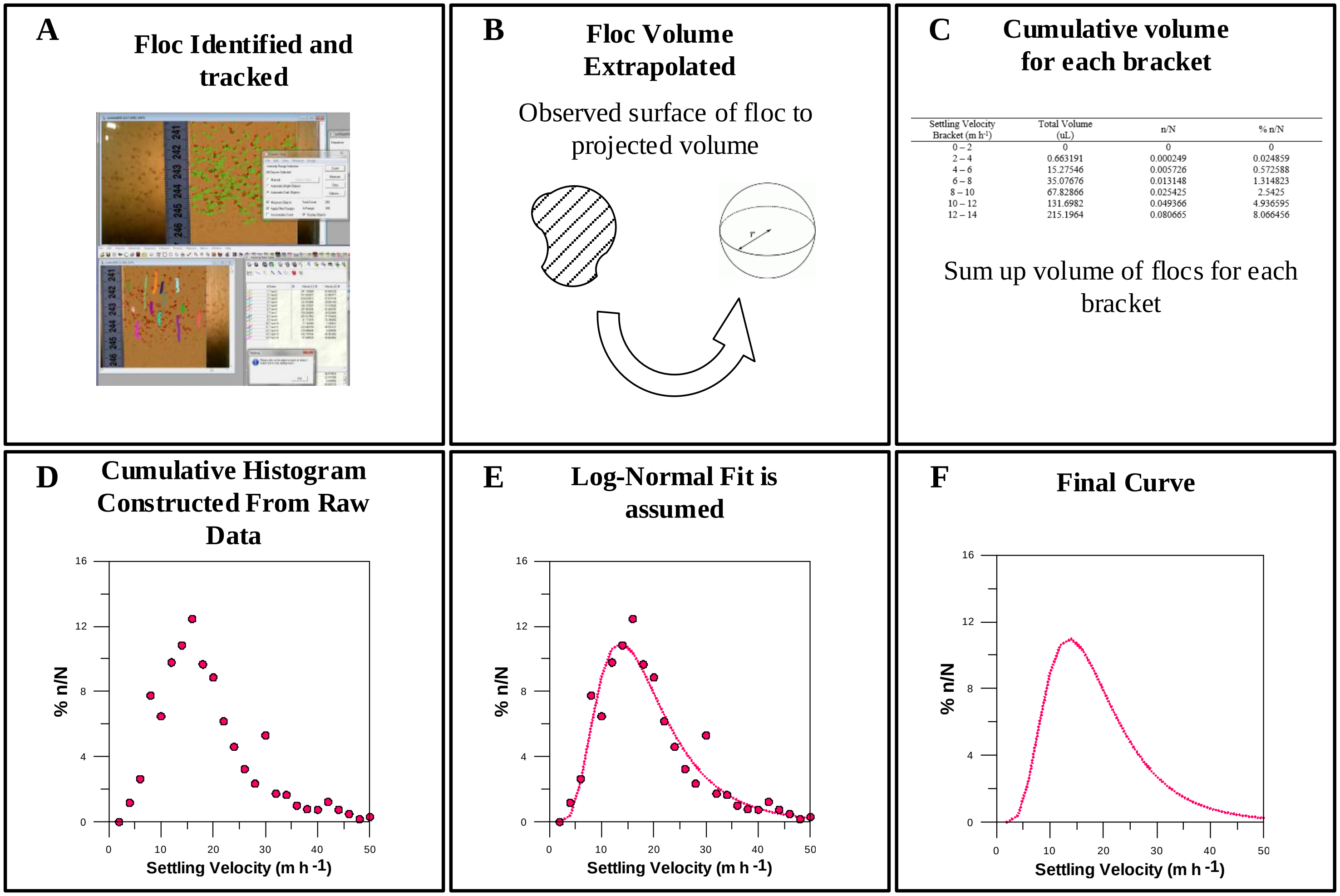


Figure E.1: (Ai) Picture and (Aii) schematic of the pilot clarifier used during the study. (Bi) Picture and (Bii) schematic of the flocculant setting column used during the study. (Ci) Picture and (Cii) Schematic of the camera and motorized dolly used for capturing the settling flocs.



Settling Velocity Bracket (m h^{-1})	Total Volume (uL)	n/N	% n/N
0 – 2	0	0	0
2 – 4	0.663191	0.000249	0.024859
4 – 6	15.27546	0.005726	0.572588
6 – 8	35.07676	0.013148	1.314823
8 – 10	67.82866	0.025425	2.5425
10 – 12	131.6982	0.049366	4.936595
12 – 14	215.1964	0.080665	8.066456

Figure E.2: Steps taken to construct both SVD and PSD. (A) After video is analyzed, (B) each floc's volume is estimated from the surface area data. (C) The sum of the total floc volume is calculated depending on the bracket, whether ferret diameter or settling velocity. (D) The data is plotted and (E) a Log-Normal fit is assumed using the curve fitting tool in MATLAB to produce (F) the final curve.

