ABSTRACT

SKIDMORE, MATTHEW BRIAN. Predicting and Improving the Dewaterability of Waste Activated Sludge Through Moisture Distribution Analysis. (Under the Direction of Francis L. de los Reyes III).

The dewatering of waste activated sludge is one of the most costly and least understood operations in wastewater treatment. While much effort has been put into streamlining operation of sludge dewatering equipment to maximize moisture removal, until recently, little attention was paid to the water within sludge. Sludge water molecules can be subdivided into several categories according to various properties with efforts made to convert sludge water from one category to another with the goal of making it easier to remove. This thesis tests two different ideas. First, that a dewaterability prediction test can be developed that is based on the distribution of moisture within activated sludge. Second, that sludge water can be converted from one form to another thereby improving sludge dewatering.

A centrifugal dewatering test was developed based on the idea that as centrifugal forces approach infinity, all of the free, or easily removable, water is withdrawn leaving only bound water. The centrifugal dewatering test consisted of centrifuging a 24-ml sample of waste activated sludge at ~150,000 x g for 30 minutes. Following centrifugation, the samples were inverted and supernatant was removed. The water remaining in the pellet was considered to be the bound water and represented the portion of sludge water that cannot be removed by mechanical dewatering equipment. Comparisons were made between the centrifugal dewatering test and full-scale wastewater treatment plant data. Significant positive correlations were made between test results and plant results for six of the eight plants

involved in the study. The tests value is that it could be used to predict trends in sludge dewaterability in advance of solids handling equipment. The purpose of developing the test was to demonstrate that investigations into sludge dewatering operations should include the entire wastewater treatment process, as well as, solids handling equipment.

The second part examines the idea that sludge moisture present as bound water can be converted to free water thereby improving the dewatering process. It was hypothesized that an input of disruptive energy could change the distribution of moisture within activated sludge. Heat treatment (40, 60, and 80°C), sonication, alkaline treatment, and cation addition were each evaluated alone, and in combination, for their ability to alter moisture distribution. Results indicated that only sonication, both alone and combined with cation addition, led to increased dewaterability and compactibility, as well as, decreased bound water. Cation addition alone led to increased dewaterability.

Predicting and Improving the Dewaterability of Waste Activated Sludge Through Moisture Distribution Analysis

by
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BIOGRAPHY

Matthew Brian Skidmore was born April 14, 1975 (same day in history that the Titanic sank and Abraham Lincoln was shot). He grew up in Wheaton, MD before his family moved one hour north to Finksburg, MD prior to high school. Matthew, or Matt, as most people call him, was never an ideal student. He was often in trouble, spent many days in detention and was suspended on more than one occasion for disciplinary reasons. He was a constant fixture in the Principal's office. Upon graduating from Westminster Senior High School, he was surprised to discover he was accepted to Towson University in Towson, MD (the only school he applied to). Being an avid drummer, he decided to study music education but soon soured on the idea of teaching. As a sophomore, he changed his educational focus to environmental science and quickly discovered that he not only liked the subject, but excelled at it. After his sophomore year, he transferred to the University of Maryland at College Park to pursue a Bachelors degree in natural resources management (where he met his future wife Lori). After a surprisingly fast two and a half years he graduated with a BS in natural resources management, concentration in plant and wildlife management. After getting hitched in 1999, Matt and Lori decided to move to Cary, NC since neither one really enjoyed life in the Washington DC metropolitan area. Matt accepted a job working for the soil science department at North Carolina State University while Lori accepted a consulting job paying twice as much. Since Matt worked for NC State University he was able to take courses for free. He soon tired of the work he did for the University. While it was rewarding in its own way, it never really challenged him and he knew he could do better. Therefore in 2000, he set off to earn a Masters degree in environmental engineering and begun the painful process of making up the undergraduate coursework necessary. Finally in 2003, undergraduate work complete, he joined the research group of Francis L. de los Reyes III to earn his Masters degree. Matt and Lori currently live in Cary, NC where they have no children, but one crazy part German Shepherd, part Siberian Husky, part something else mutt named Tybalt. Matt is currently working for ARCADIS, a consulting firm in the Durham, NC area where he hopes to make enough money to send Lori back to school to earn her PhD. After 9.5 years of college experience Matt is finally ready to call it quits and cheer on Lori whom he wises the best of luck to in her academic endeavors.

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Chapter 1. Introduction and Literature Review

There are generally two end-products associated with the treatment of domestic wastewater; effluent, which is released into a receiving body of water, and waste sludge, which is treated for pathogen reduction and removal of excess moisture prior to final disposal. Sludge disposal is one of the most costly aspects of wastewater treatment. The estimated cost of sludge handling facilities is approximately 30-40% of the total investment in wastewater treatment facilities (NRC, 2002). Furthermore, operating costs associated with sludge dewatering equipment can represent as much as 50% of a facility's operating and maintenance costs (NRC, 2002).

Waste sludge is a mixture of organic, inorganic, and dissolved solids contained in a slurry that may be anywhere from three to fifty percent solid material (following thickening and dewatering). Sludge is commonly disposed of by incineration, landfilling, or application to cropland. Whichever method is chosen, the cost of disposal is a function of the water content of the sludge. In the case of landfilling or land-application, the cost is usually based on the volume of sludge to be disposed. Removing as much water as feasible reduces the sludge volume and subsequent cost. In the case of incineration, the presence of excess moisture will require an expenditure of energy for evaporation. The more water that is removed in prior steps, the lower the energy input will be. Figures 1.1 and 1.2 (data provided by C. Michael Bullard of Hazen and Sawyer, personal communication, July 2003) show the disposal costs for landfilling and thermal drying of waste sludge as a function of percent sludge solids.

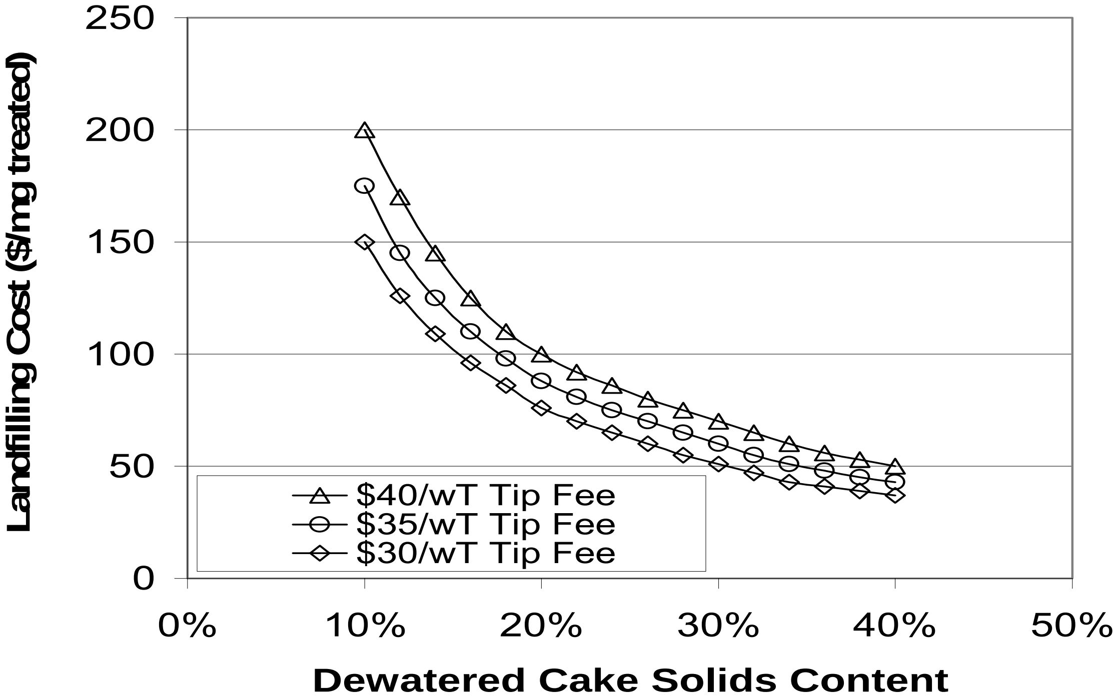

Figure 1.1. Disposal cost by landfilling as a function of dewatered cake solids.

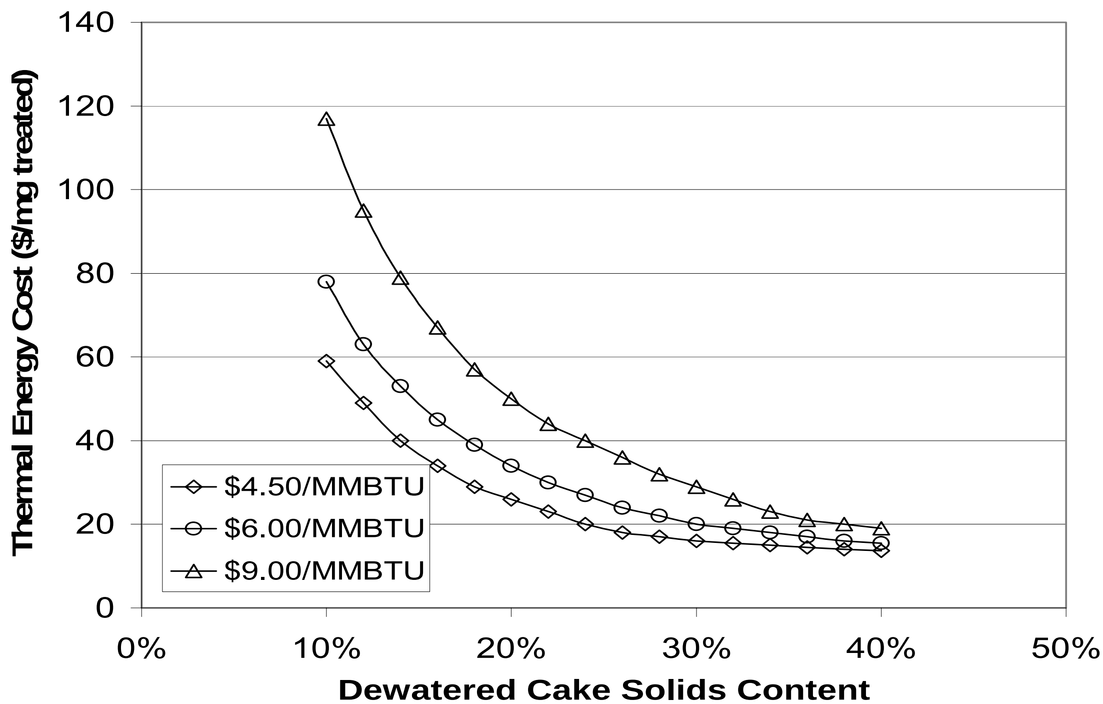

Figure 1. 2. Thermal drying energy cost as function of dewatered cake solids.

Since removal of excess moisture in sludge can lead to considerable reductions in disposal costs, sludge dewatering has become a highly important operation for many wastewater treatment plants looking to reduce financial costs.

With the high cost of solids disposal, many wastewater treatment facilities have invested time and money in the sludge dewatering process. Many wastewater treatment plants have installed mechanical dewatering equipment to remove excess moisture from sludge. Sludge polymer has become widely used for its ability to increase water removal efficiency. Polymer itself does not remove water, but when combined with mechanical dewatering equipment has been shown to improve sludge dewatering processes (Spellman, 1997). Belt-filter presses (BFP) consist of a series of rollers and belts where the sludge is squeezed between the belts and the rollers, expelling moisture. Centrifuges apply centrifugal force to the sludge by rotating at high speeds separating solid material from liquid based on density. Plate-and-frame presses rely on the pressure of two plates squeezing a volume of sludge in between the plates, and where excess moisture is pushed through a filter on one plate. Each of these methods relies on the application of pressure to separate water and solid matter.

Previous researchers have noted that dewatering activated sludge can be difficult.(Chen et al., 1996; Erdincler and Vesilind, 2000; Katsiri and Kouzeli-Katsiri, 1987; Vaxelaire and Cezac, 2004). The relationship between solid matter and water molecules is thought to control the dewaterability of activated sludge. Several properties of activated sludge make it difficult to dewater. Li and Ganczarczyk (1990) noted highly porous fractal-like structures in the interior of activated sludge flocs. Vesilind (1994) theorized that water within these interior structures difficult to remove due to strong attractive forces between solids and water molecules. These forces between solids and water molecules divide sludge water into different categories based on ease of removal from the sludge matrix. At its most basic level water is split into free water and bound water. While free water is generally thought to be easily removed from activated sludge, bound water is considered to be the portion of water that remains and is not removed, even under high pressures produced by mechanical dewatering equipment. Correlations between bound water content and sludge dewaterability have been made (Smollen, 1990). Katsiris and Kouzeli-Katsiri (1987) showed that increased bound water content led to decreases in sludge dewaterability.

The role of the sludge in dewatering has only recently begun to draw attention. Evidence that the sludge itself plays a role in dewatering can be seen in the recent study by Leonard et al (2004). The researchers subjected different sludges to identical drying conditions and saw that different sludges had different drying rates despite being nearly identical in all other aspects. Numerous sludge properties have been shown to influence the dewaterability of sludge. Properties identified include sludge pH, volatile to fixed solids ratio, septicity, sludge viscosity, compressibility, sludge particle size, and sludge surface charge (Spellman, 1997). Jin et al. (2004) showed that physical properties of sludge (flocculating ability, surface charge, hydrophobicity, and viscosity) were most important in terms of dewatering sludge while morphological properties (fractal dimension and filament index) had small impact on sludge dewaterability. Liao et al (2000) used bench scale reactors and saw that bound water was not correlated to solids retention time and surface charge. It was related however to floc size with bound water content increasing as mean floc size decreased. Lee

(1994A) also noted a correlation between bound water content and floc structure in both filamentous and non-filamentous sludges.

While extensive evidence points to the existence of bound water within activated sludge, there is uncertainty about how bound water arises. Several theories have been put forth which describe bound water as a system of layered water molecules closely spaced and arranged by their polar ends. It is thought that this layering is what gives bound water its unique properties. Szent-Gyorgyi (1957) suggested that in the presence of solids, liquid molecules layer themselves. He described the layered molecules as "ice-like" in structure and physical behavior. While "ice-like" may be an incorrect term for describing bound water in activated sludge, there is evidence that this layering does occur in the presence of solids. Previous work has shown that layered water can extend from solid surfaces up to 10 molecular diameters into the bulk liquid (Israelahvili and Kott, 1989). Drost-Hansen, (1981) noted the existence of "interfacial water" near solid surfaces and that this "interfacial water" had physical properties different from the corresponding properties of the bulk water. Several researchers have noted changes in both density and viscosity of bound water (Etzler and Fagundas, 1987; Etzler and Drost-Hansen, 1983). Vesilind (1994) hypothesized that the layering of water near solid surfaces was due to hydrogen bonding. He thought that surface interruption of normal water molecule packing led to a compensation where the molecules arranged themselves in an orderly fashion on the surface. Other researchers have noted the existence of layered water molecules near solid surfaces in other systems. Pollack (2001) argued that water molecules arrange themselves into layers based on charge. One molecule first attaches to a site on the surface (a protein in this particular case) and subsequent molecules attach based on their polar charge. This phenomenon is shown below in Figure 1.3 which is taken from Pollack (2001).

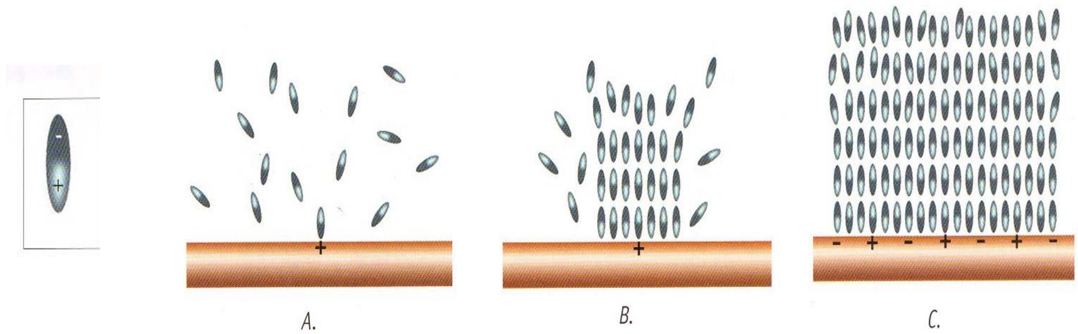

Figure 1.3. Layering of water molecules following surface attachment. Figure taken from Pollack (2001)

The fact that bound water has unique properties including density, vapor pressure, viscosity, and freezing point that are each different from bulk water is what has enabled researchers to measure bound water in activated sludge. Numerous methods have been developed to measure bound water by relying on its different properties. Several methods for bound water measurement in activated sludge are listed below in Table 1.1

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Table 1.1. Methods for measuring bound water in activated sludge systems. References are for method use in activated sludge systems only.

Method	**Principle**	**Reference**
Dilatometry	Bound water does not freeze at temperatures as low as -20C. Bound water content is calculated by measuring the free water based on expansion measurement and subtracting from total water content.	Heukelekian and Weisburg 1956
Differential Scanning Calorimetry	Bound water does not freeze at temperatures as low as -20C Enthalpy absorbed during freezing is proportional to free water and bound water content is calculated by mass balance.	Lee and Lee 1995
Centrifugation	As centrifugal force inceases and approaches infinity all free water will be forced out of floc and only bound water will remain. Bound water can be measured through total solids analysis.	Kawasaki et al 1991
Drying Tests	Thin layers of sludge are dried at temperature well below boiling point of water. Sludge weight is measured over time and changes in evaporation rate are considered to indicate different fractions of water	Halde 1979
Differential Thermal Analysis	The temperature difference between a sludge sample and a thermally inert material is measured while both are subject to identical heating and cooling. Sharp differences indicate the adsorption or liberation of heat.	Katsiris and Kouzeli-Katsiri 1987
Expression Test	Sludge is exposed to a constant high pressure (30 MPa). Subsequently all removable water is forced out and only bound water remains.	Chu and Lee 1999B
Filtration Test	Moisture content of sludge is measured following vacuum filtration. Free moisture is pulled through the filter under vacuum leaving bound water in sludge on filter.	Lee 1996

Measuring bound water is not without its difficulties. In a study comparing different methods of bound water measurement, Lee and Hsu (1995) concluded that bound water was an operationally defined value and therefore the measurement method chosen would dictate the bound water values measured. Robinson and Knocke (1992) also examined several methods of bound water measurement and concluded the same although they stated their personal preference for dilatometry due to its more reproducible results and the fact that it was less prone to error. An issue with bound water measurement is the absence of a bound water standard with which to calibrate measurements. Without such a standard, bound water measurements become comparative where the amount of bound water is measured before and after treatment.

In an attempt to better understand the concept of moisture distribution in sludge, researchers have expanded the concept of free and bound water with additional classification schemes. Several more complex classifications of moisture have been put forth.

Colin and Gazbar (1995) proposed a moisture classification scheme based on the binding energy of moisture to activated sludge surfaces. The authors measured dilatometric bound water as a function of mechanical dewatering strain and divided sludge water into the following categories; (1) Total bound water. (2) Free water – calculated as the difference between total water and total bound water. (3) Water not removable mechanically. (4) Bound water removable under a realistic mechanical strain. (5) Bound water removable under maximum applied mechanical strain.

Smollen (1988) classified water by the amount of bonding energy holding the water in place and developed four categories of sludge moisture; (1) Free moisture – loosely bound to sludge solids, removed by gravitational thickening. (2) Immobilized moisture – water that is trapped within floc structures, removed by mechanical dewatering. (3) Bound moisture – water that is adsorbed onto individual particles, removed by thermal drying. (4) Chemically bound moisture – water that bound through chemical attraction to solids, removed under high temperature drying.

Kopp and Dichtl (2000) proposed a similar classification system based on physical bonding of moisture to sludge particles including; (1) Free water – not bound to particles. (2) Interstitial water – bound by capillary forces to sludge solids. (3) Surface water – bound by adhesive forces. (4) Intracellular water.

Similarly Moller (1983) proposed the following classifications based on solid and liquid components; (1) Interspace moisture – minimally bound to solids, removed by draining. (2) Capillary water – bound by capillary pressure, can be removed by application of pressure. (3) Adhesion water – intermediately bound to solids, can be removed by pressure. (4) Adsorption water – intensely bound by ionic double layer, removed thermally. (5) Internal water – inside of individual particles, must be converted to external water before removal.

Vesilind (1994) argued similarly that four phases of water exist within activated sludge; (1) Free or bulk water – not confined to floc structures, free to move about sludge matrix. (2) Interstitial water – trapped within sludge floc, becomes free water when floc structure is disrupted. (3) Vicinal water – tightly bound layered water attached to solid surface, remains with solids following floc breakup. (4) Water of hydration – chemically bound to floc particles, removed through thermal input.

Figure 3.4 shows a visual representation of Vesilind's four categories of moisture classification using a simplified 2-dimensional model for a sludge floc particle. Bulk water can be found all around the floc particle and is free to move towards and away from the particle. The blue areas represent interstitial water which is constrained within the floc interior, but is able to move if floc structure is disrupted. The red areas represent vicinal water which is layered and is held to the floc surface regardless of floc breakup. The green areas represent the interior structures of floc particles including cells. Water of hydration is exists within these structures. To simplify nomenclature, the Vesilind (1994) moisture classifications will be used throughout this report.

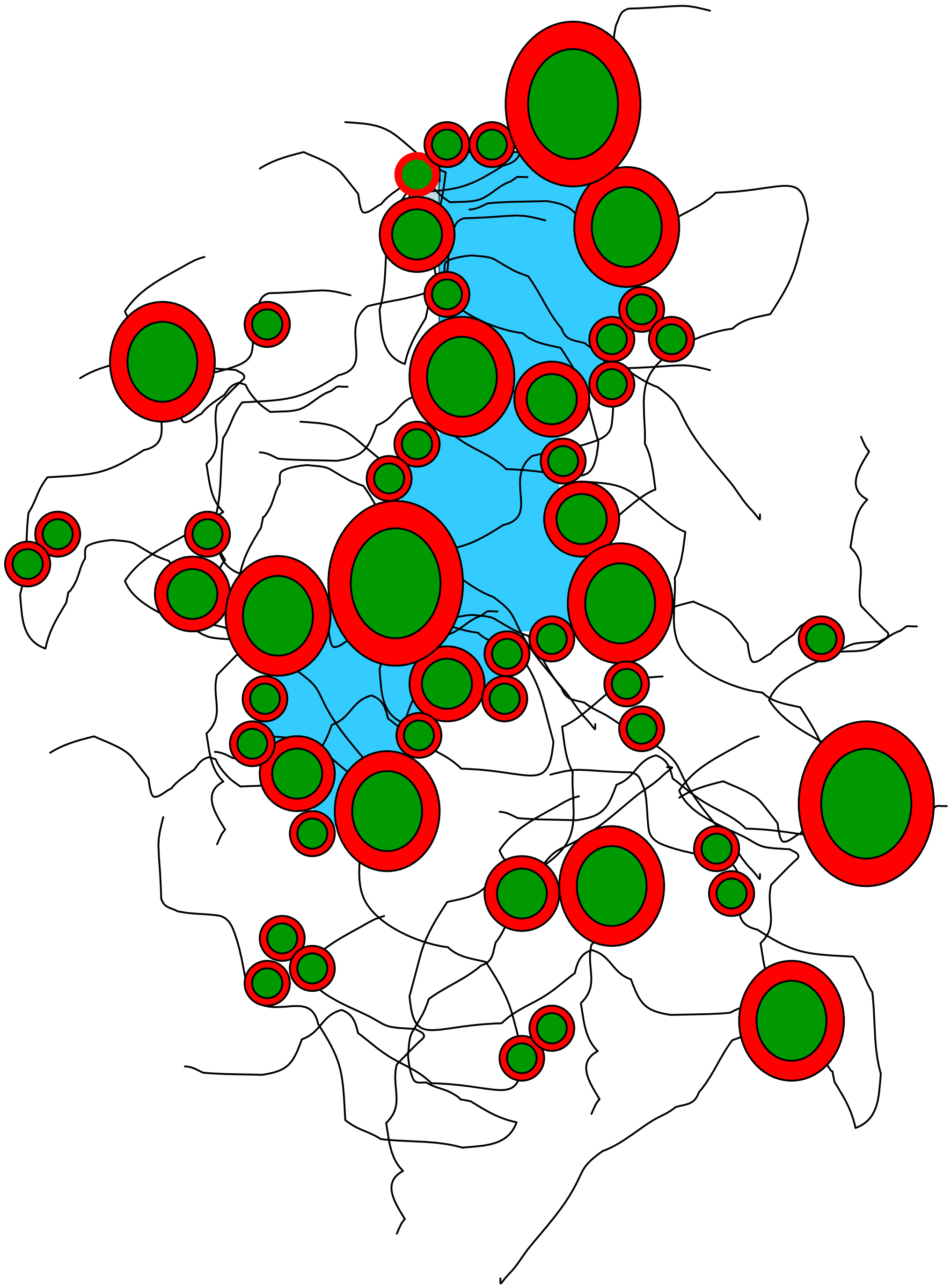

Figure 1.4 Visual presentation of four categories of sludge water as defined by Vesilind (1994).

Vesilind and Hsu (1997) suggested that bound water actually consisted of water of hydration, vicinal water, and a portion of interstitial water. Others have suggested that bound water contains only vicinal water and water of hydration (Dick and Drainville, 1995). The portions of water removable by mechanical means are also subject to intense debate. Vesilind (1994) suggests that only the free water and interstitial water can be removed by mechanical dewatering equipment. Kopp and Dichtl (2001) contend that only free water is removed by mechanical dewatering equipment.