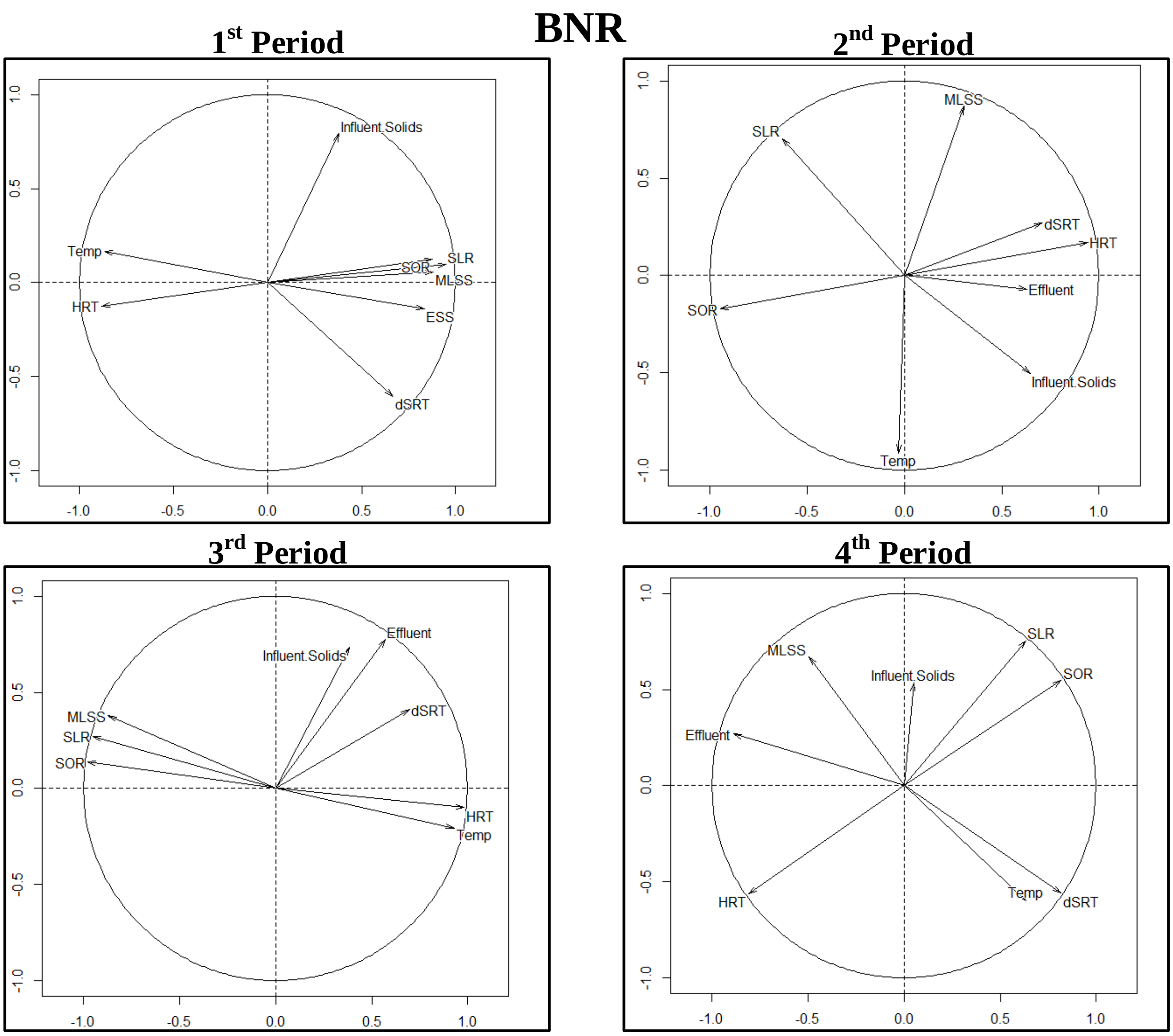


Figure E.3: PCA variable factor map for the BNR system's periods 1 to 4.