Research has suggested that bound water can be converted to free water. Katsiri and Kouzeli-Katsiri (1987) noted a decrease in bound water content with the addition of chemical coagulants. They hypothesized that coagulant molecules replace water molecules within bound water. Chu and Lee (1999A) noted that bound water content decreased with polymer addition until the charge neutralization point was reached and increased afterwards with the addition of more polymer. They agreed with Smollen (1990) that excess polymer would adsorb bulk moisture and add to the bound water content. Erdincler and Vesilind noted an increase in bound water content following heat treatment, sonication, alkaline treatment, and NaCl treatment. They attributed the increase to the creation of extra surfaces for bound water formation following disruption. However, they did suggest that the interstitial water content decreased as a result of floc disruption.

The idea that bound water can be subdivided different categories and that bound water can be converted from one form to another have been examined. Vesilind (1994) and Vesilind and Hsu (1997) suggested that being able to measure the bound water (defined as the sum of water of hydration and vicinal water plus a portion of interstitial water) is important since this point would represent the maximum theoretical limit of unconditioned sludge dewaterability. By knowing the maximum dewaterability ahead of time, one could assess sludge dewaterability not in terms of how much water is removed, but rather the percentage of free water that is removed. This approach could lead to different ideas about assessing dewaterability. Currently tests such as specific resistance to filtration (SRF) and capillary suction time (CST) are used. These tests are used to measure changes in sludge dewaterability following different treatments. They do not indicate however the maximum possible dewaterability of that sludge. A study by Chen et al (1996) showed that bound water content can not be evaluated based on CST results. The purpose of moisture distribution studies has been to change the way dewatering efficiency is measured, from a process where the total amount of water removed is measured to a process where the percentage of removable water is measured.

This approach also creates opportunities for evaluating the theory that various disruption mechanisms can change the moisture distribution of a sludge and through trial and error, a method could be developed to reduce the quantity of bound water thereby improving mechanical sludge dewatering. As mentioned above, research has shown that bound water quantity can be altered through disruption mechanisms, both alone and in combination.

This study examines whether or not a test can be developed that can accurately approximate upper limits of dewaterability by measuring bound water content based on already theorized categories of moisture and if this test can be correlated with dewatering performance of wastewater treatment plants. It also examines the possibility of changing this upper limit through disruption mechanisms designed to reduce the quantity of bound water in sludge.

Chapter 2 Centrifugal Dewatering Test: Development and Correlation to Full-Scale Plant Dewatering Performance

ABSTRACT

A centrifugal dewatering test that measured sludge dewaterability based on moisture distribution was developed. The test is based on the concept that as centrifugal forces approach infinity, all free water within sludge is removed and only bound water will remain in the solid pellet. Results showed that centrifugation at ~150,000 x g for 30 minutes led to an asymptotic value of bound water remaining in the centrifuged pellet. Sludges from eight municipal wastewater treatment plants in the state of North Carolina were sampled over a two month period and centrifugal dewatering test results were compared to full-scale plant dewatering results. Significant correlations between test results and full-scale plant dewaterability results were obtained for six of the eight plants tested while results were inconclusive for the remaining two. We conclude that the centrifuge test is able to predict trends in sludge dewaterability prior to sludge dewatering equipment. Such a test will improve decision making related to solids dewatering procedures, because it can be used to forecast dewaterability in advance of solids handling processes.

2.1 INTRODUCTION

An inherent difficulty in biological sludge dewatering is the lack of a reliable test to predict the performance of dewatering equipment. Current dewatering tests such as capillary suction time (CST) and specific resistance to filtration (SRF) measure the rate of release of water from sludge. However, these tests do not indicate the distribution of moisture within sludge. Vesilind (1994) hypothesized that certain portions of sludge water are bound to sludge solids and cannot be removed through existing mechanical dewatering equipment. This portion of sludge water that cannot be removed was termed 'bound water'. In biological sludges, one of the most important properties is the bound water content. Bound water has been shown to positively correlate with full-scale wastewater treatment plant sludge dewaterability (Katsiris and Kouzeli-Katsiri (1987); Colin and Gazbar (1995); Kopp and Dichtl (2001); Robinson and Knocke (1992)). However, correlations between bound water and dewatering tests such as CST and SRF have not been made. Chen et al (1996) concluded that bound water content cannot be evaluated based on CST measurements. No direct comparison between SRF and bound water content has been made.

Previous research has demonstrated that despite increased dewatering effort, there is a portion of water that remains associated with sludge solids. Using a constant-head piston press to separate sludge solids and liquid, Chang and Lee (1998) developed a three-stage model for dewatering. Stages one (bulk water removal) and two (interstitial water removal) correlated with previously developed models for the relationship between inorganic particulate sludge particles and sludge water. However, a third stage also existed that could not be predicted with existing models. They concluded that this third stage, seen only in biological sludges, was due to the presence of large amounts of bound water in biological sludge which would require increased pressure to remove. Chu and Lee (1999B) later obtained additional data showing that a portion of sludge water remains with sludge solids even under large amounts of pressure. Colin and Gazbar (1995) developed the concept of phases of sludge water based on binding strength. They also concluded that some portion of water was not removable by mechanical means. Vesilind and Hsu (1997) stated that accurate measurement of sludge bound water content would provide insight into the practical limit of sludge dewaterability.

One method for measuring bound water content in biological sludge is centrifugation. Centrifugation at high speeds may separate sludge into free removable water and bound water that remains with the sludge solids. In centrifugation the rotational speed is related directly to the centrifugal force. Chu and Lee (2002) identified increased rotational speed as a way to enhance centrifugal dewatering efficiency. Lee (1994B) developed a centrifuge test to measure bound water content in sludge. Their results indicated that centrifugation alone was not able to accurately measure bound water content. However, their test was conducted only at 2000 x g for 30 minutes. Using a similar experimental set-up, Yen and Lee (2001) noticed a rebound effect for sludge height following centrifugation. They also noted that a threshold rotational speed existed beyond which the rebound effect largely deteriorated. Matsuda et al (1992) postulated that if the centrifugal force goes to infinity, all void spaces within sludge would be collapsed and only sludge solids and bound water would remain. Barber et al (1995) developed a centrifugal dewatering test based on this hypothesis and the idea that bound water represents a practical limit to the dewaterability of sludge. The test was developed as part of an effort to improve solids dewatering at an industrial wastewater treatment plant, and consisted of centrifugation at 45,100 x g for ten minutes. Although they never reached the true mechanical dewatering limit where increased centrifugal force results in no increase in removal of water, their test did show significant correlations with full-scale plant dewatering performance, leading to its use as a decision making tool regarding plant operations. However, one cannot draw any conclusions about the ability of the test to predict dewatering trends at other plants.

The objective of this research was to determine if a centrifugal dewatering test could be developed that could measure the portion of water removable through mechanical equipment. One of the goals was to determine if there is a point where increasing the centrifugal force applied to a sludge sample would not increase the percent total solids of the remaining pellet using centrifugal forces of up to 225,000 x g.. Hypothetically, this would be the point where only bound water and sludge solids would remain and could theoretically be considered the maximum unconditioned (without polymer addition) sludge dewaterability. A secondary objective of the study was to determine if the developed centrifugal dewatering test could be correlated to full-scale wastewater treatment plant dewatering performance regardless of solids handling procedures used. This would demonstrate that sludge dewaterability is a function of moisture distribution and that at some point, further enhancements to dewatering equipment may not lead to comparable increases in sludge dewatering. The development of such a test could lead to: (1) a more fundamental understanding of the relationship between liquid and solids within sludge and (2) a different approach to dewatering sludge where more effort is made to control moisture distribution in sludge through operational changes to the entire wastewater treatment process.

2.2 METHODS

2.2.1 Centrifuge

A Sorvall Ultra-80 centrifuge equipped with a Beckman type 50.2-ti fixed-angle rotor and 30 ml round-bottom polycarbonate centrifuge tubes were used. This centrifuge/rotor combination was capable of speeds up to 50,000 rpm (~225,000 x g).

2.2.2 Samples

Samples of waste activated sludge (WAS) samples from four municipal wastewater treatment plants in North Carolina were collected. The plants chosen were the North Cary Water Reclamation Facility (NCWRF), the South Cary Water Reclamation Facility (SCWRF), The South Durham Water Reclamation Facility (SDWRF), and the Neuse River Wastewater Treatment Plant (NRWWTP). Approximately one gallon of sample was collected from each of the four plants in early November 2004. All of the four plants use activated sludge systems for the treatment of municipal wastewater. The four plants were chosen based on their wide variety of reactor configurations, differences in sludge handling equipment, and geographical proximity.

Verification consisted of correlating centrifugal dewatering test measurements to full-scale plant dewatering performance. Eight municipal wastewater treatment plants agreed to submit samples for testing and provide dewatering data for the specified dates. The eight plants were chosen to represent a variety of solids handling and dewatering equipment technology. The eight plants chosen included the previously mentioned NCWRF, SCWRF, SDWRF, and NRWWTP. In addition, the McDowell Creek WWTP

(MCWWTP) in Huntersville, the James A. Loughlin WWTP (JLWWTP) in Wilmington, the Town Creek WWTP (TCWWTP) in Salisbury, and the Archie Elledge WWTP (AEWWTP) in Winston-Salem agreed to participate and submitted samples.

For test correlation purposes, each plant submitted a total of 24 samples (three samples per week) over a two month period during November and December 2004. Centrifugal dewatering tests and total solids analysis were performed on all samples submitted. Three samples were collected per week and stored at 4°C. At the end of the week, all three samples were shipped to North Carolina State University for analysis. Previous researchers have seen changes in sludge properties during storage of samples (Nielsen et al., 1996; Bruus et al., 1993) so before starting the experiment, preliminary testing was performed to determine if centrifugal dewatering test results would change as a result of this storage. No significant changes were seen over a seven day period. Test data and results are included as Appendix I

All tests were performed in triplicate and centrifugal dewatering test data were compared to actual plant dewatering data to determine if the test could accurately predict trends in full scale plant dewaterability. Table 2.1 lists the locations, capacities, thickening, and dewatering equipment of all eight plants.

Table 2.1. Characteristics of wastewater treatment plants participating in dewatering study.

Plant	Abbreviation	City	County	Capacity (MGD)	Digester	Digestion Time	Thickening Method	Dewatering Equipment
North Cary	NCWRF	Cary	Wake	10	Aerobic	15 days	Gravity Belt Thickener	Belt Filter Press
South Cary	SCWRF	Apex	Wake	12.8	Aerobic	25 days	Gravity Belt Thickener	Centrifuge
Neuse River	NRWWTP	Raleigh	Wake	60	None	NA	Gravity Belt Thickener	Belt Filter Press
South Durham	SDWRF	Chapel Hill	Orange	20	Anaerobic	100 days	Settling Basin	Belt Filter Press
McDowell Creek	MCWWTP	Huntersville	Mecklenburg	12	Anaerobic	16 days	Gravity Belt Thickener	Belt Filter Press
Town Creek	TCWWTP	Salisbury	Rowan	5	Aerobic	18 days	None	Plate and Frame Press
James A. Loughlin	JLWWTP	Wilmington	New Hanover	8	Anaerobic	29 days	Gravity Belt Thickener	Belt Filter Press
Archie Elledge	AEWWTP	Winston-Salem	Forsyth	30	Anaerobic	75 days	Gravity Belt Thickener	Centrifuge

2.2.3 Test procedure

The centrifugal dewatering test is based on the method of Barber et al (1995). Initially, triplicate samples were centrifuged for 30 minutes at increasing speeds up to ~200,000 x g in increments of 25,000 x g. Immediately following centrifugation the tubes were inverted, the centrate was poured off, and the centrifuged pellet was carefully scraped out. Total Solids analysis was performed on the pellets according to Standard Methods (APHA, 1998). Percent total solids in the remaining pellet was plotted against centrifugal force, and the point where total solids ceased to increase was determined.

2.2.4 Correlations

Correlation analysis between the percent total solids from the centrifugal dewatering test and total cake solids from full-scale plant dewatering equipment was performed. Centrifugal dewatering test data was plotted versus full-scale plant performance and the correlation coefficient r was calculated. Since centrifugal dewatering tests were conducted on sludges prior to digestion, the digestion time was added for each centrifugal dewatering test date to allow accurate comparisons.

2.3 RESULTS AND DISCUSSION

2.3.1 Test Development

Preliminary tests were performed to determine the exact time and force necessary for establishing a maximum value where increased centrifugal force did not produce any further increases in solids content. It has been hypothesized that this point represents the maximum dewaterability for unconditioned sludge (Vesilind, 1994). Results of preliminary tests on sludges from NCWRF, SCWRF, SDWRF, and NRWWTP are shown below in Figure 2.1.

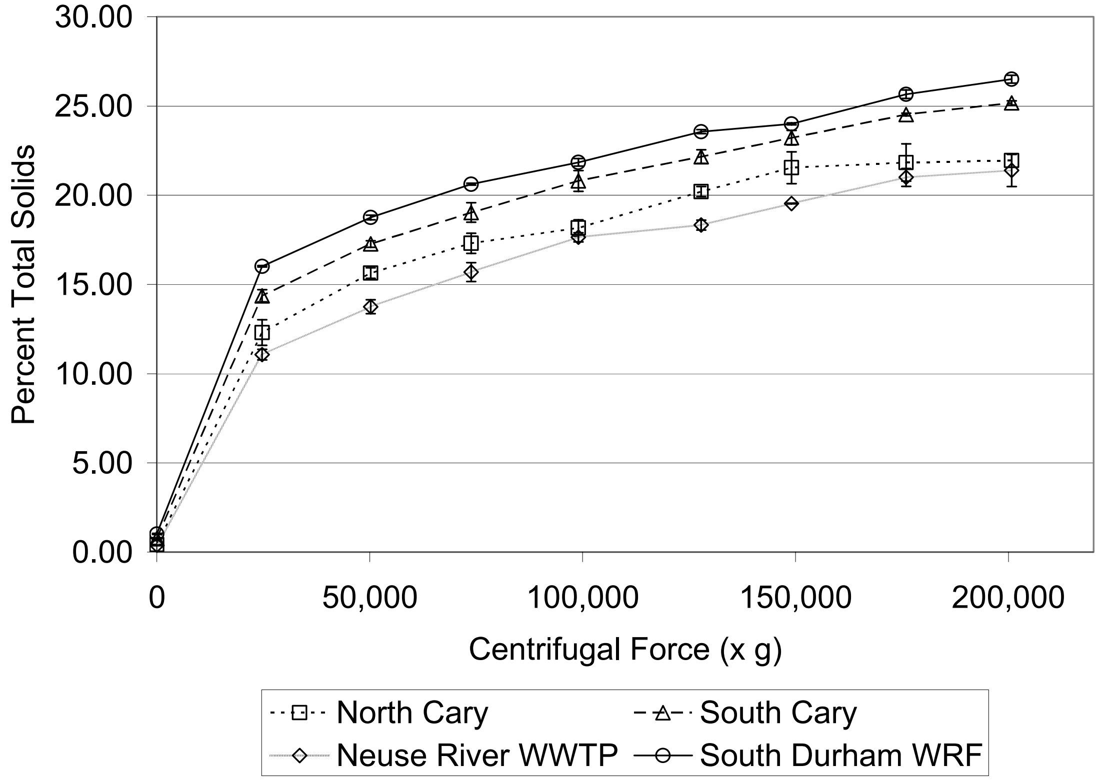

Figure 2.1. Preliminary results of centrifugal dewatering test Effects of centrifugal force on dewaterability.

NCWRF sludge indicated no statistically significant increase in percent total solids after ~150,000 x g. Results from NRWWTP indicate no significant increase following ~175,000 x g. Results from SCWRF and SDWRF showed significant increases at all speeds up to ~ 200,000 x g.

Based on the preliminary results, a centrifugal dewatering test was developed, which consisted of centrifugation at ~150,000 x g for 30 minutes. These values were chosen for measuring dewatering trends, as well as the upper limits of sludge dewaterability. This point was chosen based on the results from NCWRF, which represented the first point on any of the four curves where increasing centrifugal force did not result in increased total solids content in the remaining pellet. Having different plants tested at different centrifugal forces was considered. However, using different centrifugal force would make comparisons difficult.

2.3.2 Test Verification

Once the centrifugal dewatering test was developed, samples were collected from the eight plants participating in the correlation study. Significant correlations were established between the centrifugal dewatering test and full-scale plant dewatering performance for six of the plants in the study. Correlation study results for MCWWTP, JLWWTP, SCWRF, TCWWTP, NRWWTP, and AEWWTP are in Figures 2.2, 2.3, 2.4, 2.5, 2.6, and 2.7, respectively. Full-scale plant dewaterability data was also provided by the NCWRF and SDWRF but not enough points were provided to measure correlation. Data for these two plants are shown in Figures 2.8 and 2.9 respectively.

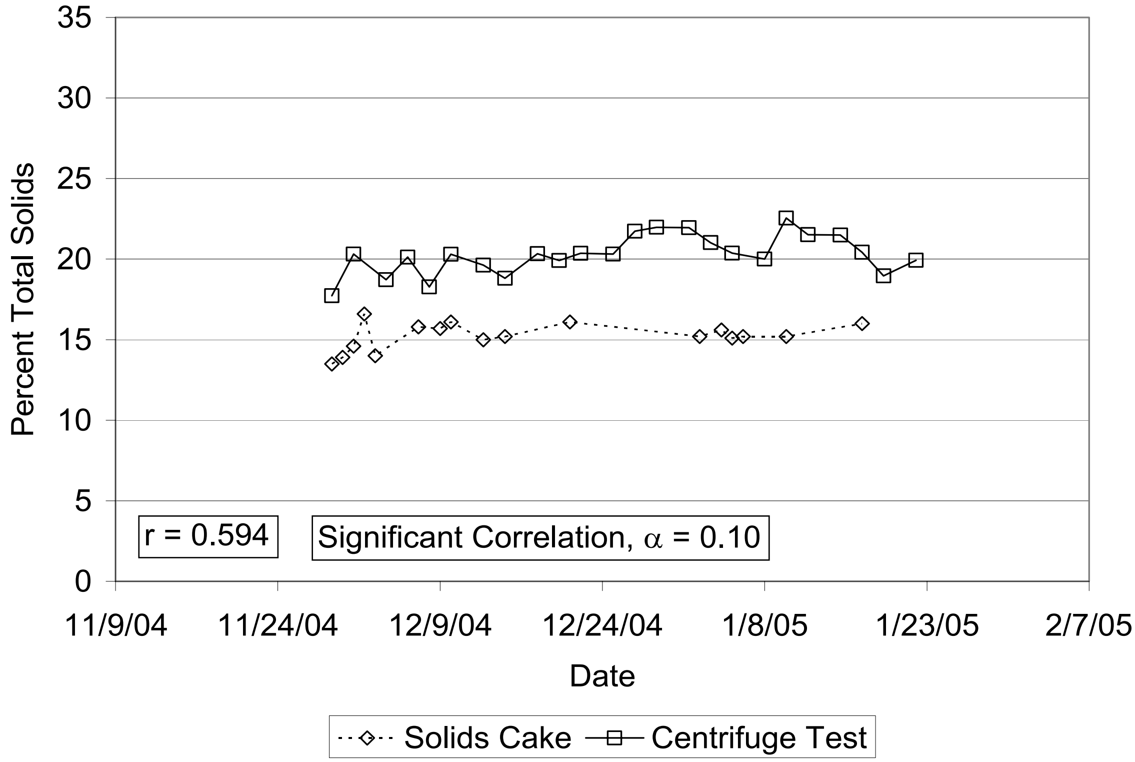

Figure 2.2, McDowell Creek wastewater treatment plant. Correlation of centrifugal dewatering test.

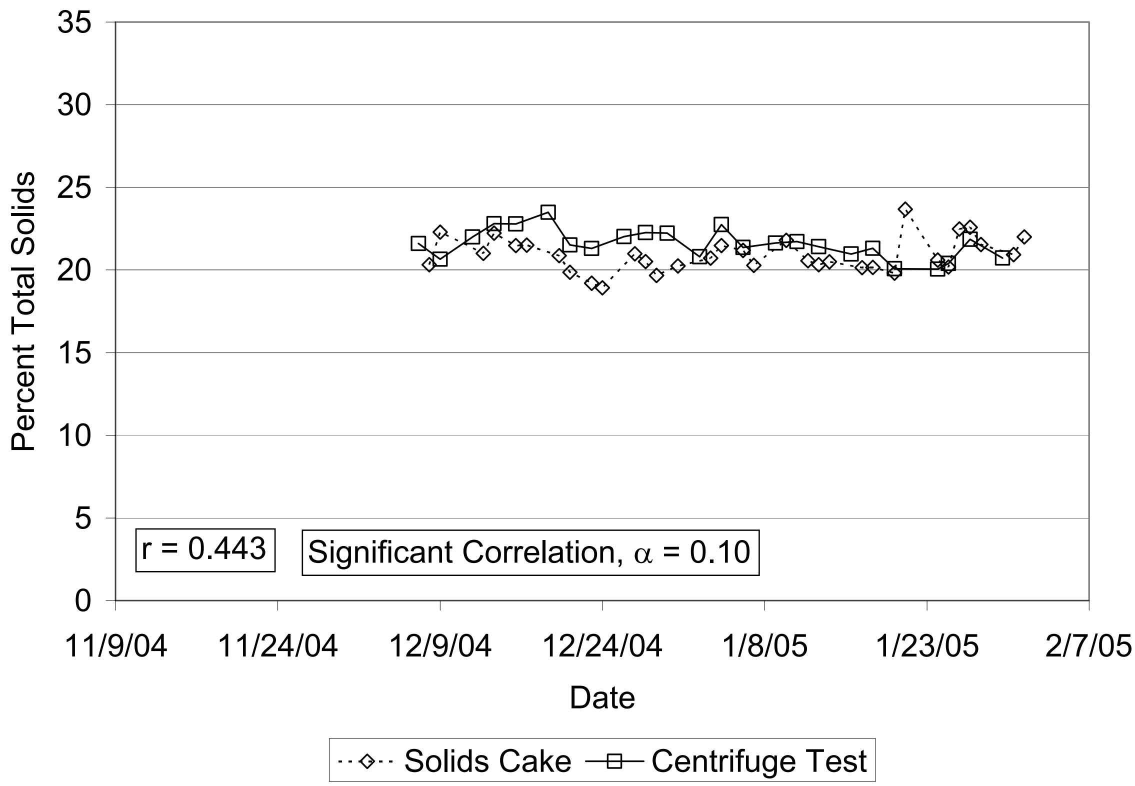

Figure 2.3, James A. Loughlin wastewater treatment plant, correlation of centrifugal dewatering test.

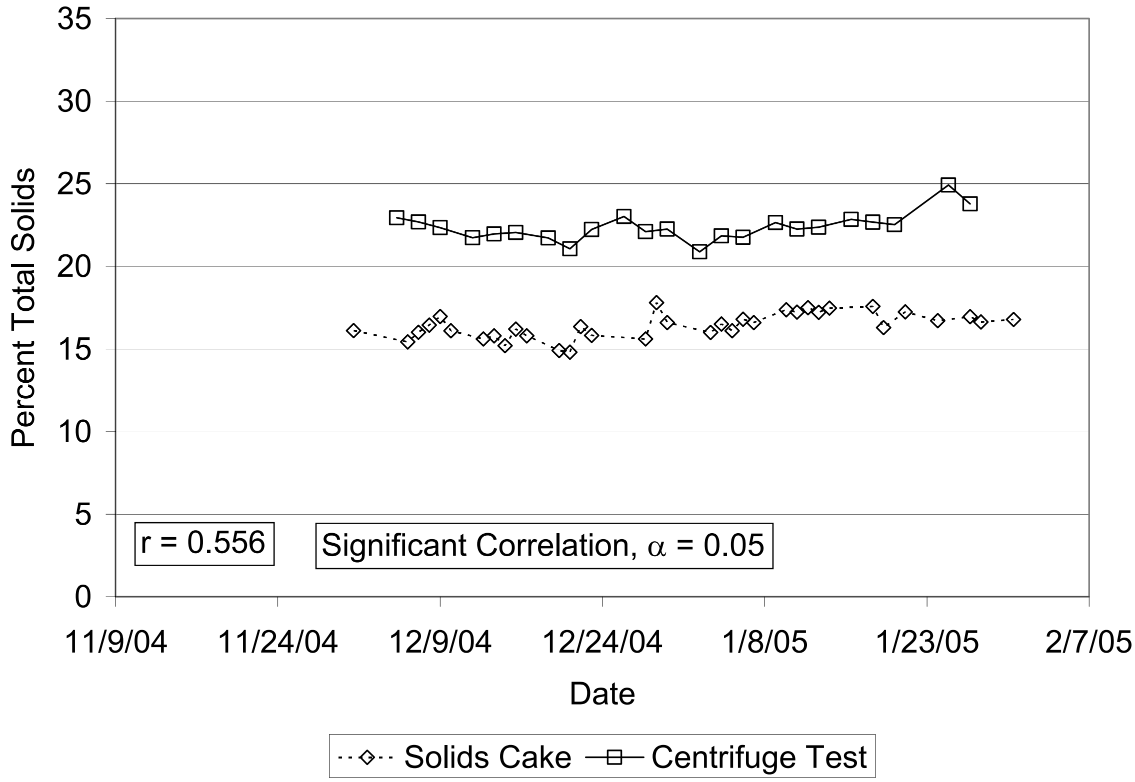

Figure 2.4, South Cary water reclamation facility, correlation of centrifugal dewatering test.