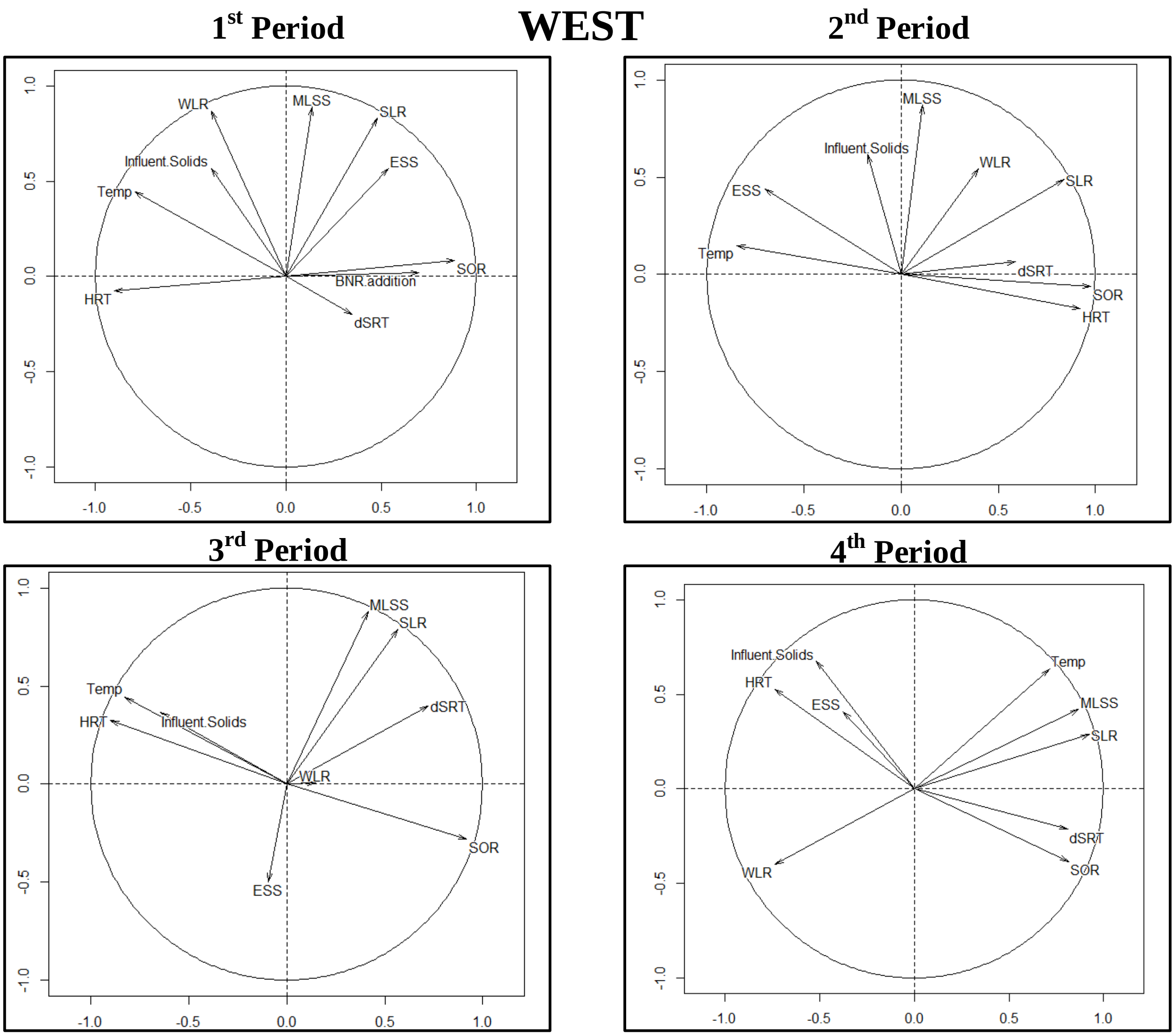


Figure E.4: PCA variable factor map for the West system's periods 1 to 4.