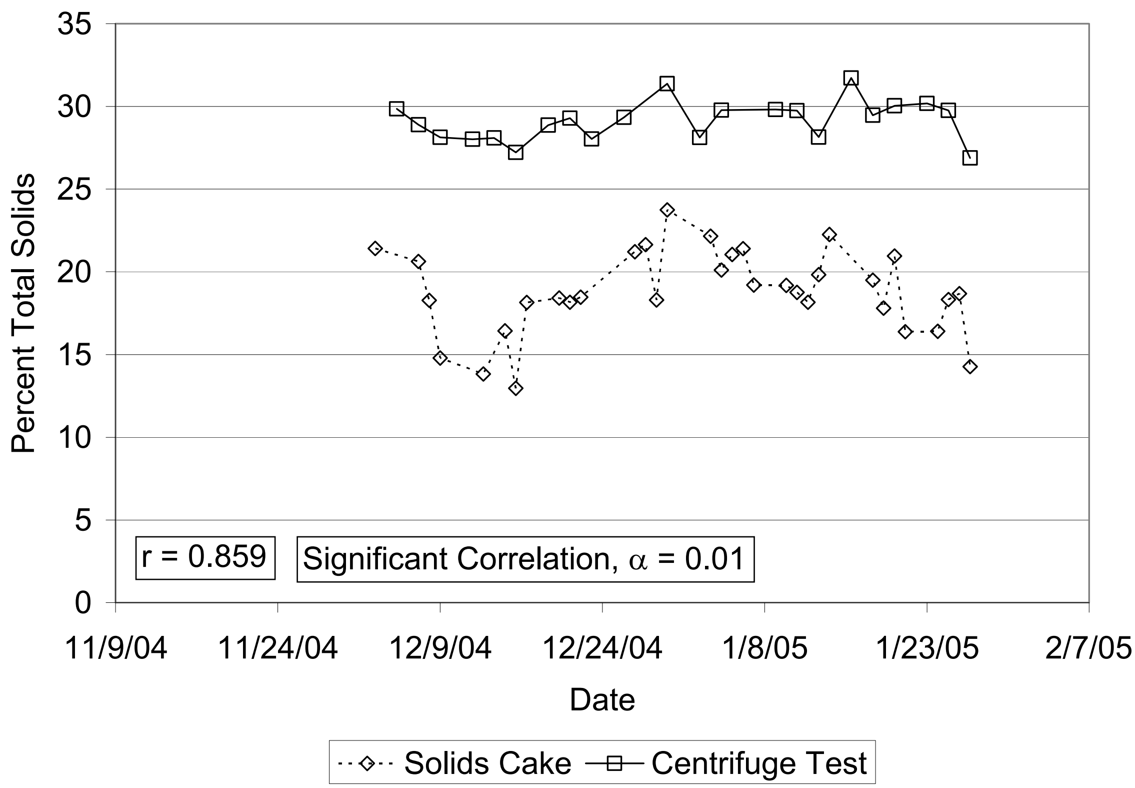

Figure 2.5, Town Creek wastewater treatment plant, correlation of centrifugal dewatering test.

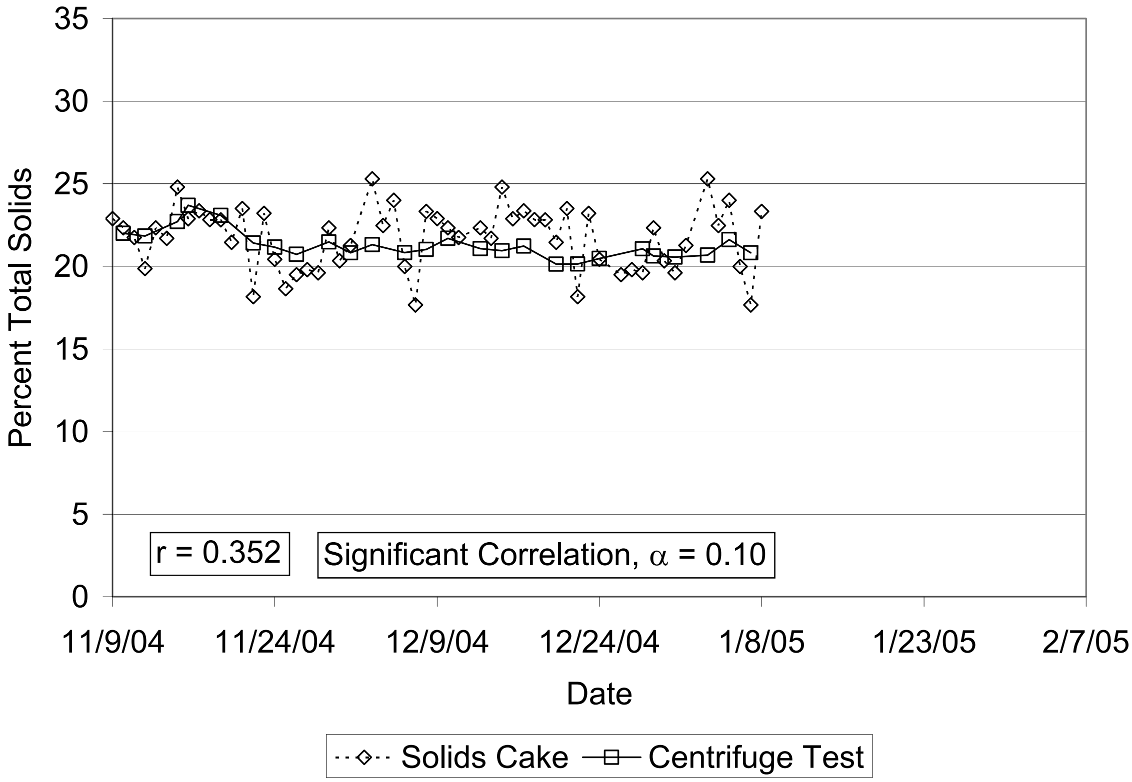

Figure 2.6, Neuse River wastewater treatment plant, correlation of centrifugal dewatering test.

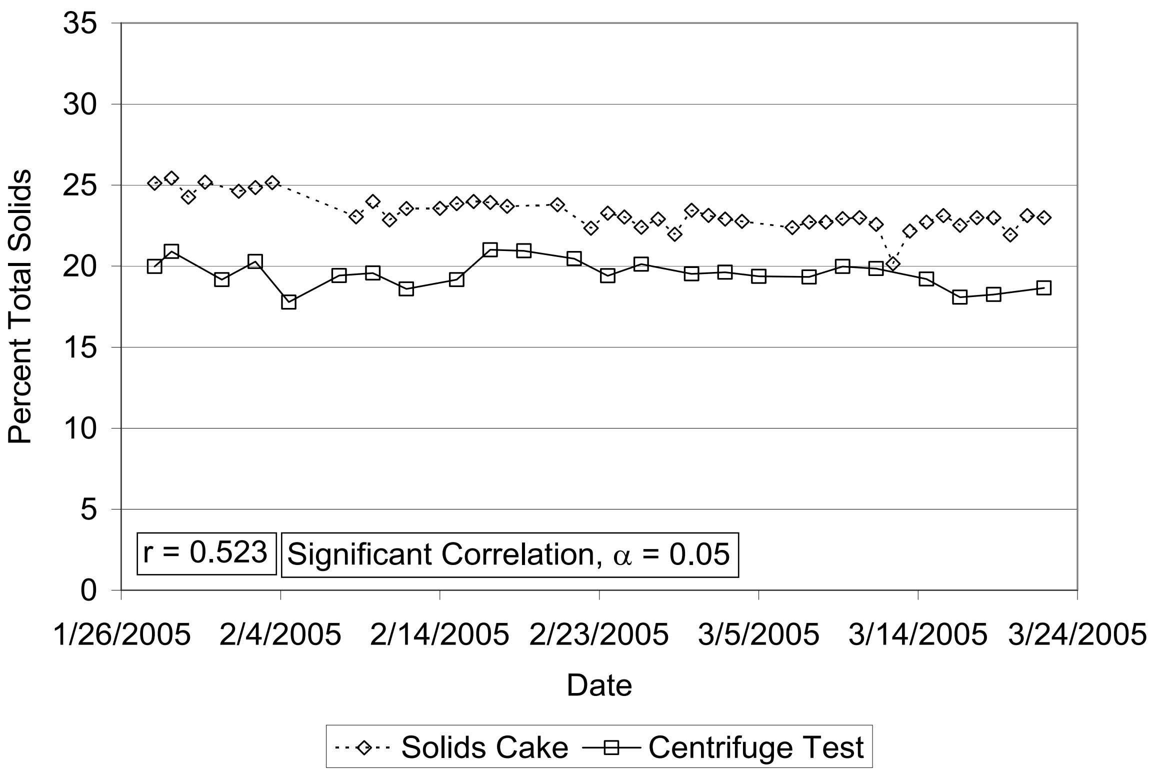

Figure 2.7, Archie Elledge wastewater treatment plant, correlation of centrifugal dewatering test.

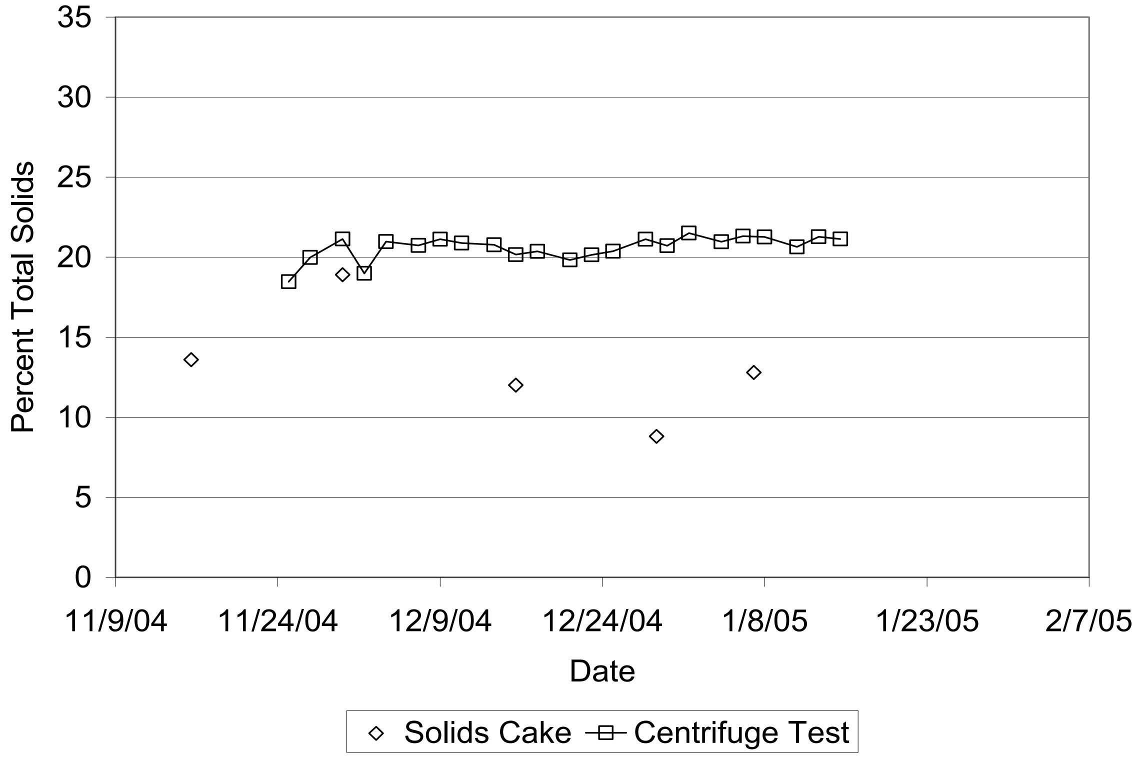

Figure 2.8, North Cary wastewater reclamation facility, correlation of centrifugal dewatering test.

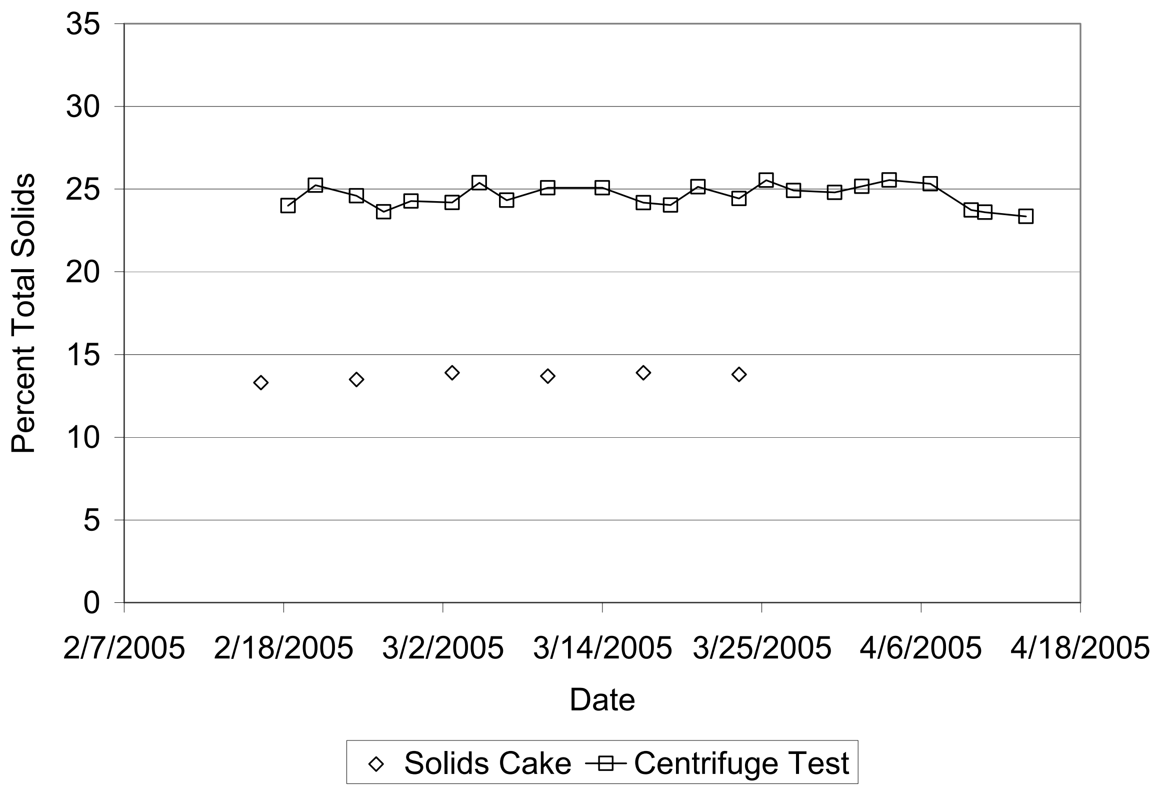

Figure 2.9, South Durham wastewater reclamation facility, correlation of centrifugal dewatering test.

Correlations between centrifugal dewatering tests and plant dewatering performance indicate that the centrifugal dewatering test could be used to predict full-scale plant dewaterability trends based on moisture distribution in sludge. Being able to predict dewatering performance in advance of digestion processes could help plant staff with decisions related to operational investment in dewatering equipment. In addition, plants may choose to modify existing design to increase dewatering potential. In the case of Barber et al (1995), operational changes were made to their activated sludge process based on sludge dewatering potential measured through their centrifuge test. This resulted in improved belt-filter press cake solids and decreased cationic polymer usage.

Another aspect of the centrifugal dewatering test is the idea that it can measure the upper limit of dewaterability for unconditioned sludge. This upper limit represents a sludge where all bulk and interstitial water has been removed and only bound water remains. Knowing this value could lead to a change in the approach to evaluating the performance of dewatering equipment. Instead of measuring dewatering efficiency by the amount of water a device is removing and adjusting equipment parameters to increase moisture removal, a plant could measure the percentage of removable water a device is removing. Full-scale dewatering data showed values that were consistently below centrifugal dewatering test values. This indicates that the difference between the centrifugal dewatering test and the plant dewatering results would be interstitial moisture that is not being removed through dewatering operations.

A summary of dewatering results including two-month averages of plant dewatering data and centrifugal dewatering test results for all eight plants is shown below in Table 2.2. The percent difference column is the percentage of the maximum unconditioned sludge dewatering efficiency (as measured by centrifugal dewatering test) that the plants dewatering equipment achieved. In cases where the percent difference is negative, the plants were able to remove more water than the centrifugal dewatering test predicted. Since the centrifuge test measures the dewaterability of unconditioned waste activated sludge it is possible through the use of sludge conditioners and polymers to increase the amount of water removable through mechanical dewatering.

Table 2.2. Results of centrifugal dewatering test and plant dewatering performance. Averaged over two-month period.

Treatment Plant	Abbreviation	Averge Centrifuge Test Result	Standard Deviation	Average Plant Dewatering Result	Standard Deviation	Percent Difference
Neuse River	NRWWTP	21.28	0.85	21.72	1.92	-2.10
North Cary	NCWRF	20.60	0.75	13.22	3.66	35.84
South Cary	SCWRF	22.38	0.84	16.42	0.77	26.64
Archie Elledge	AEWWTP	19.53	0.87	23.26	1.01	-19.09
James A. Loughlin	NSWWTP	21.57	0.89	20.89	1.03	3.15
South Durham	SDWRF	24.59	0.67	13.68	0.24	44.35
Town Creek	TCWWTP	29.13	1.22	19.28	2.81	33.79
McDowell Creek	MCWWTP	20.28	1.22	15.20	0.80	25.05

Two of the eight plants were able to achieve higher sludge dewaterability than the centrifugal dewatering test predicted (Neuse River and Archie Elledge). Achieving a dewaterability past the centrifugal dewatering test can be attributed to any number of reasons including, polymer optimization, sludge conditioning, and activated sludge basin design and operating parameters. Not surprisingly, these two plants also had the highest average dewatered cake solids of the eight plants. The two plants also have the highest treatment capacity of the eight indicating considerable investment in the sludge dewatering operations of their plants. Each of the remaining six plants had dewatering results that were less than the centrifugal dewatering test results indicating that they may not be maximizing sludge dewaterability. These six plants may be able to benefit from an evaluation of their wastewater treatment process and dewatering equipment.

When evaluating the differences between the centrifugal dewatering test and plant dewatering performance, many factors need to be considered. The centrifugal dewatering test measures dewaterability based on samples of waste activated sludge with the idea that dewaterability is a function of the entire activated sludge process, as well as dewatering equipment used. Accordingly, all parameters of the activated sludge process (mixed liquor suspended solids [MLSS], solids retention time [SRT], aeration basin configuration, clarifier performance) need to be considered before making any changes in dewatering strategy.

2.4 CONCLUSIONS

A centrifugal dewatering test was developed in an attempt to measure and predict the dewaterability of sludge from full-scale wastewater treatment plants. Significant correlations were established between the centrifugal dewatering test result and full-scale dewatering performance. Results indicate that it is possible to use centrifugal dewatering test results to predict trends in sludge dewaterability in advance of sludge handling processes such as digestion, thickening, and conditioning. The centrifugal dewatering test is potentially able to predict trends in plants using anaerobic or aerobic digestion. The test may also be able to predict trends in plants with a variety of dewatering equipment including centrifuges, belt-filter presses, and plate-and-frame press. The concept of dewaterability testing based on moisture distribution shows promise and merits further testing including longer testing time periods to allow tighter correlations. The concept of measuring the upper limits of sludge dewaterability needs to be explored further with a specific test developed for each plant being studied before any conclusions can be drawn.

Chapter 3 Disruption of Cellular and Floc Structures to Improve Dewaterability of Activated Sludge Through Changes to Moisture Distribution – Theory and Application

ABSTRACT

The ability of disruption techniques to improve the dewaterability of waste activated sludge was examined. Heat treatment, sonication, alkaline treatment, and cation addition were used alone, and in various combinations, to alter the distribution of moisture within waste activated sludge as measured by centrifugation and dilatometry. Disruption of sludge structure was measured by soluble protein content and cell lysis was measured by LIVE/DEAD epifluoresent stain. Sonication at 50 and 160 watts, as well as, heat treatment at 40, 60, and 80°C were evaluated for cell lysis, floc disruption, compactibility, dewaterability, and bound water content. Cation addition (Na^+, Mg^{2+}, and Fe^{3+}) was evaluated for changes in compactibility, dewaterability, and bound water content. Combinations of heat treatment-cation addition and sonication-cation addition were evaluated for compactibility, dewaterability, and cation addition. Sonication led to significant changes in moisture distribution of samples as evidenced by increased dewaterability and compactibility. Heat treatment led to insignificant changes in moisture distribution and no changes to compactibility or dewaterability. Cation addition alone led to changes in moisture distribution. The addition of cations had no additional effect, when used after heat treatment or sonication. Disruption techniques used in this study did not lead to large increases in cell lysis as was hypothesized, but rather, led to increases in soluble protein due to EPS breakup. The measurement of moisture distribution for assessing sludge dewaterability is a technique that shows promise, but needs to be investigated further.

3.1 INTRODUCTION

One of the least understood aspects of sludge dewatering is the structure of water within sludge. At the most basic level, water in sludge is considered free or bound (Vesilind, 1994; Kopp and Dichtl, 2001). Free water is able to move within floc structures and is unaffected by sludge solids. On the other hand, bound water is associated with sludge solids and cannot be removed by mechanical dewatering equipment. An existing hypothesis is that bound water represents the theoretical maximum limit of unconditioned sludge dewaterability through mechanical equipment (Vesilind, 1994). Therefore several researchers have explored the idea that bound water can be converted to free water and therefore removable through common dewatering operations.

To obtain a better understanding of bound water several researchers have developed classification systems for the water within sludge. Vesilind (1994) developed a classification system based on energy required to separate solids and liquids. His definitions are listed below.

1. Bulk water – water that is not associated with solid material and is easily removed
2. Interstitial water – water that is held within floc structures. Upon deterioration of floc this water is removed.
3. Vicinal water – water that is structured in layers according to electrostatic charge. Solid surfaces hold onto water molecules through hydrogen bonding. Upon deterioration of floc this water is not removable.

4. Water of hydration – water that is chemically bound to sludge particles and removable only through thermal input.

Accurate measurement of these portions of water within sludge is considered important to improving sludge dewatering. Techniques that convert bound water to free water can increase moisture removal by conventional dewatering equipment. Minimization of void structures within floc structure would lead to a decrease in interstitial water volume. Floc structure has been shown to be an important part in sludge bound water content and sludge dewatering (Lee, 1994A; Liao et al., 2000; Guan et al., 2003). Either by destroying floc structure or by shrinking floc structure to effectively remove pore space, bound water could be converted to bulk water and expelled from the floc structure.

Polymer conditioning has been shown to reduce bound water content and therefore improve sludge dewaterability. Chu and Lee (1999A) observed decreasing bound water concentration with polymer addition up to the charge neutralization point. Beyond that point, bound water increased as a result of further polymer addition. Katsiris and Kouzeli-Katsiri (1987) noted decreased bound water content following addition of chemical coagulants $FeCl_3$, $AlCl_3$, and $FeSO_4$. Wu and Wu (2001) noted decreases in bound water content following conditioning with cationic polymer. All three research groups hypothesized that bound water was decreased as water molecules were replaced by polymer molecules on the sludge surfaces.

The addition of cationic salts has been shown to change bound water content and improve dewaterability. Jin et al. (2004) noted improved dewaterability with high concentrations of

Ca^{2+}, Mg^{2+}, Fe^{3+}, and Al^{3+}. Novak et al. (2001) noted increased floc strength and improved dewaterability in the presence of iron. Choi and Chung (2000) and Choi et al. (2004) noted improved dewaterability and decreased bound water following leaching of divalent cations from humus soil amended activated sludge. Nielsen and Keiding (1998) observed a decrease in sludge dewaterability due to floc deterioration following the addition of sulfides. They hypothesized that ferric iron was reduced to FeS and therefore removed from the floc structure. Similarly, Bruus et al. (1992) saw decreased dewaterability following the removal of Ca^{2+} by ion exchange resin.

Tezuka (1969) theorized that divalent cations bridged negatively charged sites on sludge particle surfaces. He termed this the divalent cation bridging (DCB) theory for floc formation. Higgins and Novak (1997A, 1997B) discussed the use of the DCB theory for improved floc properties including sludge dewatering. In their experiments they noted that addition of monovalent sodium led to deteriorated floc structure and decreased dewaterability. Addition of divalent calcium and magnesium reversed this deterioration and improved sludge dewaterability. They concluded that for optimum dewatering the ratio of monovalent to divalent cations should be 2:1 and that dewatering is deteriorated when this ratio is exceeded. In a paper examining bioflocculation in the presence of cations, Sobeck and Higgins (2002) concluded that the DCB theory could best explain the role of cations in flocculation. They noted that deteriorating floc properties due to sodium addition could not be explained by other theories.

The physical disruption of sludge structure has been shown to change the partitioning of sludge water. Katsiris and Kouzeli-Katsiri (1987) observed a 30% decrease in bound water content following heat treatment of activated and digested sludge at 130°C. Lu et al. (2003) saw increased dewatering efficiency with the use of Fenton's reagent, a powerful oxidizing system. Chu et al. (2001) noted increased bound water content following sonication. Ormeci and Vesilind (2001) saw increased dewaterability in alum and activated sludges following freeze-thaw treatment.

Various methods have been developed for disrupting activated sludge structures. Heat treatment has been used previously to promote hydrolyzation of cellular structure in advance of digestion processes (Tanaka et al., 1997). Rocher et al. (1999) saw complete cell deactivation at temperatures above 55°C. Sonication has been shown to disrupt sludge and cellular structures by the process of acoustic cavitation (Yin et al., 2004). Gas and vapor bubbles generate, grow, and collapse violently leading to turbulent conditions. High temperature and pressure develop inside the collapsing bubbles, which break apart floc and cellular structures. Alkaline treatment of activated sludge has been shown to disrupt cellular structures (Vlyssides and Karlis, 2004). At high pH values, cell walls lose viability leading to leakage of intracellular material. Furthermore, high pH values can lead to a cell surface with an increasing negative charge resulting in the desorption of extracellular polymers (Katsiris and Kouzeli-Katsiri, 1987).

Disruption in activated sludge is commonly measured by soluble protein content. The main drawback in measuring disruption by protein analysis is that protein in activated sludge systems can be both intracellular and extracellular in nature. Sludges in activated sludge systems are known to contain various amounts of extracellular polymeric substances (EPS), a gel-like slime layer that surrounds the outside of microbial cells serves as a food source and is known to promote flocculation. Vallom and McLoughlin (1984) showed that cell lysis led to a release of intracellular polymers, which in turn became part of the cellular EPS and aided in flocculation Bura et al (1998) showed that protein accounted for approximately 85% of extracted EPS. Dignac et al (1998) arrived at a similar conclusion. Any material that resides inside a cell in an activated sludge system can also reside in the EPS of the cell as well and protein analysis alone cannot distinguish between the two.

Cell lysis is sometimes employed in wastewater treatment to improve sludge digestion by expediting the limiting step of cell hydrolysis (Camacho et al., 2002; Egemen et al., 1999; Sakai et at., 1997). It has been hypothesized that cell lysis can also improve sludge dewaterability by making intracellular water available for removal by dewatering equipment (Erdincler and Vesilind, 2000). Since biological sludges contain large numbers of cellular organisms, there is a great potential for moisture removal following cell lysis.

The purpose of this study was to determine if disruption of cellular and floc structures would have any affect on the distribution of moisture and sludge dewaterability as measured by dilatometry and centrifugation. Whereas most previous studies have focused on disruption of sludge structure to change dewaterability, it was our intent to demonstrate that cell lysis would have an effect on sludge dewatering. Furthermore, by using the newly developed centrifugal dewatering test, the simultaneous measurement of bound water and dewaterability would be possible.

3.2 METHODS

3.2.2 Samples

Samples of return activated sludge (RAS, approximately 2 gallons per visit) were collected from the North Cary Water Reclamation Facility. Samples were stored at 4°C and analyzed within 48 hours of sampling except in one case where a sample was stored for one week. Prior to use the samples were removed and allowed to come to room temperature (~24°C).

3.2.3 Disruption

<u>Heat Treatment</u>

Samples were heated to 40ºC, 60ºC, or 80ºC by placing a volumetric flask containing 200 ml of sample in a pre-heated waterbath (Fisher Scientific Isotemp model 228). The temperature of the samples was measured using a thermometer inserted into an extra sample. The samples were allowed to reach equilibrium temperature in the waterbath and held at that temperature for 5, 15, 30, 60, or 120 minutes. To eliminate variations in density due to temperature, sludge samples were allowed to return to room temperature following heat treatment.

<u>Sonication</u>

Sonication was performed using a Fisher Scientific 550 sonic dismembrator at a frequency of 20 kHz. Previous research has indicated that maximum disruption occurs at sonication frequencies of 20 – 40 kHz (Chu et al, 2001). Two levels of sonication were used. A low level sonication, at approximately 50 watts was used. This level represented the point at which sludge particles were visibly moving around in the sludge during sonication. A high level sonication at approximately 160 watts was also used. This level corresponded to the maximum intensity of sonication that could be reached without ejecting the sludge from the sample container. Each disruption test consisted of sonicating a 200 ml sample of RAS for time ranging from 10 seconds to 5 minutes. Slight increases in temperature were noted during extended sonication at 160 watts. All sonicated samples were allowed to return to room temperature before further tests were conducted.

Alkaline Treatment

Sludge pH was increased from 6.7 to 12.0 using 1M solutions of NaOH and KOH and samples were collected at pH 7.0, 8.0, 9.0, 10.0, 11.0, and 12.0. Sludge solutions were mixed constantly during pH adjustment using a stirring plate. To ensure that all disruption was due to pH and not due to monovalent cations, an equivalent amount of Na^+ and K^+ was added as chloride salt solutions to another set of samples. No significant disruption was noticed upon addition of salt solution indicating that all changes in sludge properties were due to pH increase.

3.2.4 Disruption Measurements

Soluble Protein Measurement

The level of sludge disruption that occurred during heat treatment, sonication, and cation addition was measured by comparing soluble protein levels. Studies have shown that protein analysis can be used to measure the degree of disruption in activated sludge systems (Schmitz et al, 2000). The amount of protein in solution following disruption was measured in triplicate.

Protein analysis was performed using the Bio-Rad RC-DC assay (Bio-Rad, Hercules CA). The assay is based on the Lowry method of protein measurement (Lowry et al, 1951) and has been shown to work well in the presence of numerous reducing agents and detergents that are commonly found in activated sludge systems. Solid material was separated from the bulk matrix by centrifugation at 10,000 x g for 5 minutes.

Cell Lysis

To determine if soluble protein released was intracellular or extracellular cell lysis was measured using the LIVE/DEAD fluorescence microscopy stain. The stain is a combination of Propidium Iodide and Syto 9. Syto 9 alone will penetrate cells with intact membranes and cause them to fluoresce green using epifluorescnt microscopy. These cells are termed LIVE. Cells with disrupted membranes will allow both Syto 9 and Propidium Iodide to penetrate and will fluoresce red, these cells are termed DEAD. Photographic images were obtained using a Photometrics Sensys charge-coupled device camera mounted on a Nikon Optiphot II fluorescence microscope. All images were taken under 400X magnification. Image analysis was performed using MetaMorph 5.0 software (Universal Imaging Corp., Silver Spring, MD). LIVE/DEAD stain has been used previously in activated sludge systems (Ramirez et al, 2000; Riley and Forster, 2001; Vollertsen et al, 2001; Seka et al, 2003). Images of RAS disrupted by heat treatment and sonication were obtained. Images were taken of RAS samples heated to 40, 60, and 80°C at periods of 0, 1, and 2 hours. Images of RAS samples sonicated at 50 and 160 watts for periods of 1 min, 2 min, and 5 min were also taken. For each disruption method tested, eight photos were taken from each of four prepared slides for a total of 32 images for each disruption.

Photographic analysis consisted of manually thresholding each image and measuring the total pixel area that was LIVE and the total area that was DEAD. The total area was then calculated by the summation of the two areas, and the percentage of lysed cells was calculated by dividing the DEAD area by the total area and multiplying by 100. Sample images for each treatment are included as Appendix II.

3.2.5 Cation Addition

All cations were added as chloride salt solutions. One molar solutions of K^+, Na^+, Ca^{2+}, Mg^{2+}, and Fe^{3+} chloride salt were prepared, and were added to 200 ml batch samples of RAS. To limit the effects of dilution, samples were allowed to settle for approximately 30 minutes and a volume of clear supernatant equal to the volume of chloride salt solution to be added was removed. Cation additions of 5, 15, and 25 meq were used. Following cation addition, samples were allowed to equilibrate for two hours. In tests where cation addition was combined with sludge disruption techniques, disruption was performed firs, then cationic solutions were added immediately and allowed to equilibrate for two hours.

3.2.6 Bound Water

Bound water was measured by the dilatometric method established by Smith and Vesilind (1995). Dilatometry is based on the principle that the freezing point of bound water differs from that of bulk water. Dilatometers measure the expansion as bulk water freezes. The total amount of water in the sample is calculated by measuring the percent total solids according to Standard Methods (APHA, 1998). The amount of frozen water is calculated by measuring the increase in volume due to freezing and using a predetermined coefficient of expansion for freezing water. The amount of bound water is then the difference between the total water and the frozen water.

Dilatometers (Ace Glass, part no. 6282-10) were modified to match those used in the established method. Figure 3.1 shows three of the dilatometers used in the study.

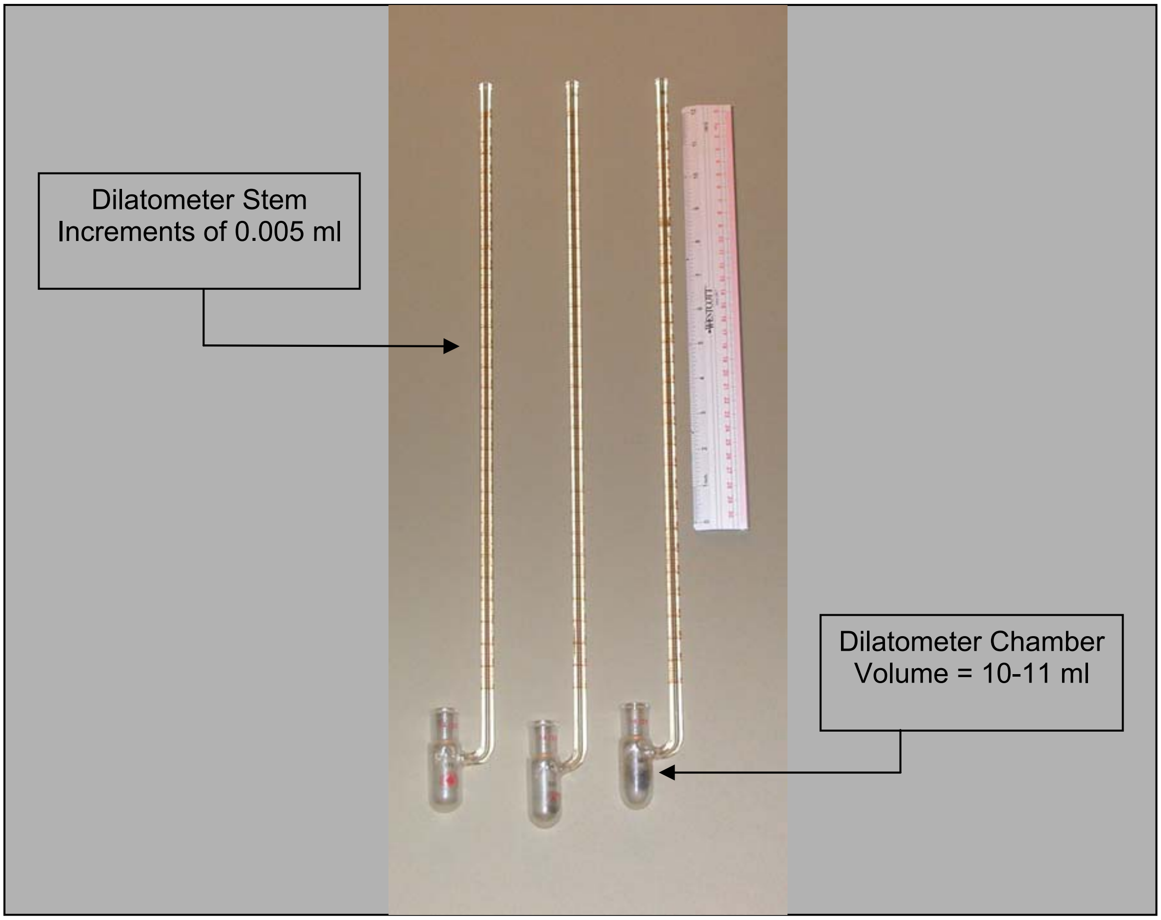

Figure 3.1. Dilatometers used in study

Low-temperature hydraulic oil was used as the indicator fluid. Samples of RAS were thickened by gravitational settling. The supernatant was then poured off to increase the percent total solids to at least 1% solids. Smith and Vesilind (1995) established that the dilatometric method is most accurate when using sludge between 1% and 3% solids. Dilatometers were filled with 5.0 g of sludge. Low-temperature hydraulic oil was added so that the head space was completely filled and no air was trapped in the chamber. The units were then placed in a –20ºC freezer. To measure the temperature of the dilatometers, a thermometer was placed in a container of comparable size to the dilatometer sample chamber that had been filled with low-temperature hydraulic oil. After approximately 3 hours, the volumes on each unit were read, the temperature was recorded, and bound water values were calculated. Table 3.1 below shows the calculations involved in determining bound water content in activated sludge.

Table 3.1. Dilatometry calculations for bound water measurement.

Measured Values	Abbreviation	Units	Determined
Hydraulic Oil	HO	g	Directly Measured by Balance
Initial Temperature	TI	°C	Directly Measured by Thermometer
Final Temperature	TF	°C	Directly Measured by Thermometer
Initial Dilatometer Level	DI	ml	Directly Measured on Dilatometer Stem
Final Dilatometer Level	DF	ml	Directly Measured on Dilatometer Stem
Indicator Fluid	IF	g	Directly Measured by Balance
Sludge Weight	S	g	Directly Measured by Balance
Fraction Dry Solids	ds	unitless	Standard Method for total dry solids
System Expansion	SysE	ml	Directly Measured on Dilatometer Stem
Temperature Change Upon Freezing	dT	°C	Directly Measured by Thermometer

Calculated Values	Abbreviation	Units	Equation Used
Temperature Change - Hydraulic Oil	dT HO	°C	=Ti - TF
Hydraulic Oil Level Change	HOLC	ml	= DF - DI
Unit Indicator Fluid Contraction	UIFC	ml / (gram °C)	= HOLC / (HO * dT HO)
Indicator Fluid Contraction	IFC	ml	= IF * UFC * dT
Expansion of Water Upon Freezing	EW	ml/g	Experimentally (1)
Sludge Expansion	SE	ml	= IFC * SysE
Total Water in Sludge Sample	W_T	g	$= S * (1 - ds)$
Frozen Water in Sludge Sample	W_F	g	= SE / EW
Unfrozen Water in Sludge Sample	W_U	g	$= W_T - W_F$
Bound Water	W_B	g/g dry solids	$=W_U / (S * ds)$

The Expansion of water upon freezing (EW) was determined experimentally. The EW coefficient was determined with filtered centrate from North Cary RAS samples. The value was measured by adding 5 g of filtered centrate to a known mass of indicator fluid. The unit was then placed in the freezer for 3 hours. At the end the expansion of the system was measured by subtracting the initial dilatometer level from the final level. The Indicator Fluid Contraction (IFC) was measured accordingly and the system expansion was calculated by subtracting the IFC from the final dilatometer level. The EW coefficient was then determined by dividing the system expansion (ml) by the total mass of water in the dilatometer (g). EW values were approximately 0.10 ml expansion/g frozen water and did not change significantly due to any sludge treatments used in the study. This coefficient of expansion is similar to previously reported studies where dilatometry was used to measure bound water in activated sludge systems (Wu et al, 1998; Smith and Vesilind, 1995; Liao et al, 2000). A detailed description of all dilatometry calculations involved can be found in Smith (1992)

3.2.7 Dewaterability

Dewaterability was measured using the high-speed centrifugal dewatering test described in Chapter 2. The test consisted of centrifuging a 30 ml sample at ~150,000 x g for 30 minutes and measuring the percent total solids in the remaining pellet. The centrifugal dewatering test removes all of the free and interstitial water from sludge samples so that only the vicinal water and water of hydration remain.

Dewaterability measurements following cation addition were made using percent volatile solids to account for the possible effect of increased cationic salts in the centrifuged pellet.

3.2.8 Compactibility

Sludge compactibility was measured through centrifugation. A 30 ml sample of RAS was pipetted into a 50 ml polyethylene centrifuge tube. Samples were then spun at 3000 x g for 30 minutes. Following each centrifugation, the tubes were removed, inverted, and the supernatant decanted immediately. The remaining pellet was carefully scraped out with a round-bottom scoopula and analyzed for total solids content according to Standard Methods (APHA, 1998). The compactibility test removes the free water and a portion of the interstitial water. A portion of the interstitial water remains, as well as the vicinal water and water of hydration.

Compactibility measurements following cation addition were made using percent volatile solids to account for the possibility of increased cationic salts in the centrifuged pellet affecting the results.

3.2.9 Time-to-Filter

Time-to-Filter (TTF) tests were conducted according to Standard Methods (APHA, 1998). The test was used in this study to asses the impact of sludge disruption treatment on filtration dewatering. The disruption techniques used in this study have been shown in previous research, to cause mechanical communition and decrease average particle size through creation of fine particles (King and Forster, 1990; Barjenbruch and Kopplow, 2003; Rocher et al., 2001). Increased amounts of fine particles have been shown to decrease dewaterability by clogging filters and screens (Bruus et al. 1992; Mikkelsen and Keiding, 2001). It was thought that sludge disruption techniques used in this study may lead to changes in sludge water content and phase as measured by centrifugation, but increases in fine particles may prohibit improved mechanical dewaterability due to filter clogging.

3.3 RESULTS AND DISCUSSION

3.3.1 Alkaline Treatment

Adjusting the sludge pH from 7 to 12 led to significant increases in soluble protein release as seen in Figure 3.2.

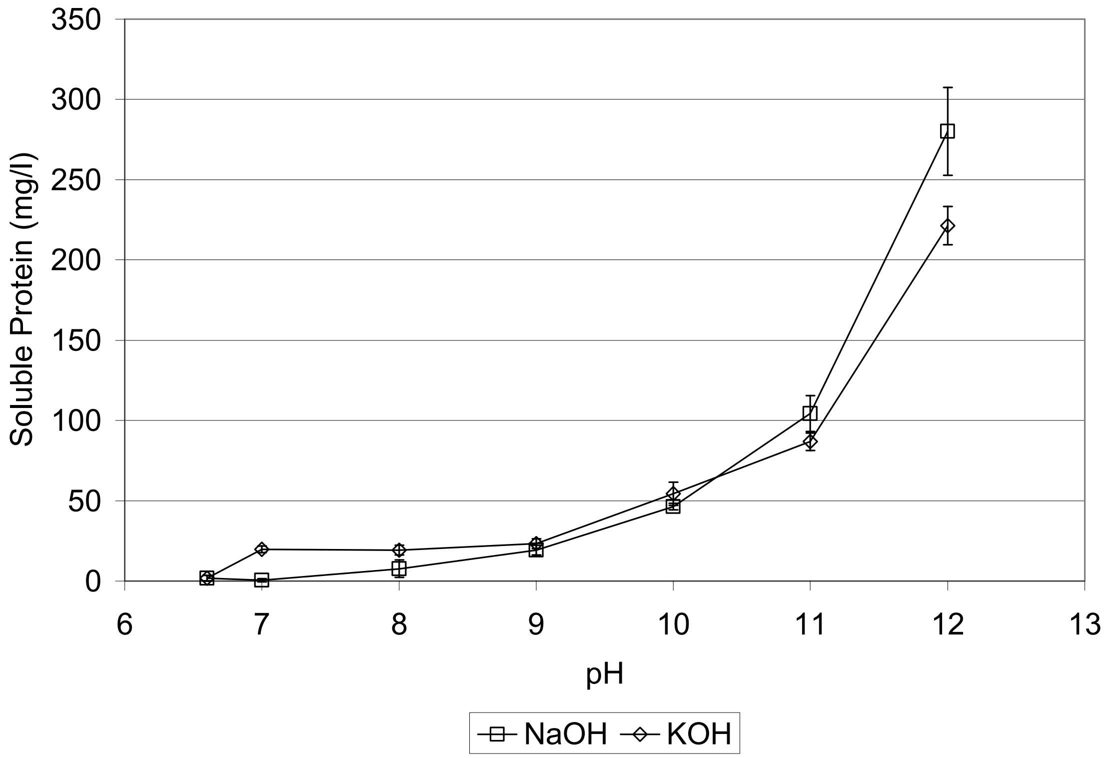

Figure 3.2. Soluble protein release due to alkali treatment

Alkaline treatment at pH 12 had the greatest soluble protein release of all disruption methods tested. However, measurements showed that a dramatic decrease in the compactibility of sludge subjected to alkaline treatment as shown in Figure 3.3.

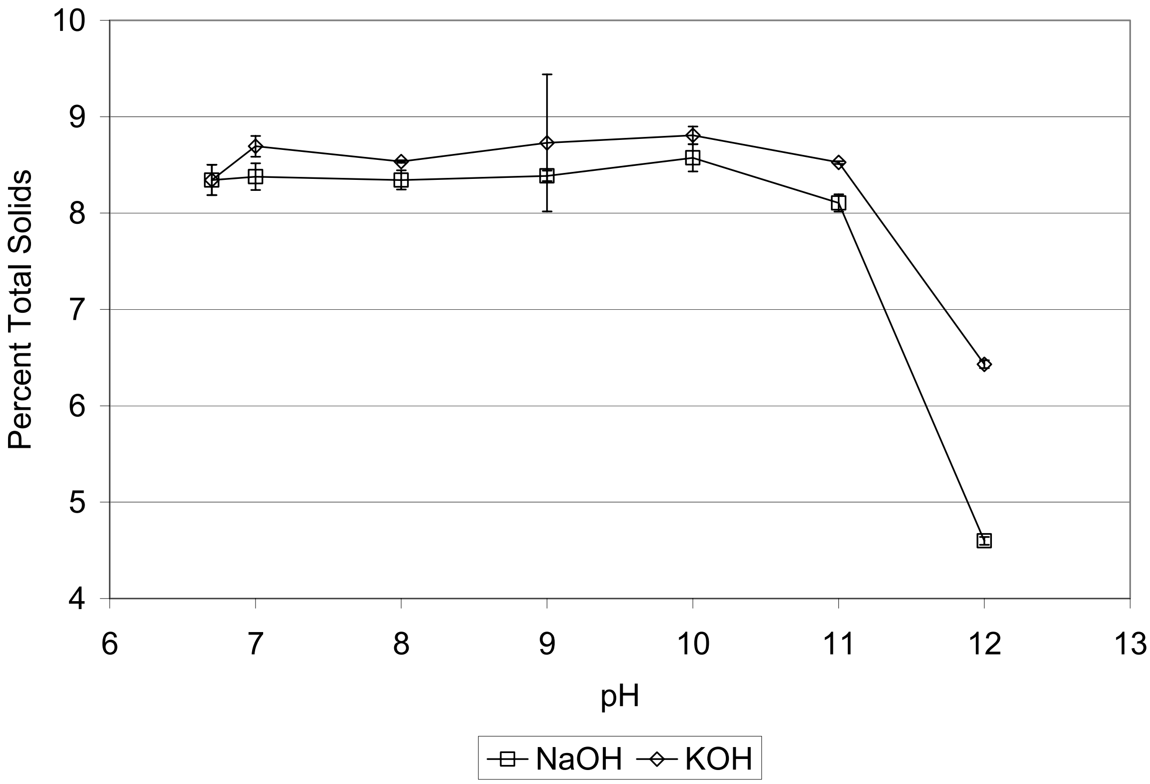

Figure 3.3. Compactibility test – Alkali treatment (error bars represent one standard deviation)

This large decrease in compactibility led to alkaline treatment being omitted from further tests on the basis that it would not improve dewaterability if it did not improve compactibility. The hypothesis is that the increasing sludge pH led to an increase in the net surface charge as theorized by Surucu and Dilek Cetin (1989). However, they saw decreased dewaterability and increased compactibility with adjustment only up to pH 9.0. In contrast, the decreased compactibility observed in this study supports the work of Tixier et al (2003) who saw an increase in sludge viscosity with increasing pH up to 12.0. There is a disagreement in the literature over the effects of pH change on sludge dewaterability and compactibility. The main problem is the lack of a standard test to measure these properties. With different research groups assessing dewaterability and compactibility using different tests, it becomes difficult to compare results and draw fundamental conclusions.

3.3.2 Heat Treatment

Heat treatment has the ability to disrupt floc and cell structure through hydrolysis of cell wall components. Soluble protein, cell lysis, and fine particle content were all measured. Temperature adjustment up to 40, 60, and 80°C led to significant increase in soluble protein content as shown in Figure 3.4.

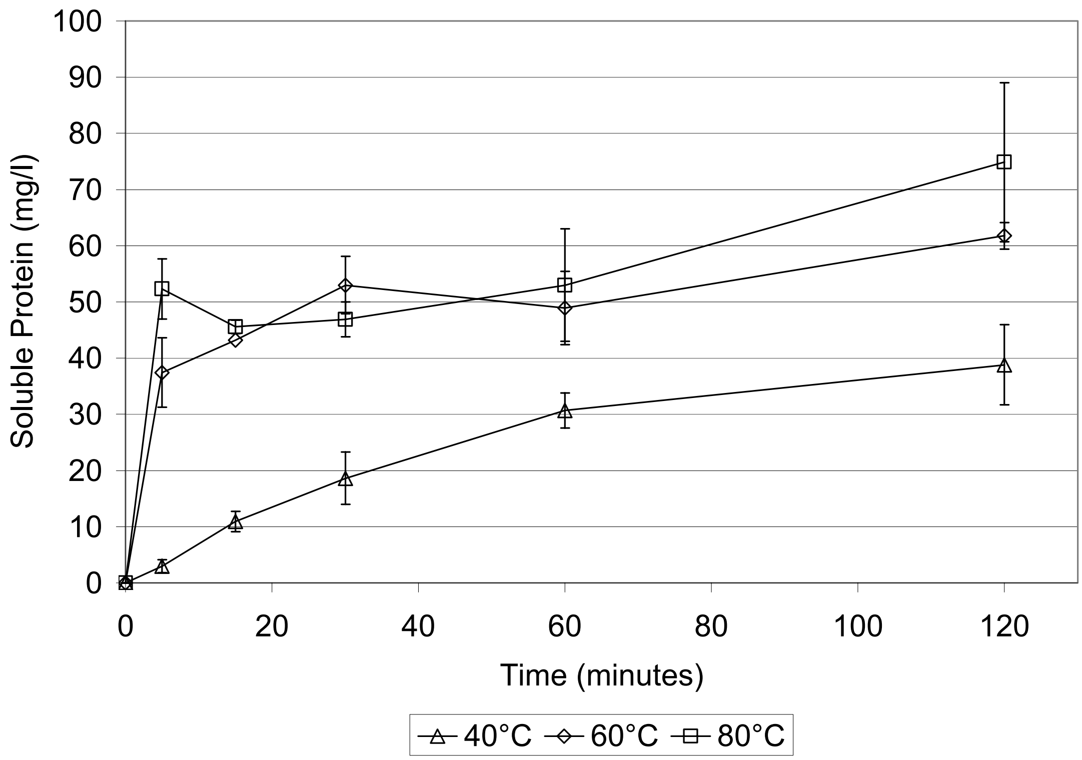

Figure 3.4. Disruption due to heat treatment as measured by soluble protein analysis.