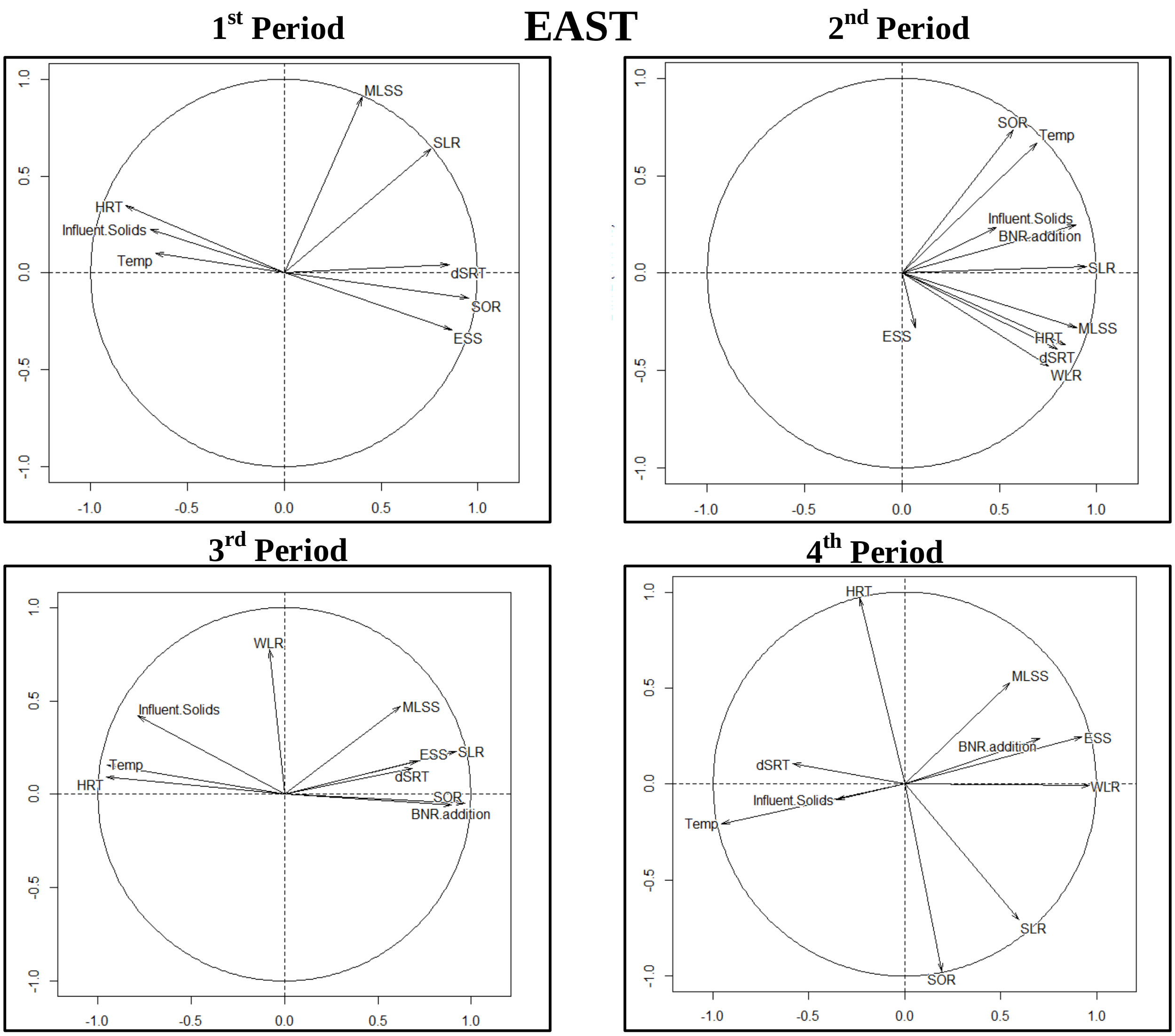


Figure E.5: PCA variable factor map for the East system's periods 1 to 4.

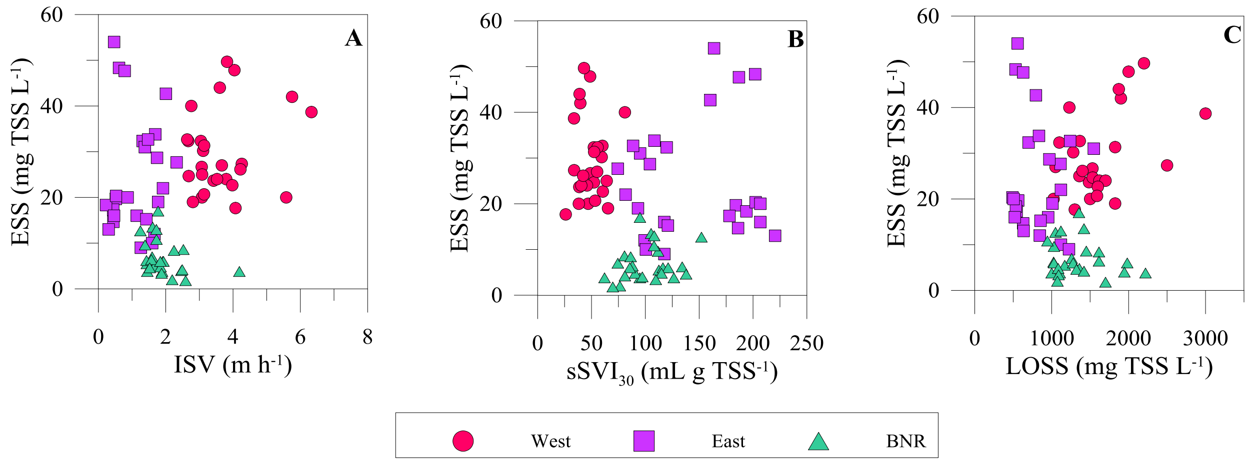


Figure E.6: : A) Initial settling velocity (ISV), (B) stirred sludge volume index (sSVI30), and (C) limit of stokesian settling (LOSS), versus effluent suspended solids (ESS) over 1 year of monitoring for West, East, and BNR systems.