Protein release at 60 and 80°C showed statistically similar results while treatment at 40°C had significantly lower results. Cell lysis as measured with LIVE/DEAD imaging showed increases in cell lysis compared to a control sample for 80°C only. Results are shown below in Figure 3.5.

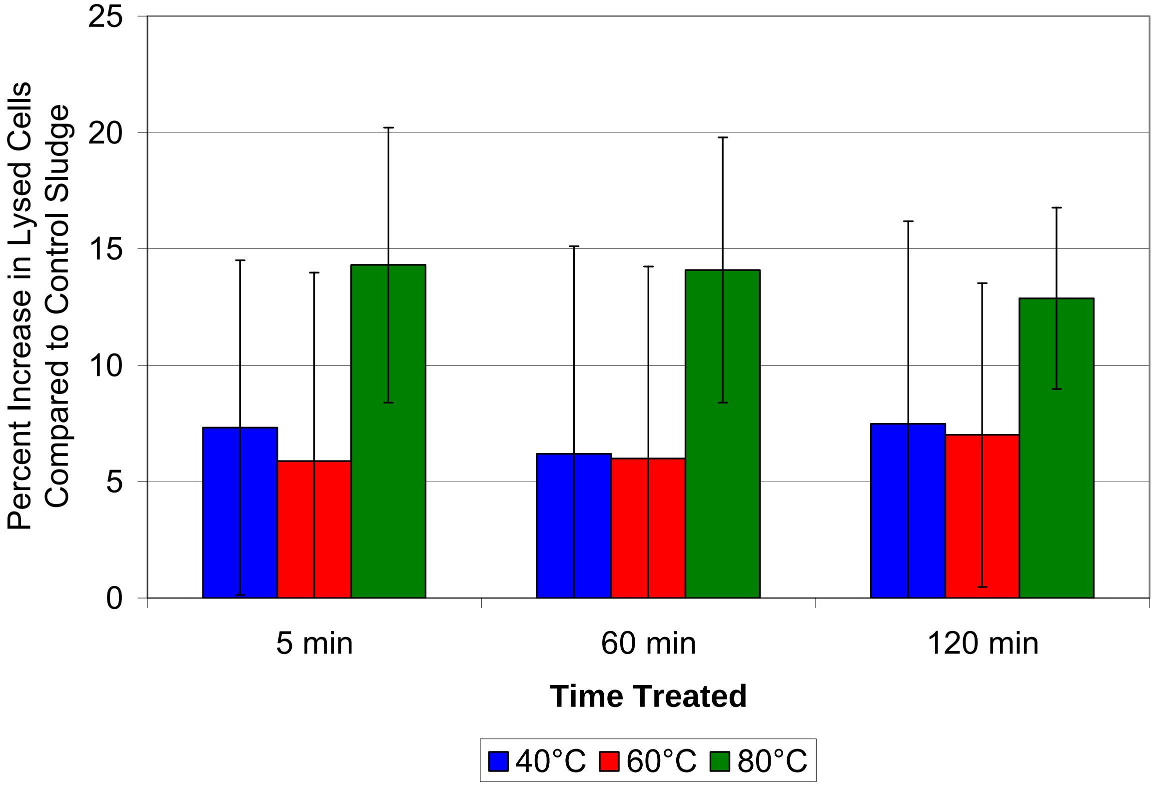

Figure 3.5. Increased cell lysis due to heat treatment as measured by LIVE/DEAD imaging.

The only significant increase in cell lysis over the control samples was seen at 80°C. There was no difference statistically between lysis measurements at 40 and 60°C compared to control sludges. There also was no significant difference between lysis measurements at 80°C and lysis measurements at 40 and 60°C.

A comparison of soluble protein data and cell lysis measurements shows that the protein released by heat treatment is mainly extracellular in nature, but at 80°C a significant portion could be intracellular. While soluble protein increases were seen at all time periods up to 120 minutes at 80°C, cell lysis values did not increase after 5 - 60 minutes at 60 and 80°C.. The release of soluble protein into bulk solution resulting from heat treatment has been seen before. Erdincler and Vesilind (2000) noted an increase in soluble protein following heat treatment at 120°C for five minutes. They did not speculate whether the protein was intracellular or extracellular. Neyens et al (2004) saw a release of protein into solution following thermal treatment at 120°C for 60 minutes. Her group believed that the protein released was extracelluar in nature and was part of the EPS. Tanaka et al (1997) saw an increased solubilization of protein following treatment at 130°C for five minutes in advance of anaerobic digestion but did not speculate whether it was intracellular or extracellular.

Fine particle content was measured through time-to-filter (TTF). Increased TTF indicates an increase in fine particles and a clogging of filter media. TTF results are shown below in Figure 3.6.

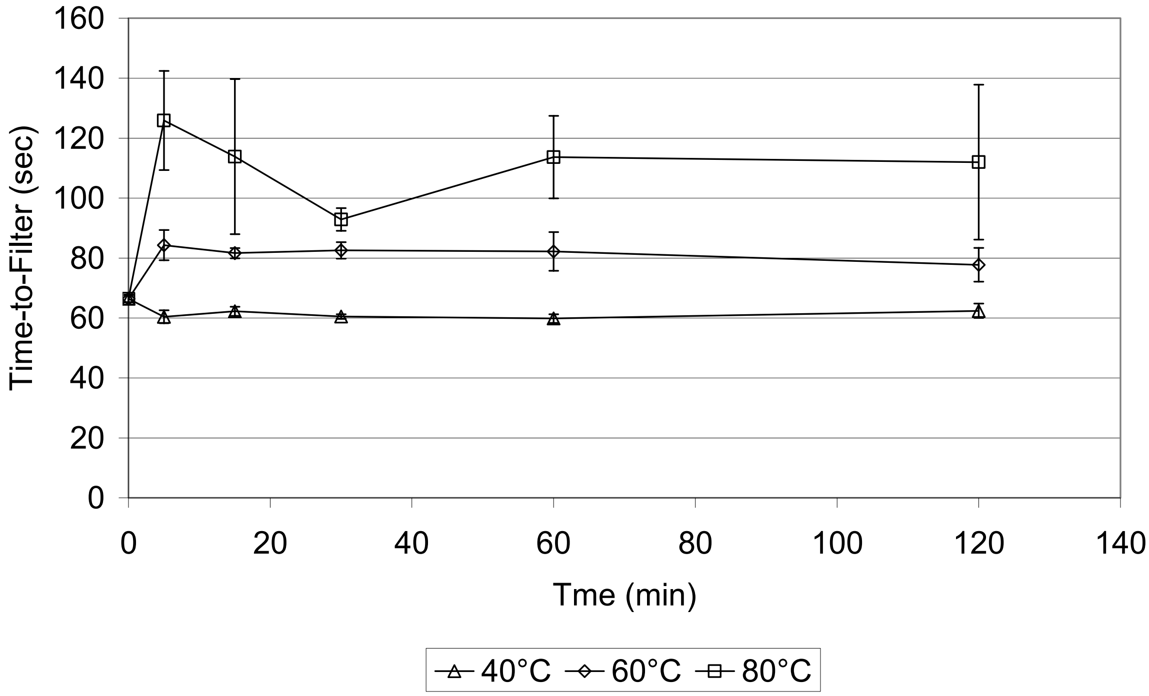

Figure 3.6. Time-to-filter values resulting from heat treatment.

Treatment at 40°C showed no significant change from the control sludge values (time = zero minutes). Treatment at 60 and 80°C showed significant differences from one another with 80°C having the largest increase. Both treatments were significantly higher than control values indicating that the number of fine particles increased following heat treatment. However, there was no correlation between protein release and TTF or between cell lysis and TTF.

Compactibility, dewaterability, and bound water were measured following heat treatment. The results are shown below in Figures 3.7, 3.8, and 3.9 respectively.

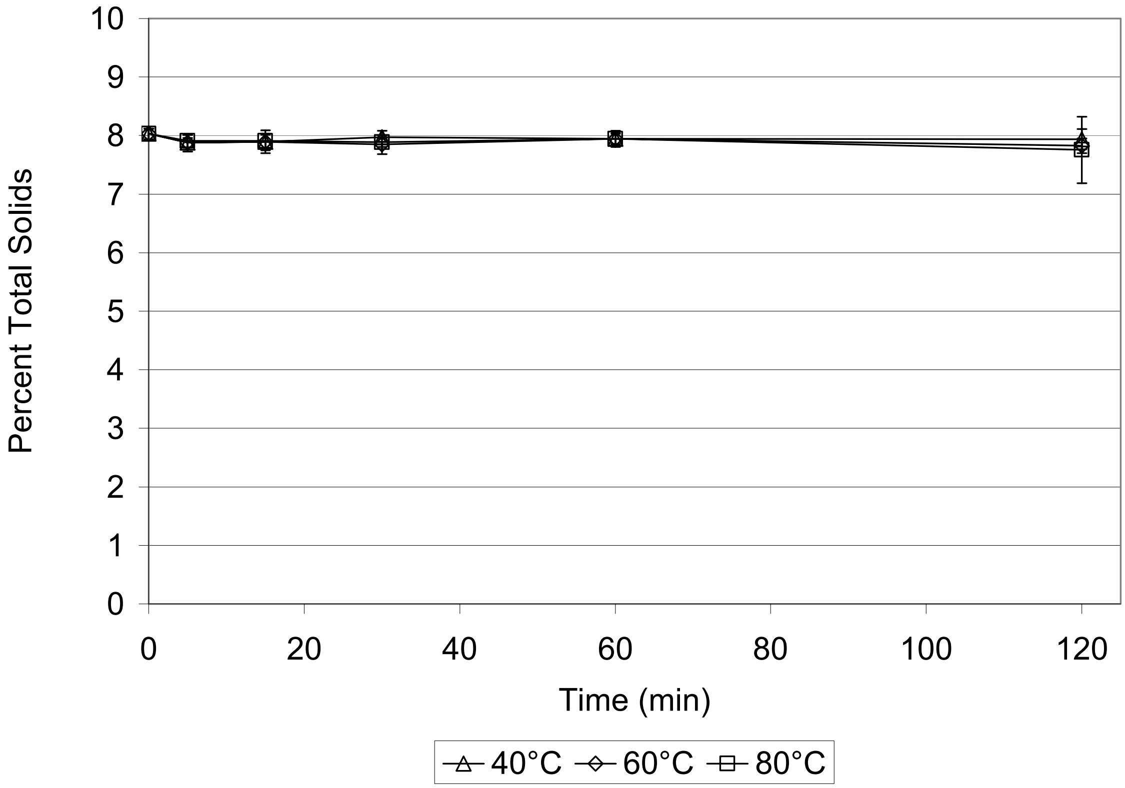

Figure 3.7. Compactibility due to heat treatment

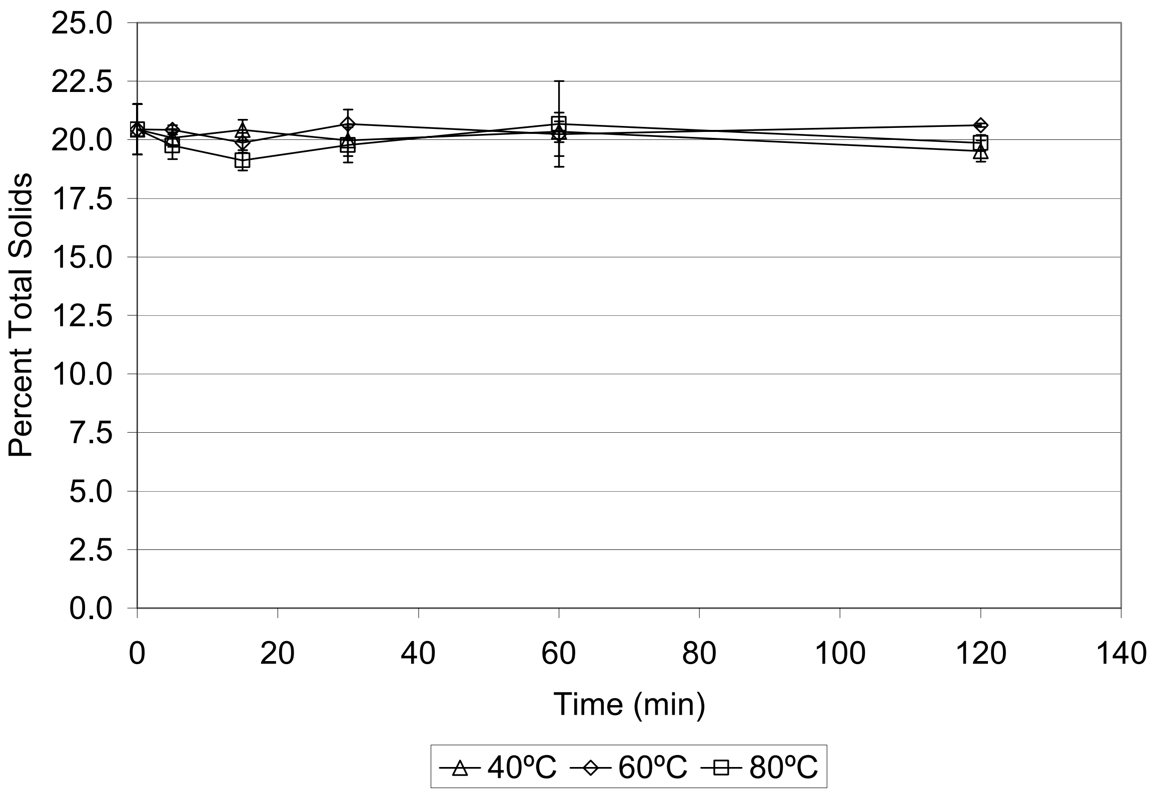

Figure 3.8. Dewaterability response to heat treatment.

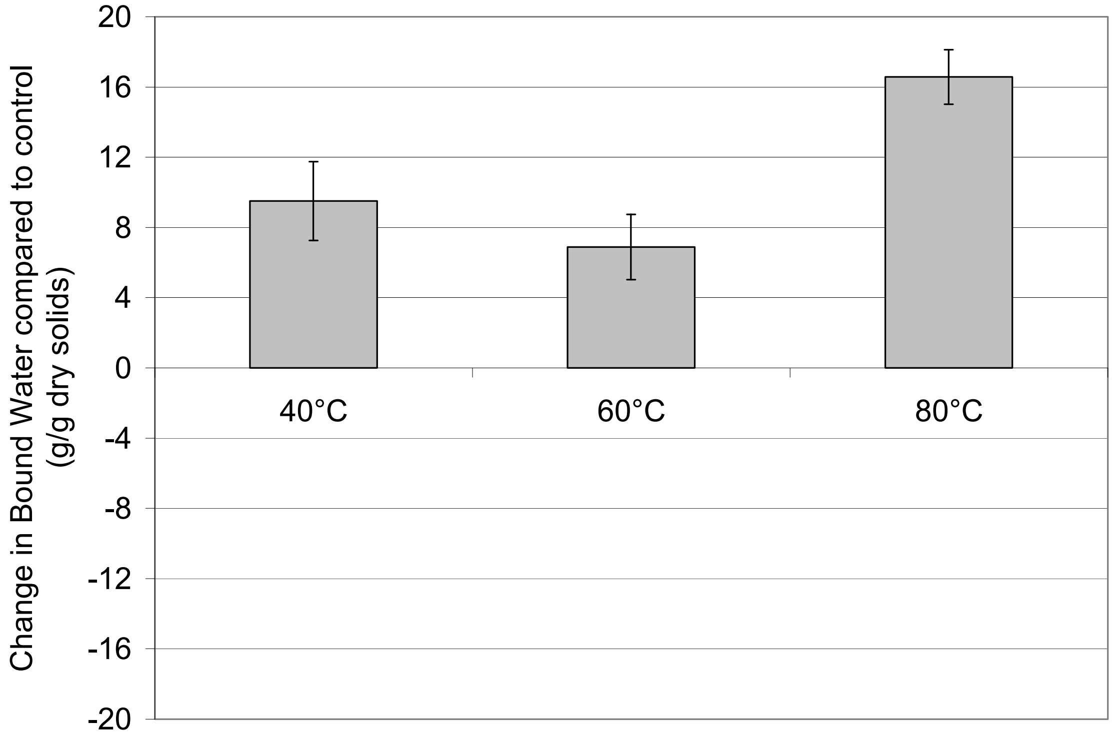

Figure 3.9. Bound water response to heat treatment as measured by dilatometry.

No significant change in moisture structure was indicated by compactibility or dewaterability measurements. Heat treatment did not change the quantity of vicinal water or water of hydration as indicated by centrifugal dewatering. Dilatometry measurements showed significant increases in bound water over control samples. The largest increases were seen with heat treatment at 80°C for 120 minutes. There are two possible explanations for the increase in dilatometric bound water observed following heat treatment; (1) heat treatment led to increases in interstitial water content as proteins were coagulated and this increase was not detected by compactibility measurements as the increased interstitial water was forced out during testing. (2) heat treatment led to increased soluble microbial products, which in turn caused a greater portion of the water to remain unfrozen during dilatometry measurements. The common explanation given in literature (Erdincler and Vesilind, 2000) that disruption leads to increased surface area for bound water formation due to floc breakup does not seem to fit. Changes in TTF values, indicative of small, if any, changes to floc size and structure should theoretically have no affect on bound water measurements. However, a significant increase in bound water was observed. The second explanation is supported by the work of Vesilind and Hsu (1997) who theorized that the release of soluble products and increased dissolved solids would lead to more unfrozen water during dilatometry tests. The dissolved products would become concentrated in the unfrozen water since the freezing front would move from the outside of the sludge floc to the inside thereby concentrating dissolved solids inwards.

The ability of heat treatment to affect the compactibility, dewaterability, and bound water content of sludge has been studied. Barjenbruch and Kopplow (2003) noted differences in dewaterability at 90°C and 120°C compared to a control reactor. They noted increased dewaterability at 90°C and decreased dewaterability at 120°C. They did not offer an explanation for these changes as the purpose of their research was to determine the effect of heat treatment on digester foaming, and dewaterability measurements were a side note. Surucu and Dilek Cetin (1989) noted decreases in dewaterability and compactibility following heat treatment above 25°C. Chu et al (2002) noted a decreased filterability accompanied by an increase of soluble EPS following liquid boiling.

A summary of the results of all measured parameters resulting from heat treatment is shown below in Table 3.2

Table 3.2. Summary of results following heat treatment for 120 minutes

Parameter	**40°C**	**60°C**	**80°C**
Soluble Protein	↑	↑↑	↑↑
Cell Lysis	↑	↑	↑↑
Time-to-Filter	—	↑	↑↑
Compactibility	—	—	—
Dewaterability	—	—	—
Bound Water	↑	↑	↑↑

↑↑ = Sharp Increase, ↑ = Increase, — = No Change, ↓ = Decrease, ↓↓ = Sharp Decrease

Cell lysis did not lead to any significant changes in dewaterability or compactibility. This indicates that water within cellular structures was not removed upon lysis. Bound water increase was possibly related to TTF values. This agrees with the explanation of Erdincler and Vesilind (2000) that heat treatment leads to increases in surface area on and around floc structures resulting in more available surfaces for bound water to form on. They noted increases in bound water following heat treatment. Similarly, Liao et al (2000) noted that an increase in bound water content correlated strongly with smaller median floc size indicating that there was a larger surface area available. However, exactly how the increase in floc surface area occurred is in question. Heat treatment may have led to coagulation of soluble microbial products and in turn, created more surface area. TTF values do not indicate a change in floc structure at 40°C, yet an increase in bound water was observed. The commonly used explanation has been that disruption leads to the breakup of floc structures and that the additional surface area is due to pieces of actual floc, not the creation of new surfaces. While this research does not conclusively show that new structures are created following heat treatment, it does raise the possibility and merits further examination.

There was no correlation between soluble protein content and compactibility or dewaterability. This conflicts with the research of Neyens et al (2003A, 2003B, 2004) who observed increases in dewaterability following thermal treatment of excess sludge combined with surfactant addition. She theorized that heat treatment led to a destruction of EPS components, which, in turn, made dewatering easier. However, results of this study showed that EPS is not destroyed, but it is solubilized.

3.3.3 Sonication

Protein analysis showed significant increases in soluble protein following sonication. Results are shown in Figure 3.10.

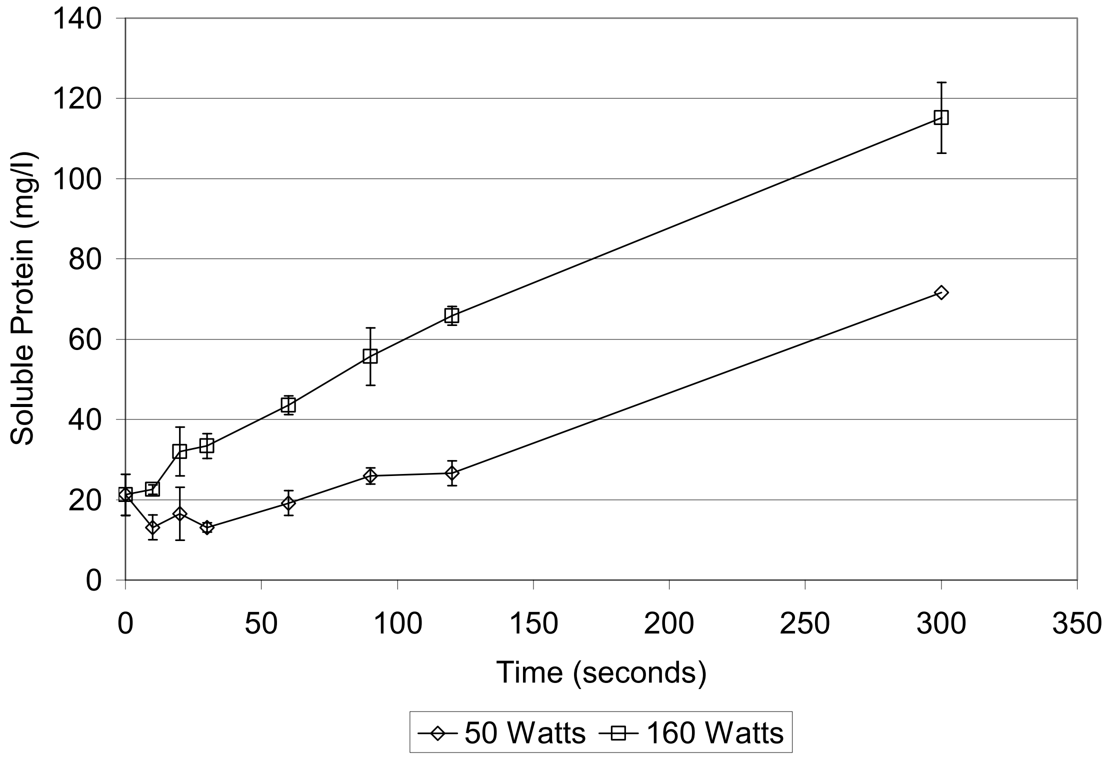

Figure 3.10. Disruption due to sonication as measured by soluble protein analysis.

Sonication at 50 watts and 160 watts produced significantly different levels of soluble protein. Significant increases in soluble protein levels over control samples were first seen at 20 seconds with 160 watt sonication and at 300 seconds for 50 watt sonication. Cell lysis values were measured for sonication at both levels at sonication times of 60, 120, and 300 seconds. Results are shown in Figure 3.11.

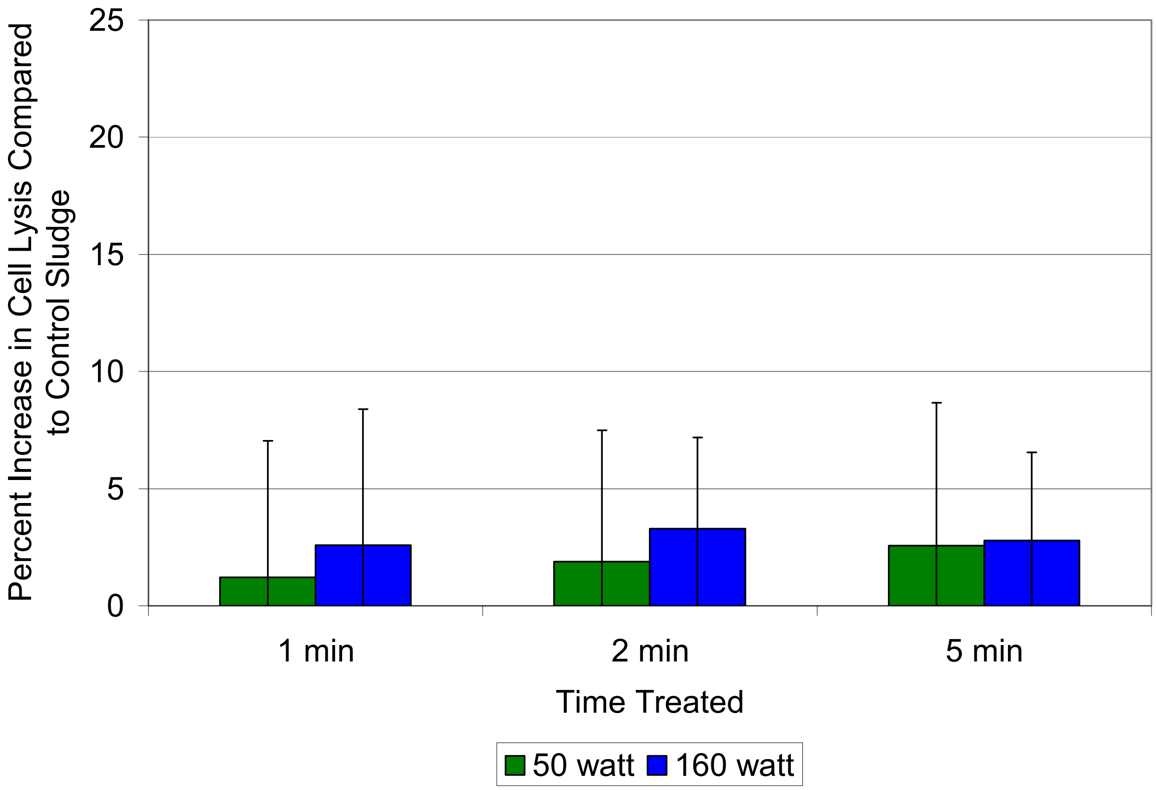

Figure 3.11. Cell lysis due to sonication as measured by LIVE/DEAD imaging.

None of the sonication values tested led to significant increases in lysed cells compared to control samples. There was no significant difference between sonication at 50 watts and 160 watts compared to the control. Protein release due to sonication was considered to be entirely extracellular because small, if any, increases in cell lysis were accompanied by large increases in soluble protein content. It has been demonstrated that sonication can remove extracellular products from activated sludge floc structure. Matais et al (2003) used sonication (40 watts) to extract EPS (including protein) from activated sludge samples. They concluded that sonication under 30 seconds would not cause significant cell lysis (as measured by total cell count) and removed considerable EPS including protein. Erdincler and Vesilind (2000) noted a significant increase in soluble protein following sonication (wattage not given) of *Flavobacterium* cells. However, they assumed that sonication would lead to cell lysis and considered the soluble protein to be entirely intracellular.

Fine particle content following sonication was measured using TTF. The results are shown in Figure 3.12.

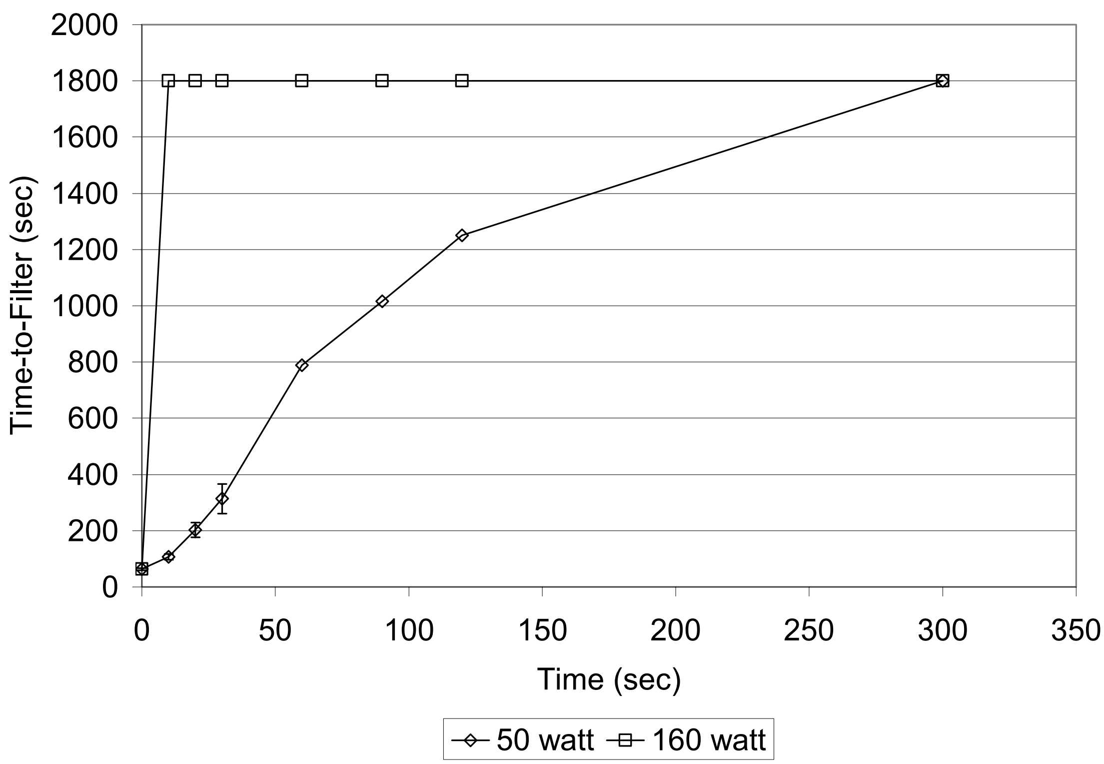

Figure 3.12. Time-to-filter results from sonication.

Time-to-filter values showed steady increase at 50 watts. However, a marked increase was noted at 160 watts (TTF values were measured up to a maximum value of 30 minutes or 1800 seconds). Sonication has been known to deteriorate floc structures and decrease average floc size. Jorand et al (1995) and Morgan and Forster (1992) sonicated samples of activated sludge and noticed a general trend toward smaller average particle sizes with longer sonication times. The presence of smaller particles would lead to decreased filterability.

Significant increases in compactibility and dewaterability, as well as a significant decrease in bound water content all with sludge treated with sonication. Compactibility, dewaterability, and bound water content results are shown below in Figures 3.13, 3.14, and 3.15 respectively.

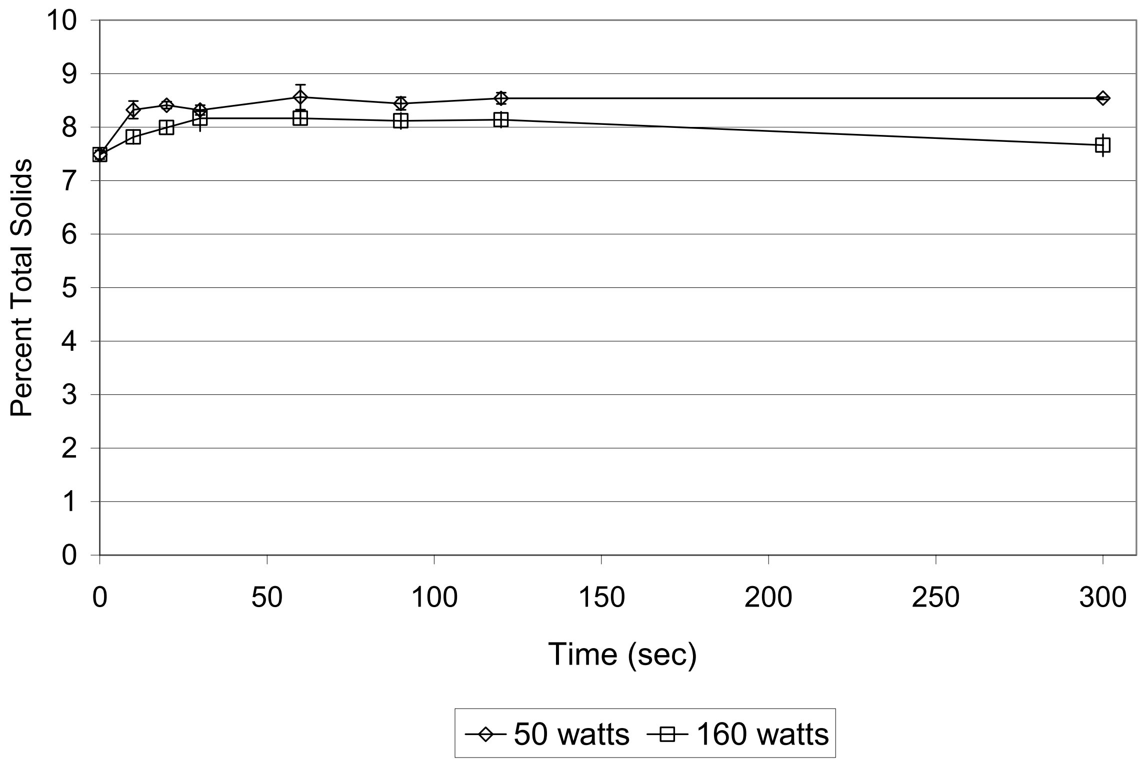

Figure 3.13. Compactibility results following sonication. Error bars represent one standard deviation

Significant increases in compactibility were seen following sonication at 50 and 160 watts after 30 seconds. This is in agreement with the work of Erdincler and Vesilind (2000) who noted an increase in compactibility of sludge samples following sonication. Bien et al (1997) also noted a decrease in water content of sludge cake and decreased resistance to filtration following sonication.

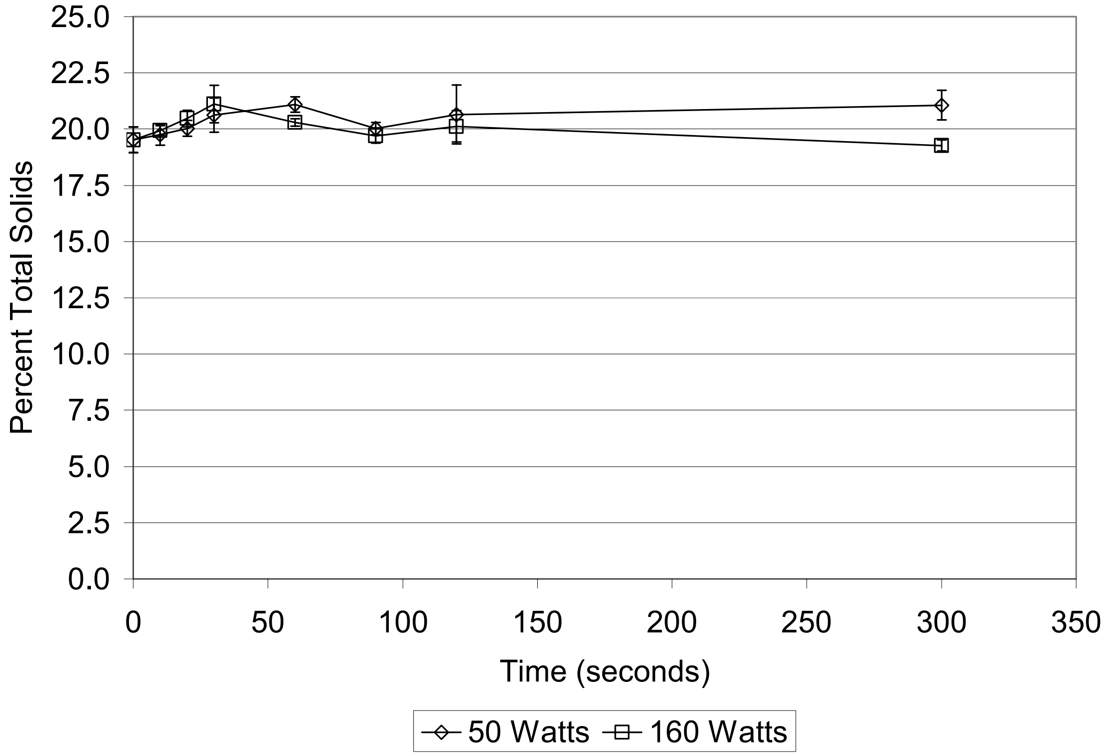

Figure 3.14. Dewaterability results following sonication. Error bars represent one standard deviation.

Significant increases in dewaterability were seen following sonication at 50 watts and 160 watts for 30 seconds. Increased dewaterability is not often seen following sonication. This is due largely to the fact that previous tests to measure dewaterability in fact measure filterability. Both Chu et al (2001) and King and Forster (1990) noted decreased filterability of sonicated sludge samples.

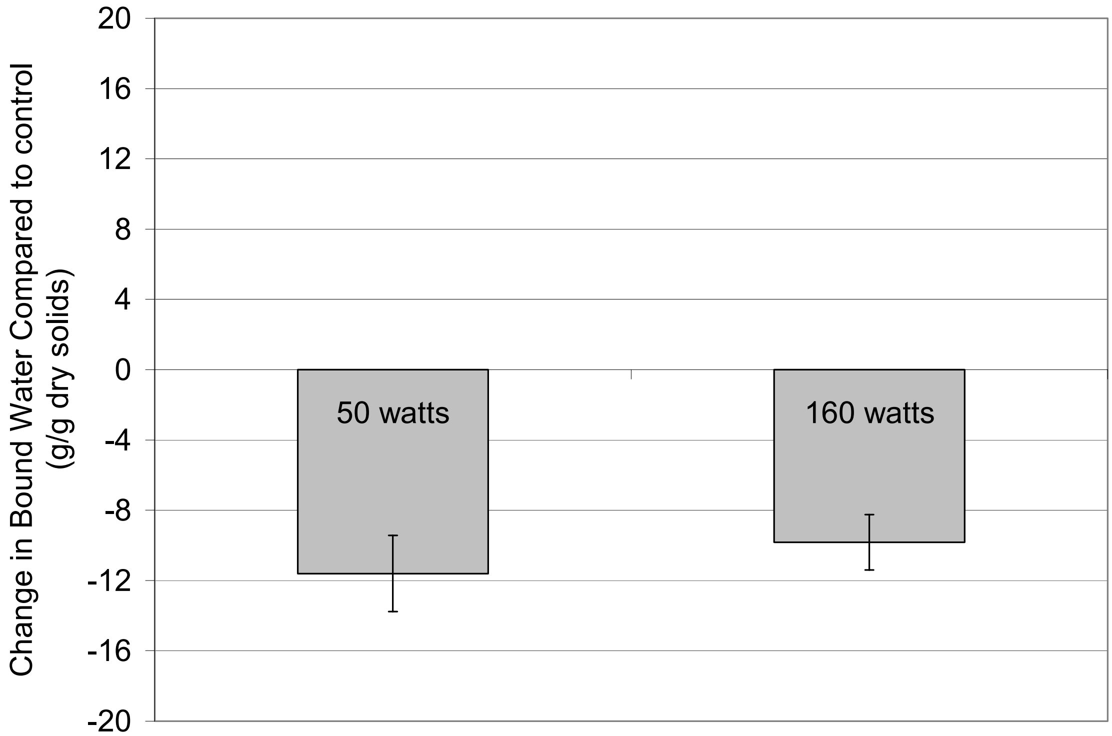

Figure 3.15. Bound water content following sonication. Error bars represent one standard deviation

Bound water content decreased significantly following sonication. A possible explanation for the decreased bound water content could be the evacuation of interstitial and vicinal water due to loss of floc structure and input of sonic energy. Decreases in interstitial water could lead to decreased bound water as it is hypothesized that dilatometry measures interstitial water as part of bound water (Vesilind, 1994). This decrease in bound water content also explains the increased dewaterability measurements as more water is now in the bulk form. A summary of all measured parameters is shown in Table 3.3

Table 3.3. Summary of results following sonication for 60 seconds

Parameter	50 Watts	160 Watts
Soluble Protein	—	↑↑
Cell Lysis	—	—
Time-to-Filter	↑	↑↑
Compressibility	↑	↑
Dewaterability	↑	↑
Bound Water	↓↓	↓↓

↑↑ = Sharp Increase, ↑ = Increase, — = No Change, ↓ = Decrease, ↓↓ = Sharp Decrease

As was the case with heat treatment, there was no correlation between cell lysis and dewaterability or compactibility. Compactibility and dewaterability showed similar increasing trends. Increases in compactibility and dewaterability can be attributed to the removal of interstitial water and vicinal water caused by the breaking apart of floc structures and input of sonic energy. TTF values indicate a sharp increase in the number of fine particles following sonication at both 50 and 160 watts. Such a change in floc structure could lead to a release of interstitial portions of sludge water and in turn, create increases in compactibility and decreased dilatometric bound water. Loss of vicinal water is seen by the increase in centrifugal dewatering test results following sonication. Such increases indicate that vicinal water is separated from the sludge solids and is then available for removal. The loss of floc structure could also explain the reason why bound water values dropped sharply following sonication as it is thought that dilatometry measures interstitial water as a part of bound water.

TTF measurements indicate a radical shift towards smaller particles following sonication, whereas heat treatment did not cause such a shift. This difference in fine particles is what makes the difference between heat treatment and sonication. Sonication caused a thorough destruction of floc structure while heat treatment left floc structure intact. Again, the prevalent theory of disruption leading to increases bound water through creation of additional surfaces does not seem to hold up.

While it is possible that heat treatment led to creation of new surfaces through SMP coagulation, it is unlikely that sonication would do the same. The lack of additional created surfaces would mean less formation of bound water following sonication, which is what was seen. The destruction of floc structure would also not allow for the measurement of interstitial water. The decrease in bound water measured by dilatometry indicates that it may preferentially measure the interstitial portion of water as has been hypothesized by Dick and Drainville (1995)

3.3.4 Cation Addition

Cations were added as chloride salts to 200 ml samples of activated sludge. Soluble protein measurements were taken after addition of salt solution. There was no significant increase in soluble protein following cation addition (data not shown). Cell lysis measurements were not taken following cation addition. TTF measurements were taken following cation addition. The results show a trend of decreasing TTF with increasing cation valence state. TTF results are shown in Figure 3.16.

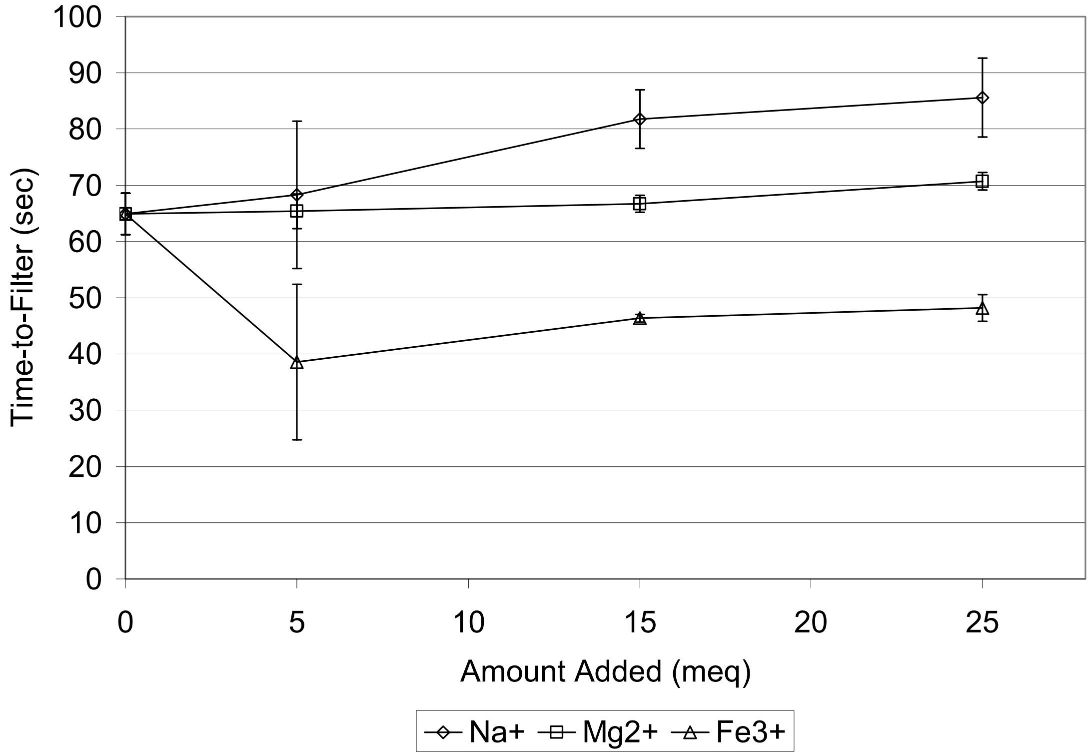

Figure 3.16. Time-to-Filter results following cation addition. Error bars represent one standard deviation.

Addition of monovalent sodium led to significant increases in TTF values while trivalent iron led to decreases in TTF values. Divalent magnesium led to no significant changes when compared to control values.

Compactibility, dewaterability, and bound water content were all measured following cation addition. It should be noted that for compactibility and dewaterability measurements, percent volatile solids was measured instead of percent total solids. This was done so that any cationic salts remaining in the centrifuged pellet would not skew the results of total solids analysis. Since the same sludge was used in each test, it was assumed that the fraction volatile solids for each sample was the same so the percentage of volatile solids remaining after centrifugation should be a reflection of the total solids contained in the centrifuged pellet. Results of compactibility, dewaterability, and bound water are shown in Figures 3.17, 3.18, and 3.19 respectively

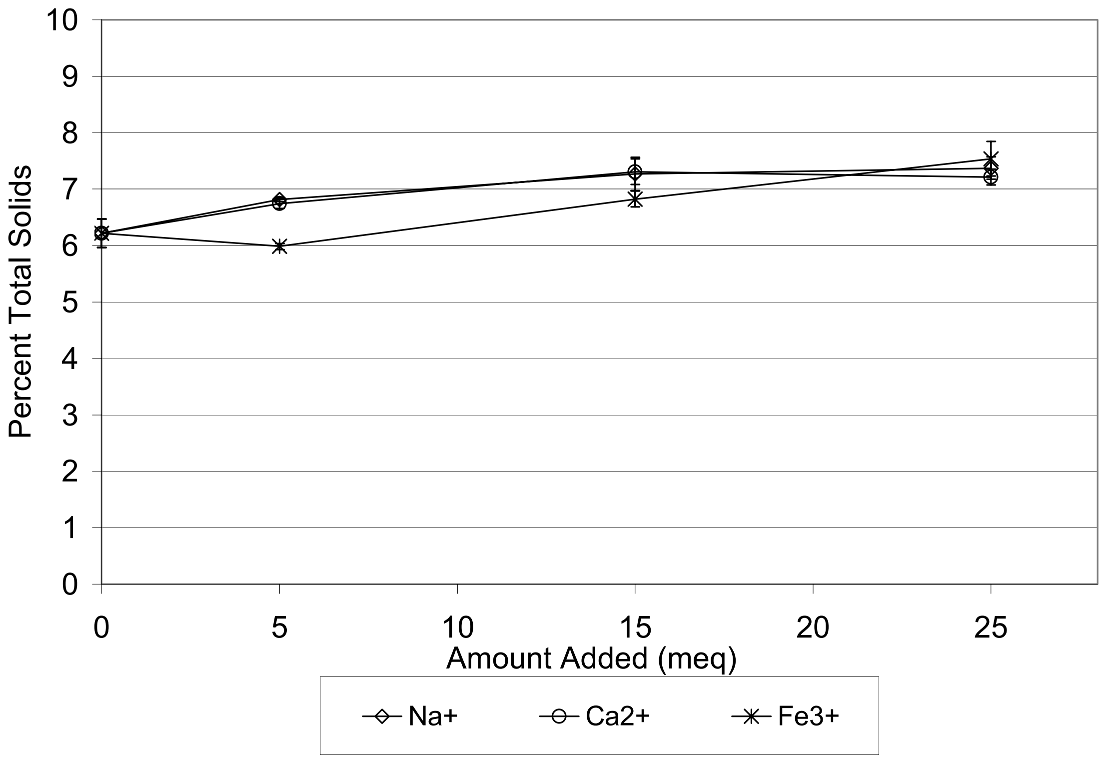

Figure 3.17. Compactibility following cation addition. Error bars represent one standard deviation.

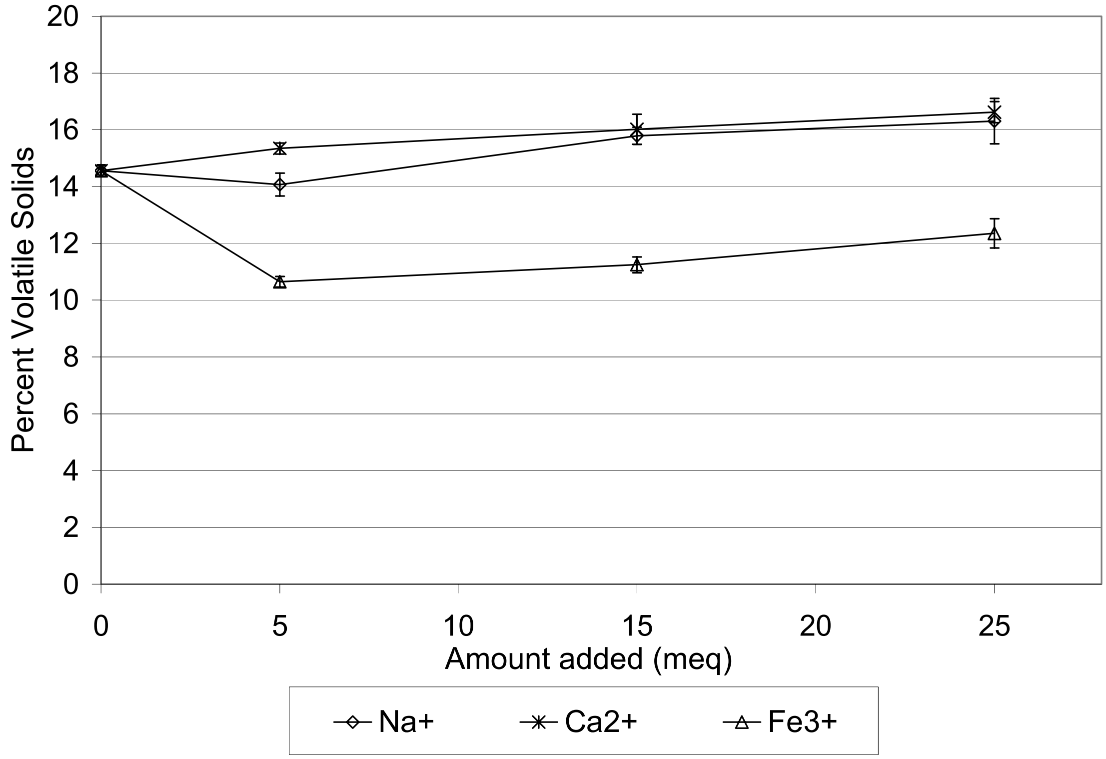

Figure 3.18. Dewaterability following cation addition. Error bars represent one standard deviation.

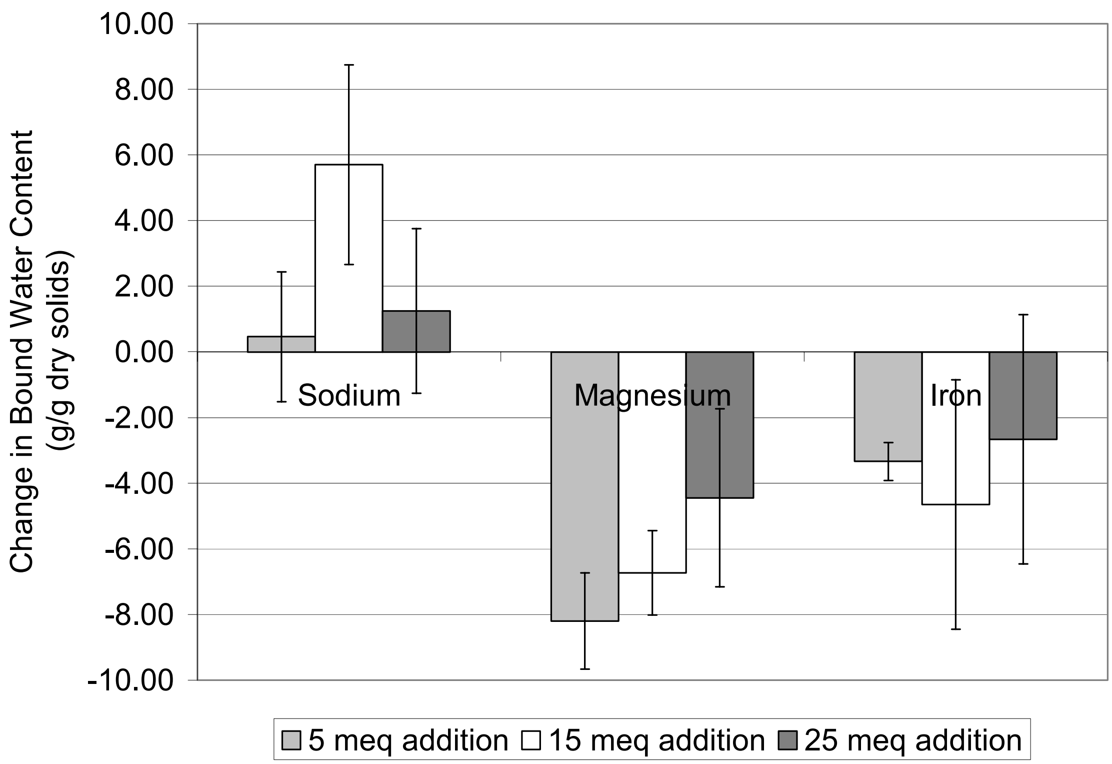

Figure 3.19. Change in bound water content following cation addition. Error bars represent one standard deviation.

Addition of monovalent and divalent cations led to small increases in compactibility. Addition of trivalent iron led to significant increase only with 15 and 25 meq additions. Significant increase was seen in dewaterability resulting from monovalent or divalent cation addition at 15 and 25 meq. Addition of trivalent iron led to significant decreases in dewaterability. However, based on visual inspection of the centrifuge tube it was concluded that the problem was with the ferric chloride addition leading to the sludge sample sticking to the side of the tube and therefore leading to reduced dewaterability. Therefore, the reduction in dewaterability is a function of the centrifuge tube and added chemicals and not an indication of how sludge treated with ferric chloride would react to dewatering equipment.

Bound water content showed either no change or significant increase following monovalent sodium addition while significant decreases were seen when divalent magnesium and trivalent iron were added.

Table 3.4 lists a summary of the properties of sludge samples following cation addition.

Table 3.4. Summary of valuess following cation addition.

Parameter	Monovalent	Divalent	Trivalent
Soluble Protein	—	—	—
Cell Lysis	*	*	*
Time-to-Filter	↑	—	↓
Compressibility	↑	↑	—
Dewaterability	—	—	**
Bound Water	↑	↓	↓

↑↑ = Sharp Increase, ↑ = Increase, — = No Change, ↓ = Decrease, ↓↓ = Sharp Decrease,
** = Not Measured*
*** = test was unable to measure due to compatibility issue*

Changes in TTF and bound water are in agreement with the DCB theory of Tezuka (1969) and Higgins and Novak (1997A, 1997B). Addition of sodium led to a deterioration of floc properties, which would cause an increase in fine particles and a reduction of filtration dewatering. Floc deterioration would also lead to increased bound water as measured by dilatometry due to increased floc volume due to expansion. Trivalent iron addition would improve floc structure by increasing its strength and possibly reducing the size as it cross-links surface sites tighter according to Pollack (2001). Trivalent iron addition would also push out interstitial water as the floc structure is compressed due to tighter cross-linking of surface sites. Addition of divalent cation would be expected to improve floc properties as well. However, no change was observed which cannot be explained with the available data.

3.3.5 Combination Study – Heat Treatment and Cation Addition

Disruption methods and cation addition were tested in combination to determine if there were any combined effects. The hypothesis behind the combination studies was that by disrupting sludge and cell structures bound water can be converted to free water, and therefore be removed through dewatering equipment. Cations were added to bridge negatively charged floc structures and promote flocculation. We hypothesized that following disruption, cation addition would lead to more stable floc structures and ultimately squeeze out more water from sludge solids. Compactibility measurements were made on heat treatment combined with cation addition. Sodium, magnesium, and iron were each added at 5, 15, and 25 meq to 200 ml samples of WAS heat treated to 40, 60, and 80°C for 2 hours. The results for sodium, magnesium, and iron are shown in Figures 3.21, 3.22, and 3.33 respectively.

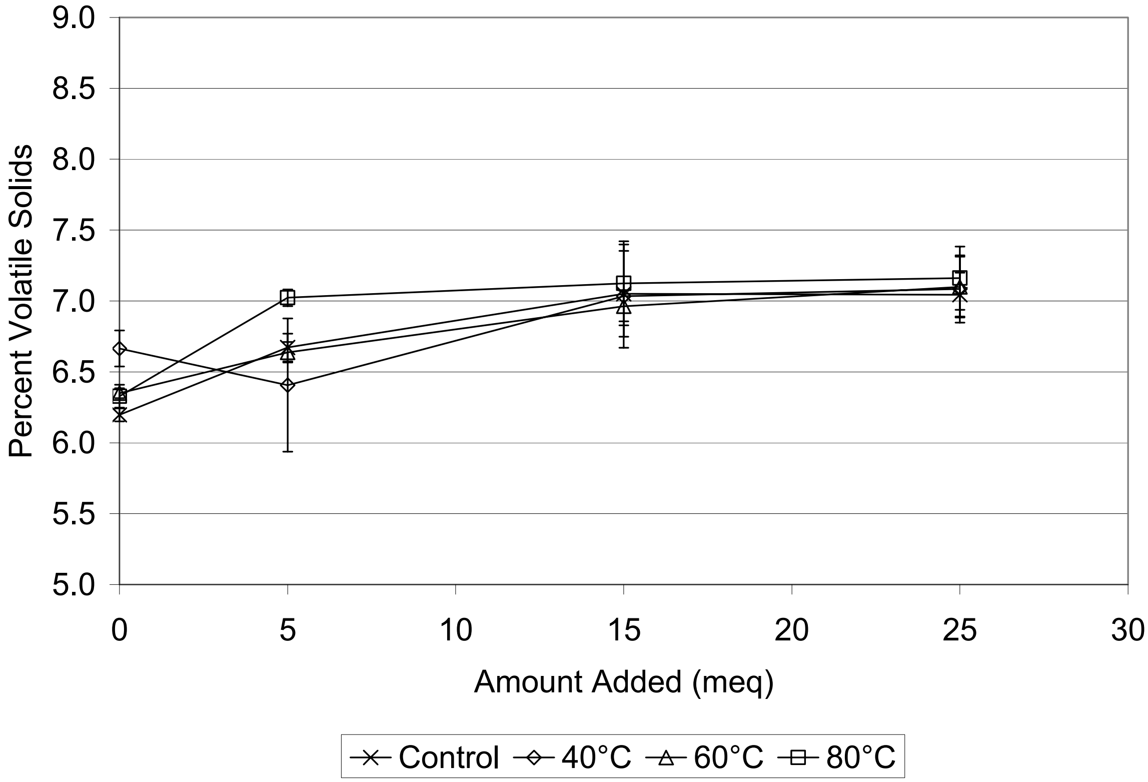

Figure 3.20, Compactibility measurements, heat treatment combined with sodium addition

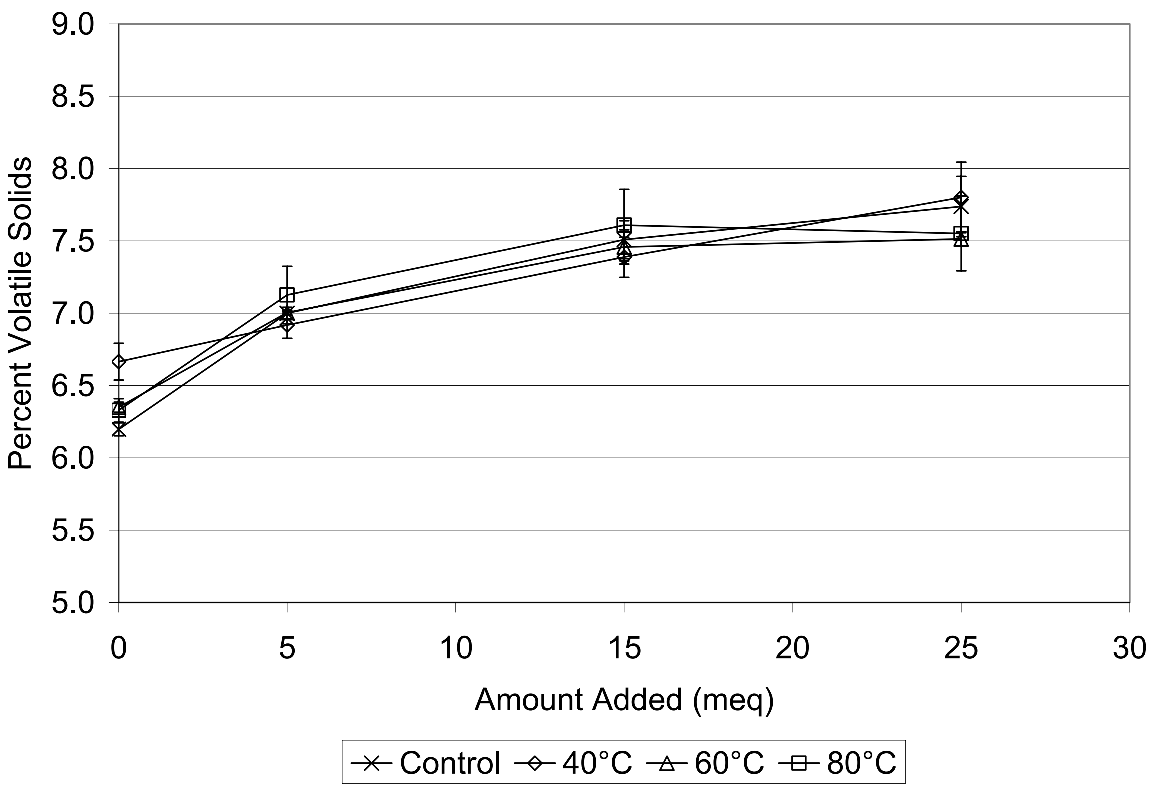

Figure 3.21, Compactibility measurements, heat treatment combined with magnesium addition.

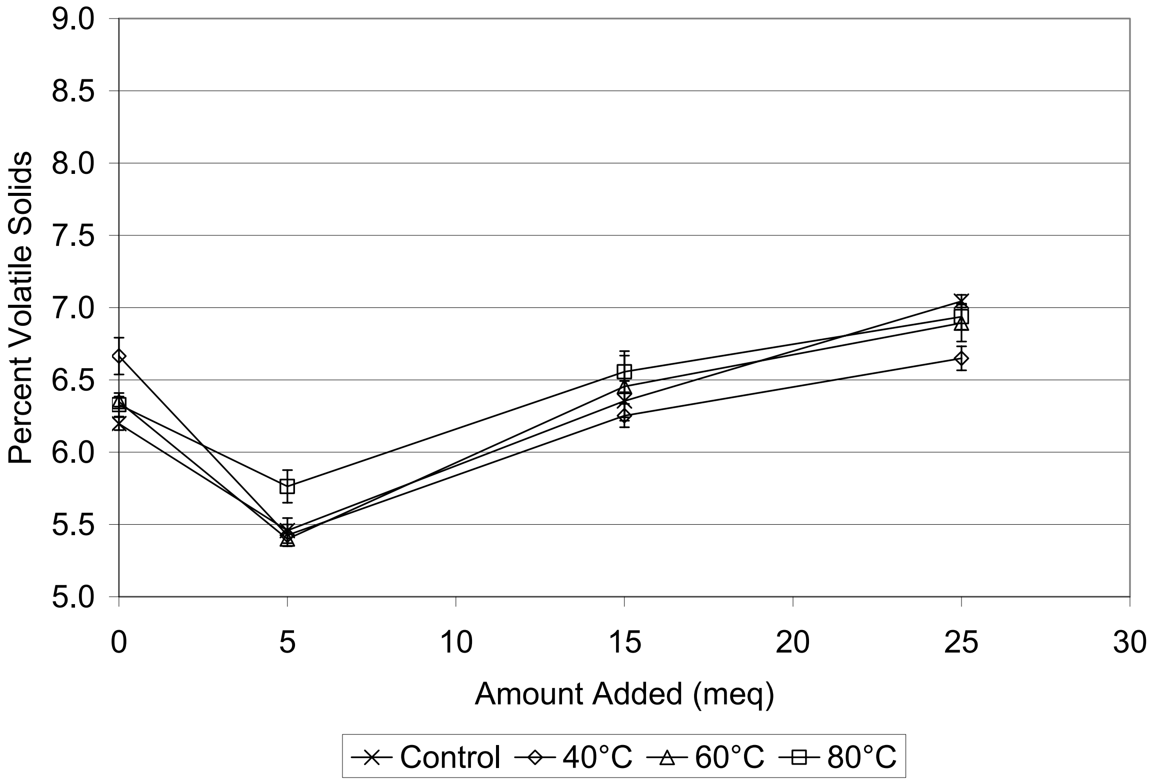

Figure 3.22, Compactibility measurements, heat treatment combined with iron addition.

Temperature had no effect on compactibility following heat treatment for any of the cations tested. Dewaterability tests were not performed for the combination of heat treatment and cation addition based on heat treatment not affecting the ability of cations to improve compactibility.

Bound water was measured following treatment with the combination of heat treatment and cation addition. 200 ml WAS samples were heat treated to 40, 60, and 80°C for 2 hours then had 5 meq of sodium, magnesium, and iron added. Dilatometric bound water measurements were then made the results of which are shown in Figure 3.24.

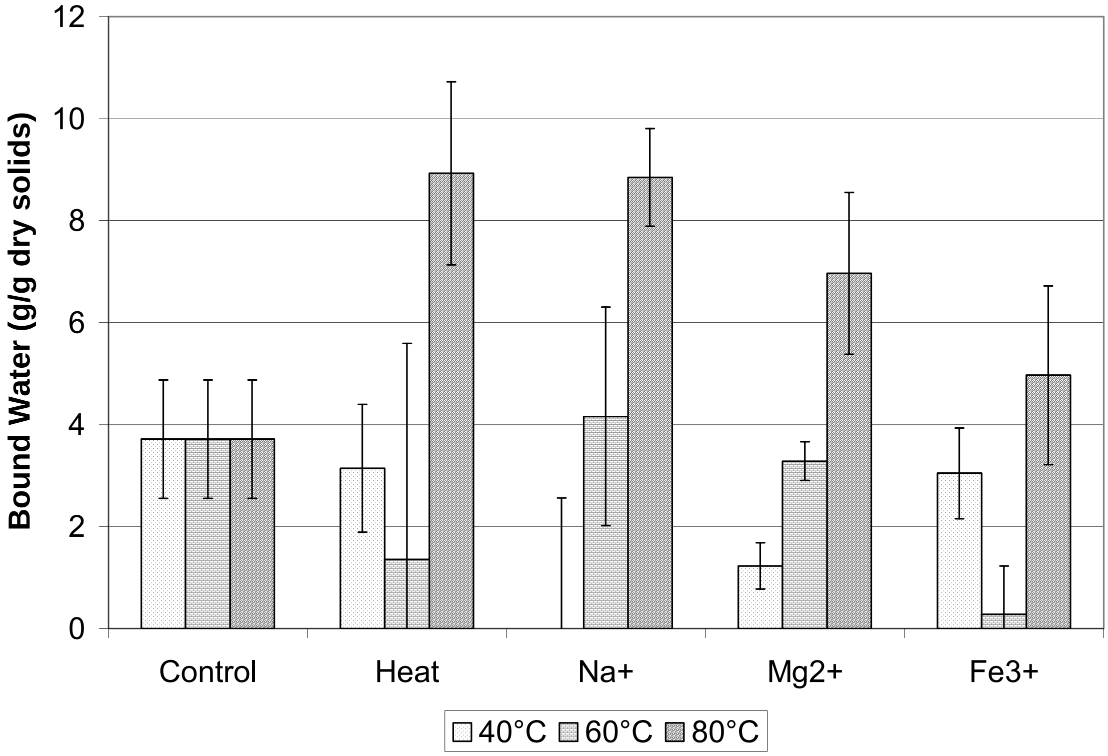

Figure 3.23. Bound water results, heat treatment combined with cation addition.

Addition of iron led to a decrease in bound water compared to the samples treated only at 80°C. At 40 and 60°C the addition of iron led to no significant change compared to the heat treated sludge. Addition of iron was only able to reduce bound water below unheated control samples in the case of 60°C. Addition of magnesium led to decreased bound water compared to both the heat treated sample and the unheated control at the 40°C level. Addition of sodium never led to significant change compared the heat treated sample and the unheated control at any temperature used in this study.

The ability of iron to reduce bound water at the 80°C level and the ability of magnesium to reduce bound water at the 40°C level can be attributed to the divalent cation bridging (DCB) theory of Tezuka (1969) and Higgins and Novak (1997A, 1997B). Iron and magnesium have the ability to tightly constrict floc structure thereby expelling more interstitial water. Monovalent sodium is not able to hold floc structure together with the same degree of contraction thereby allowing interstitial water to remain and be measured as bound water. As mentioned previously, it is believed that additional surface areas were created through heat treatment and this resulted in the increase in bound water content following heat treatment. Whereas increases in bound water were seen at all temperatures before, only increases resulting from treatment at 80°C were seen this time. A possible explanation is the use of different sludge samples for the study of heat treatment and the study of heat treatment combined with cation addition. Sludges were taken from the same plant, but were taken at different times.

3.3.6 Combination Study – Sonication and Cation Addition

The effect of combining sonication and heat treatment was studied. 200 ml samples of RAS were subjected to sonication at 50 watts and 160 watts for 30, 120, and 300 seconds. Following sonication 5, 15, and 25 meq of sodium, magnesium, and iron were added to samples. Dewaterability measurements were made for each cation sodium, magnesium, and iron. The results of which are shown in Figures 3.24, 3.25, and 3.26 respectively.

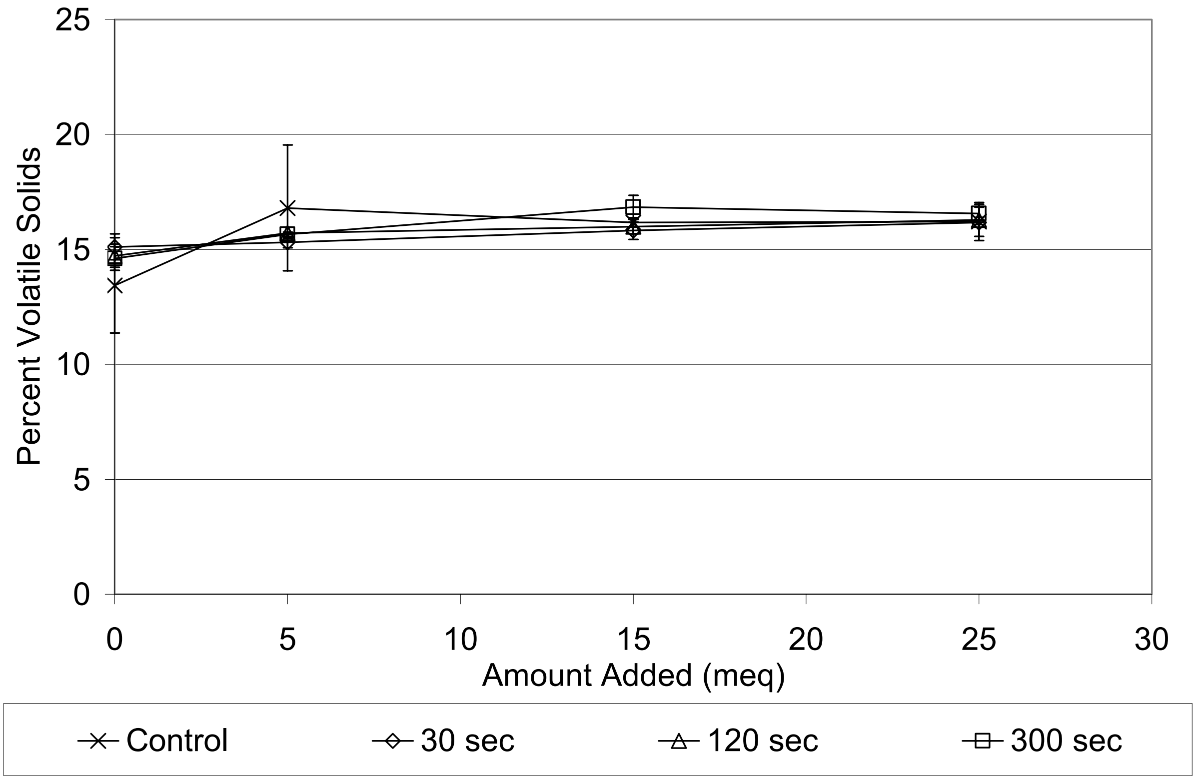

Figure 3.24. Dewaterability measurement, sonication combined with sodium addition.

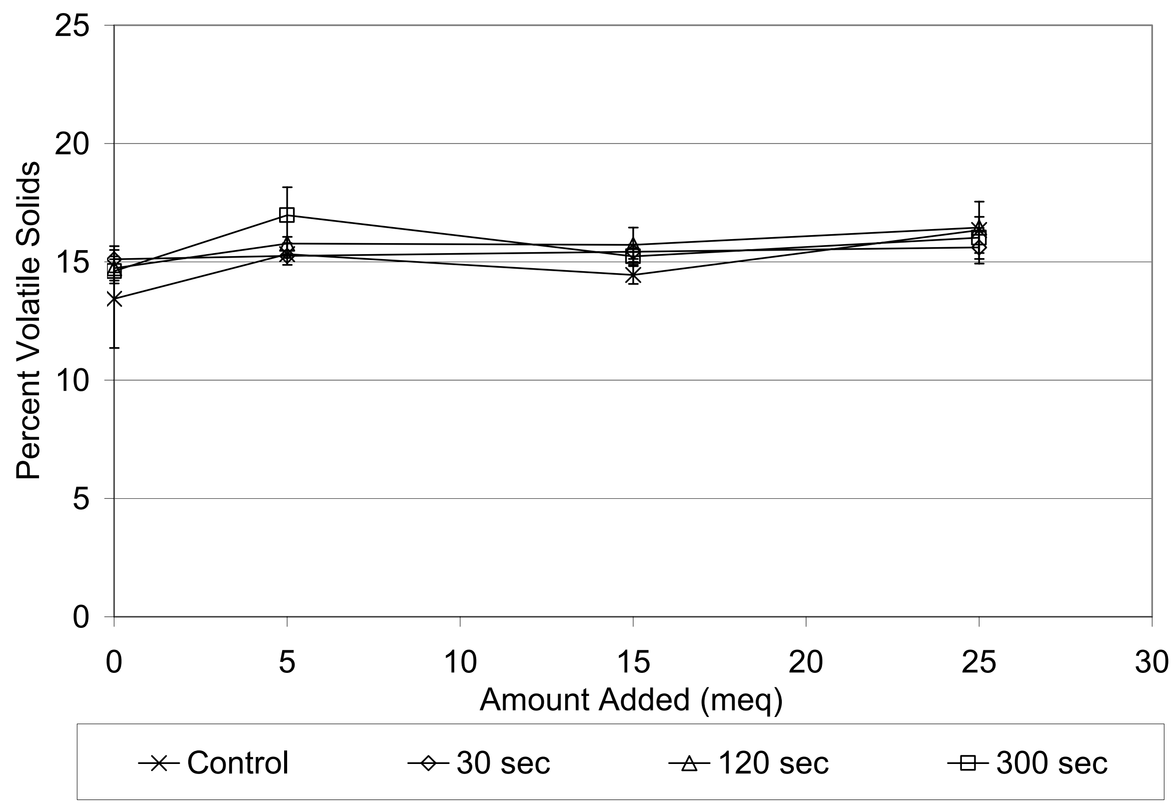

Figure 3.25. Dewaterability measurement, sonication combined with magnesium addition.

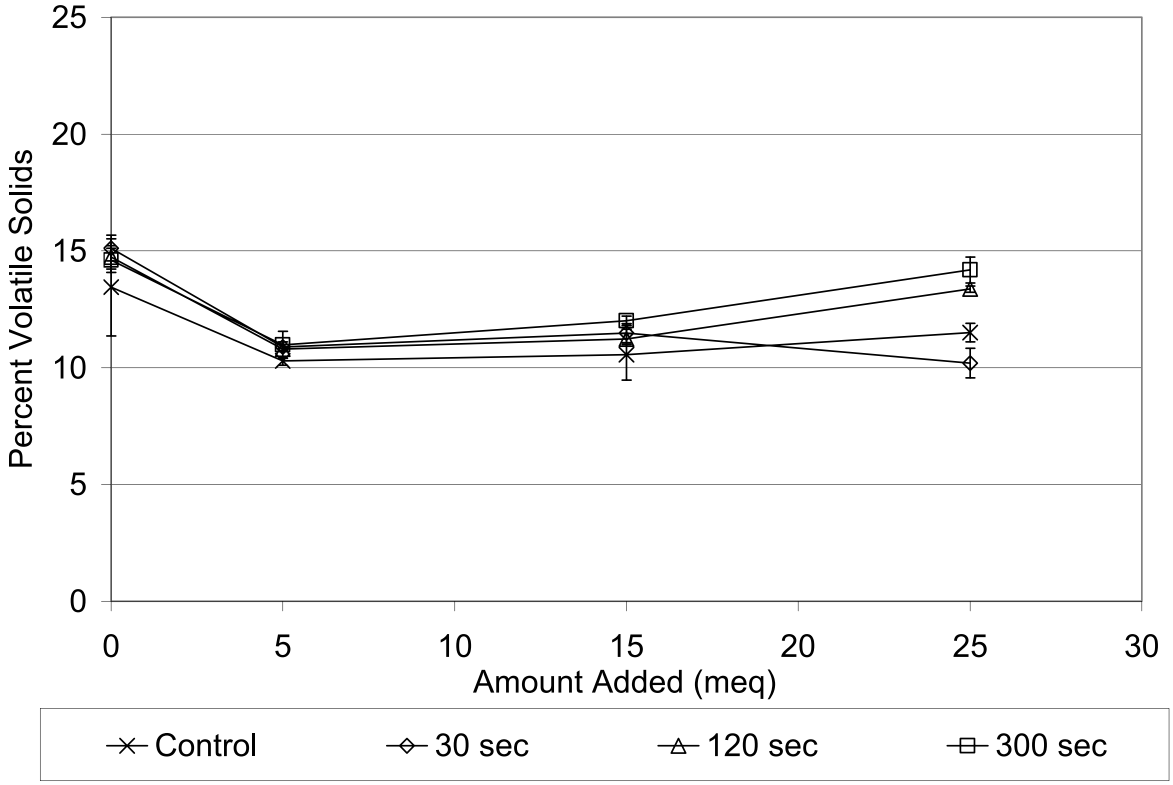

Figure 3.26. Dewaterability measurements, sonication combined with iron addition.

Sodium addition led to no significant changes in dewaterability measurements at any of the three amounts added. The addition of magnesium also led to no significant changes in dewaterability at any amount. As seen before, the addition of iron led to a sharp decrease in measured dewaterability. However, there may be an issue with the centrifuge tube material reacting with the ferric chloride to make the dewaterability test results invalid.

Bound water was measured in sludge disrupted by sonication that had cations added. 200 ml samples of gravity-thickened RAS from were sonicated at 50 and 160 watts for 30 seconds, which correspond to points of greatest increase in centrifugal dewatering tests. Additions of

5 meq of sodium, magnesium, and iron were made immediately following sonication. The results of this study are shown in Figure 3.27.

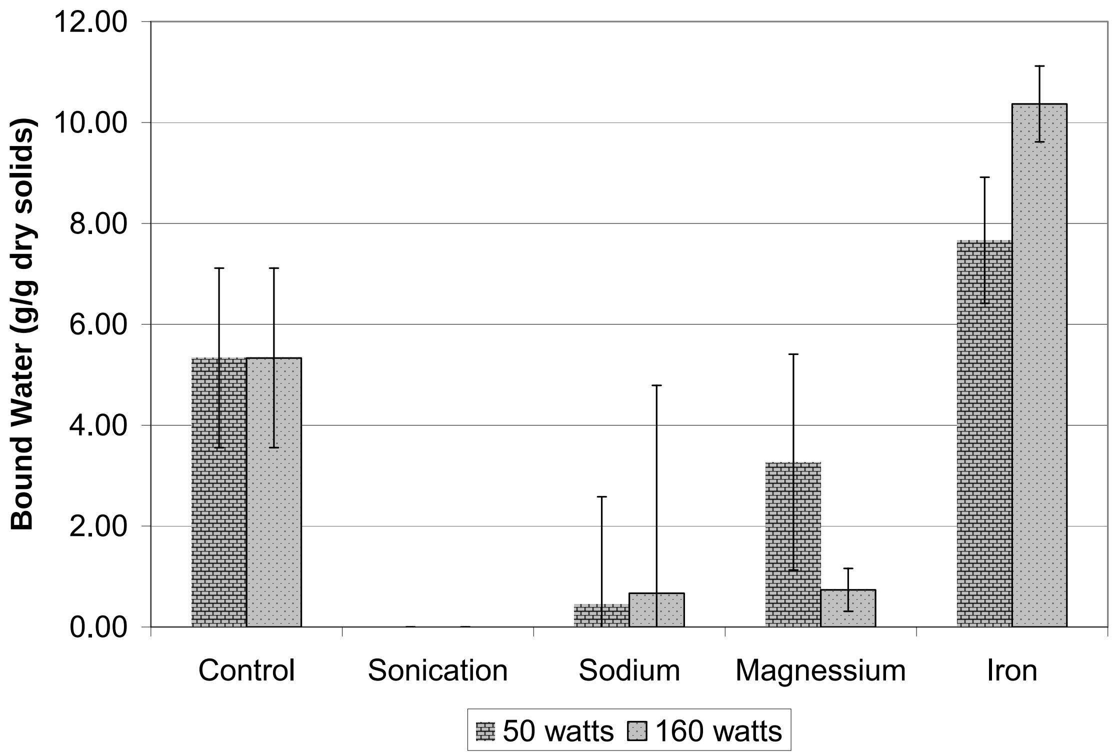

Figure 3.27. Bound water results, sonication combined with cation addition.

Sonication alone caused a complete reduction in bound water. This could be due to the deflocculation of activated sludge following sonication and the subsequent release of interstitial water. Vicinal water, which would remain despite the deflocculation, could also be removed as EPS substances, most notably protein, are displaced into solution by sonication and may carry vicinal water with them.

Again there is an association between cation valence and bound water content. Divalent (magnesium) and trivalent (iron) cations showed an increase in bound water content while monovalent (sodium) showed a slight increase. This bound water increase can be due to the

DCB theory. Divalent and trivalent cations can link floc structures and encourage reflocculation. This reflocculation in turn can lead to the accumulation of interstitial water that is shown by increased bound water.

3.4 CONCLUSIONS

Methods of disruption including sonication, heat treatment, alkaline treatment, and heat treatment were evaluated for their ability to increase sludge compressibility and dewaterability. In addition, soluble protein, cell lysis, time-to-filter, and bound water content were measured. The following conclusions were made

1. Achieving a high percentage of lysed cells in activated sludge systems proved to be difficult. In systems where cell disruption is measured through soluble microbial products such as protein, polysaccharide, and DNA, the products may not be intracellular in nature but rather of extracellular. A hypothesis is that further increasing sonication power or temperature may eventually lead to increased cell lysis. However, this increased energy input may make such a system undesirable based on costs to a municipality.

2. When cell lysis was observed, there was no correlation between increased cell lysis and dewaterability or compactibility for any disruption method tested. This makes the argument that intracellular water is vicinal in nature and is bound to cellular structures. This water was not removable even under high centrifugal forces up to ~200,000 x g following cell lysis resulting from heat treatment at 80°C.

3. Sonication showed increases in both compactibility and dewaterability. A decrease in bound water content indicated that interstitial water could have been converted to free water and therefore removed following sonication. Lack of significant cell lysis indicates that extracellular protein was removed from the floc structure. Smaller floc sizes were seen with sonication, indicating possible problems with filtration dewatering operations even though more water is theoretically removable.

4. Cation addition showed a similar pattern to the DCB theory. Addition of monovalent sodium led to increased bound water content and increased TTF indicating that deterioration of floc structures had occurred. Addition of divalent and trivalent cations led to decreased bound water and decreased or unchanged TTF. No significant changes were seen in dewaterability upon cation addition leading to the belief that changes are made only to floc structure and interstitial water and that vicinal water is not affected by cation addition.

5. Differences between bound water measured by dilatometry and centrifugal dewatering test indicate that dilatometry preferentially measures interstitial water as bound water. In heat treatment, increases bound water was not accompanied by decreased dewaterability. In sonication, decrease in dilatometric bound water were accompanied by disintegration of floc structures following sonication. This information warrants a further comparison of dilatometry and centrifugal dewatering for bound water determination.

6. The existing theory that bound water increases as a result of disruption because of the creation of additional surfaces as flocs break apart does not appear correct in this case. While heat treatment did lead to increased bound water content, there was no associated floc breakup as indicated by TTF measurements. In the case of sonication, floc size and structure was completely changed, which resulted in a decrease in bound water content measured by dilatometry. It was thought that heat treatment led to the creation of additional surfaces through coagulation of SMP not by the breakup of floc structure. Whereas sonication did not result in the creation of additional surfaces since no coagulation took place. Again, this information warrants further examination of the causes of bound water increases resulting from disruption of sludge structure.

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APPENDIX 1

Study to Evaluate the Effects of Storage on Centrifugal DewateringTest.

OBJECTIVE: To examine the effect of sample storage on centrifugal dewatering test results.

Samples of waste activated sludge were due to be collected from eight municipal wastewater treatment plants in North Carolina. In order to save shipping costs it was decided to have the plants collect samples three times a week (Mon, Wed, Fri or Tue, Thurs, Sat) and ship all three sets of samples in one package. This would mean that the plants would have to store samples on site for up to 5 days.

Samples of waste activated sludge were collected from the North Cary Water Reclamation Facility for testing. Approximately 1 gallon of sample was collected on October 14, 2004. A set of 12 subsamples was then prepared from the bulk sample. The samples were labeled as follows

-3 samples labeled “control”
-3 samples labeled “1”
-3 samples labeled “2”
-3 samples labeled “3”

The samples were subject to the following storage patterns.

“control” samples – analyzed immediately following collection

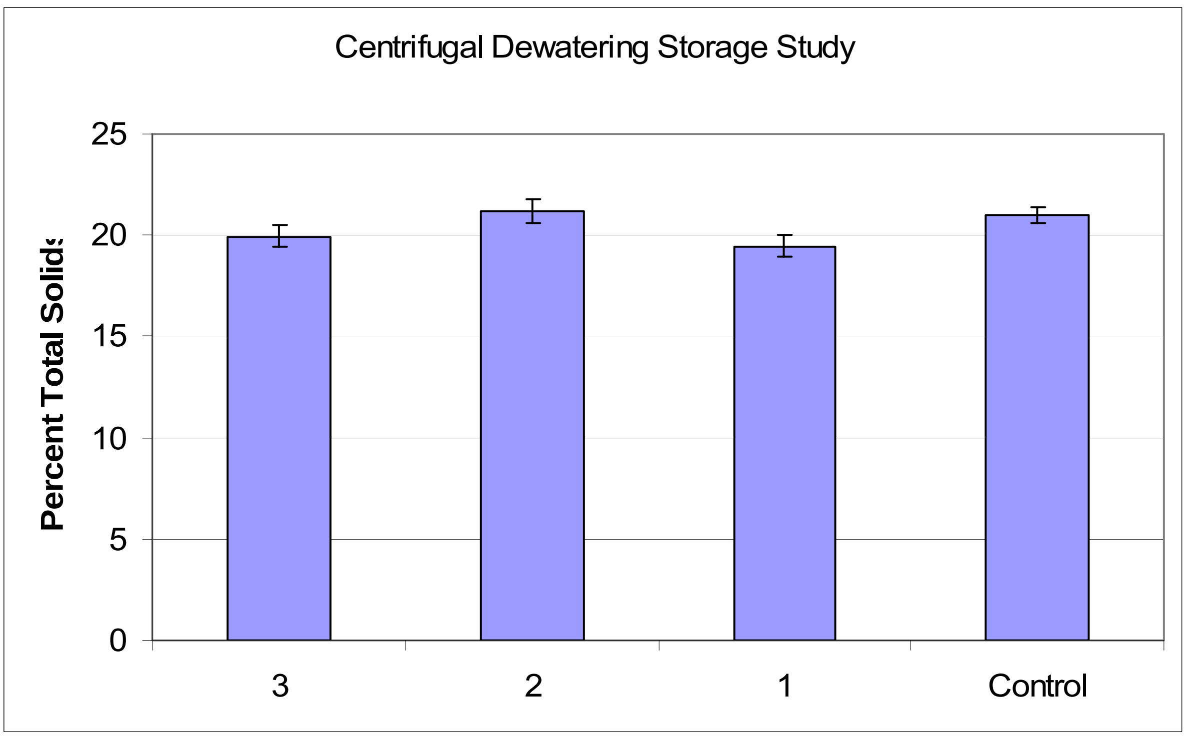
“1” samples – stored at room temperature for 3 days and analyzed. This represents a sample that a wastewater treatment plant would take on Friday and then immediately ship to NC State over the weekend.

“2” samples – stored at 4°C for 2 days then at room temperature for 3 days and analyzed. This represents the sample that a wastewater treatment plant would take on Wednesday, store in their refrigerator until Friday and then ship to NC State over the weekend.

“3” samples – stored at 4°C for 4 days then at room temperature for 3 days and analyzed. This represents the sample that a wastewater treatment plant would take on Monday, store in their refrigerator until Friday and then ship to NC State over the weekend.

Results of storage study are shown below.

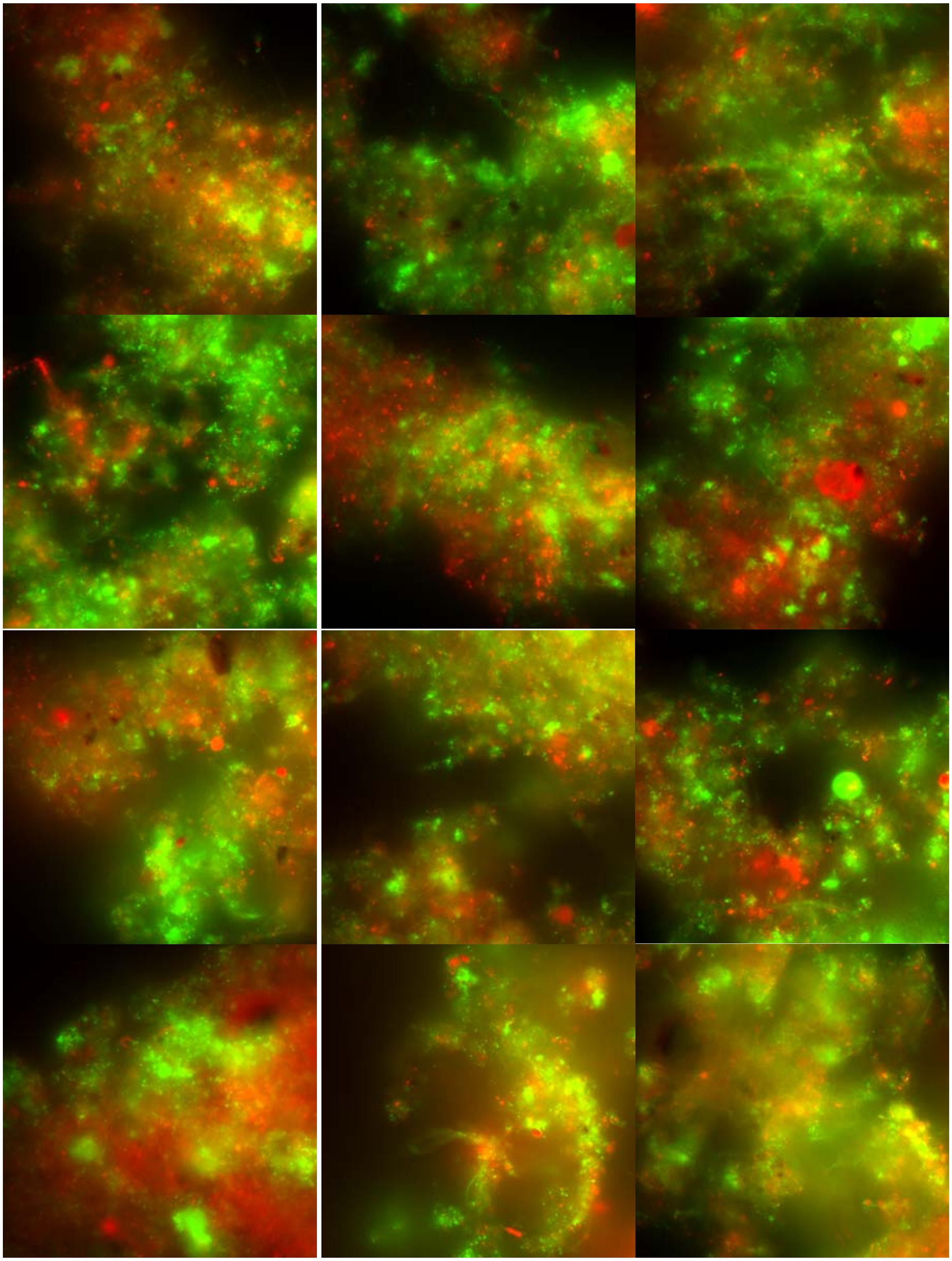
Sample	Refrigerated	Room Temperature	Analyzed	% Total Solids
control			10/14/2004	20.56
control			10/14/2004	21.06
control			10/14/2004	21.30
1		10/14/2004	10/17/2004	18.85
1		10/14/2004	10/17/2004	19.78
1		10/14/2004	10/17/2004	19.76
2	10/14/2004	10/16/2004	10/19/2004	21.42
2	10/14/2004	10/16/2004	10/19/2004	20.51
2	10/14/2004	10/16/2004	10/19/2004	21.62
3	10/14/2004	10/18/2004	10/21/2004	19.36
3	10/14/2004	10/18/2004	10/21/2004	19.99
3	10/14/2004	10/18/2004	10/21/2004	20.48



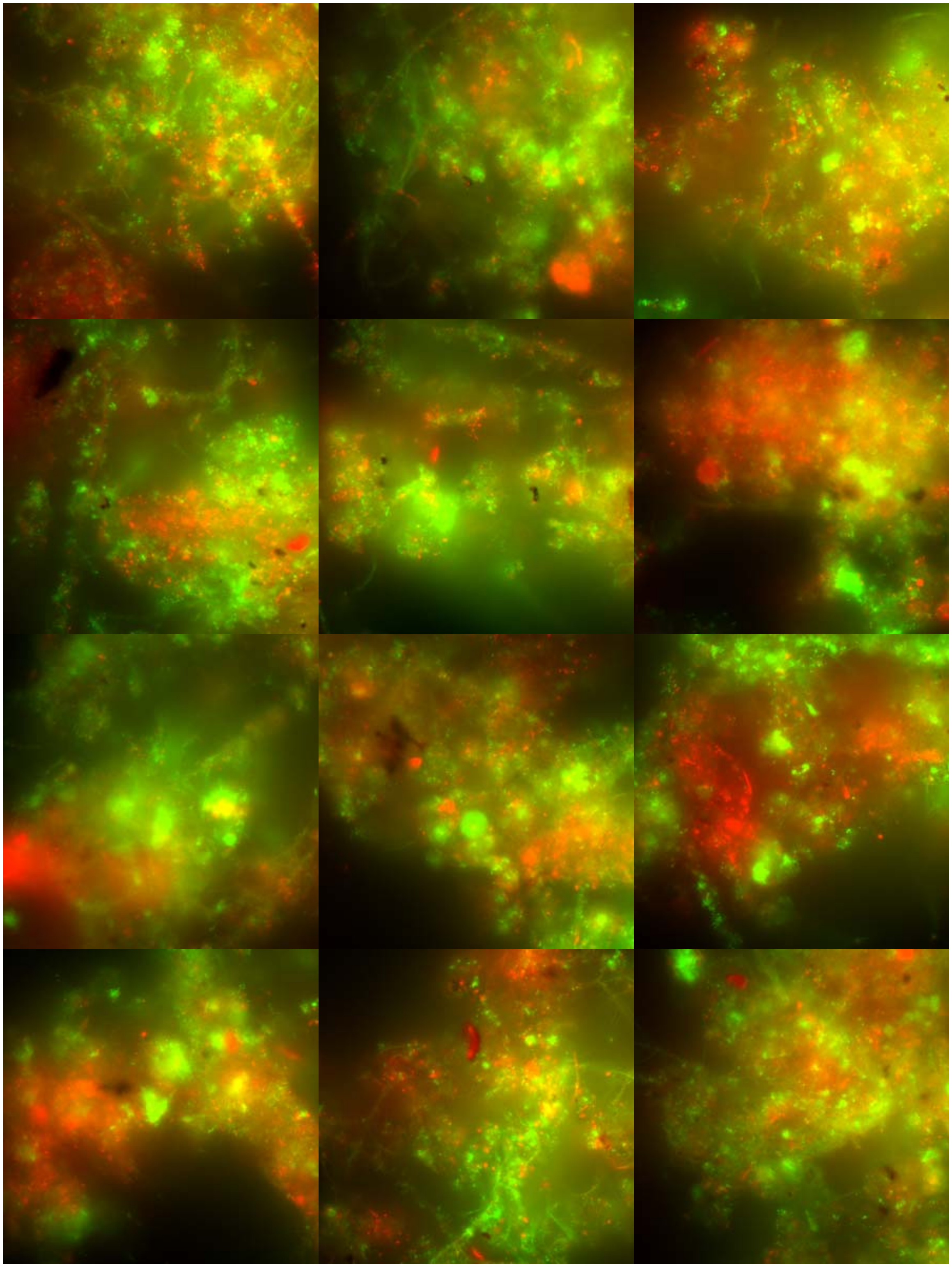
APPENDIX 2

LIVE/DEAD Microscopic Imaging: Proof Sheets for Treatments Used in Study.

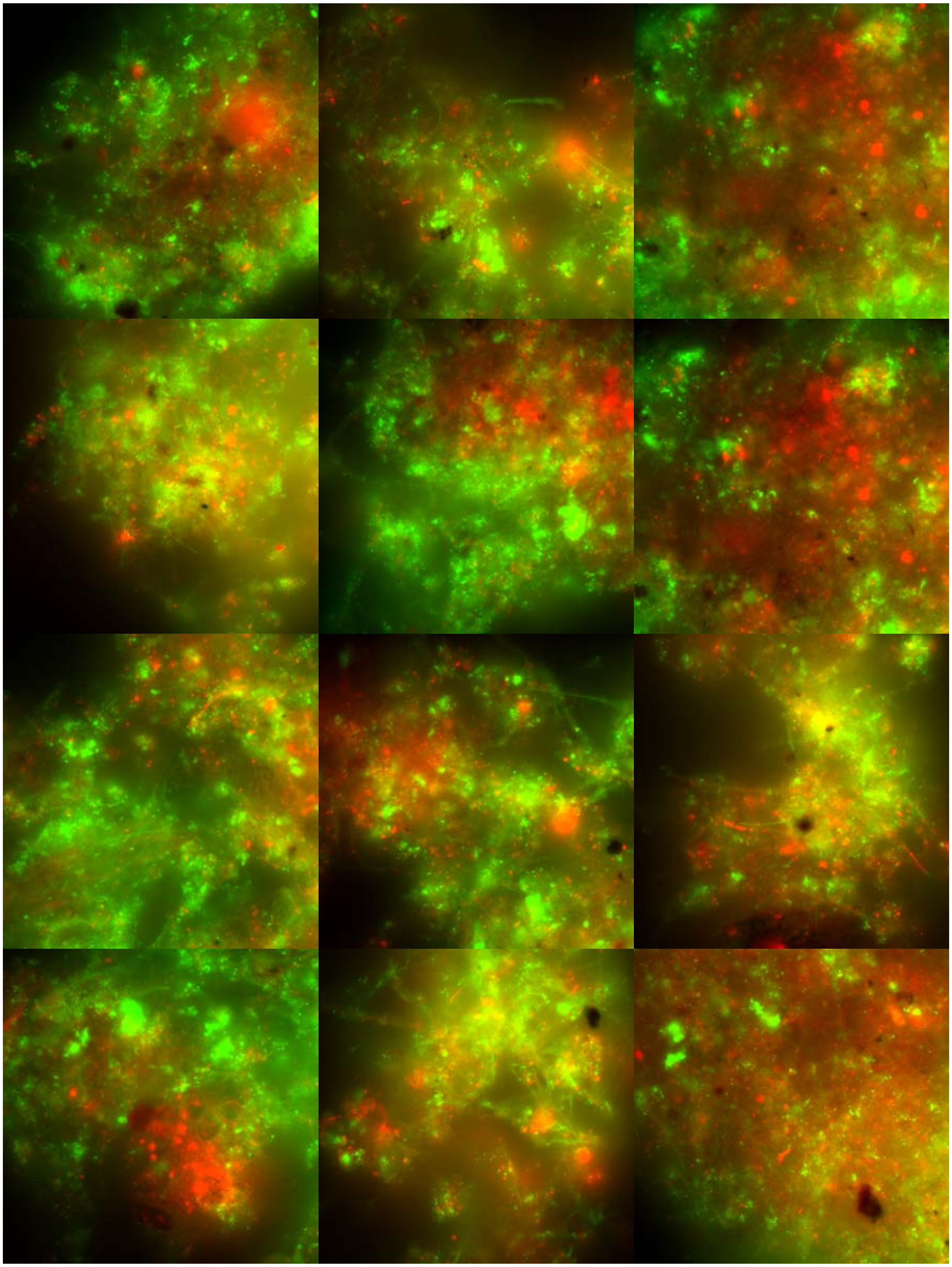
Control, Heat Treatment



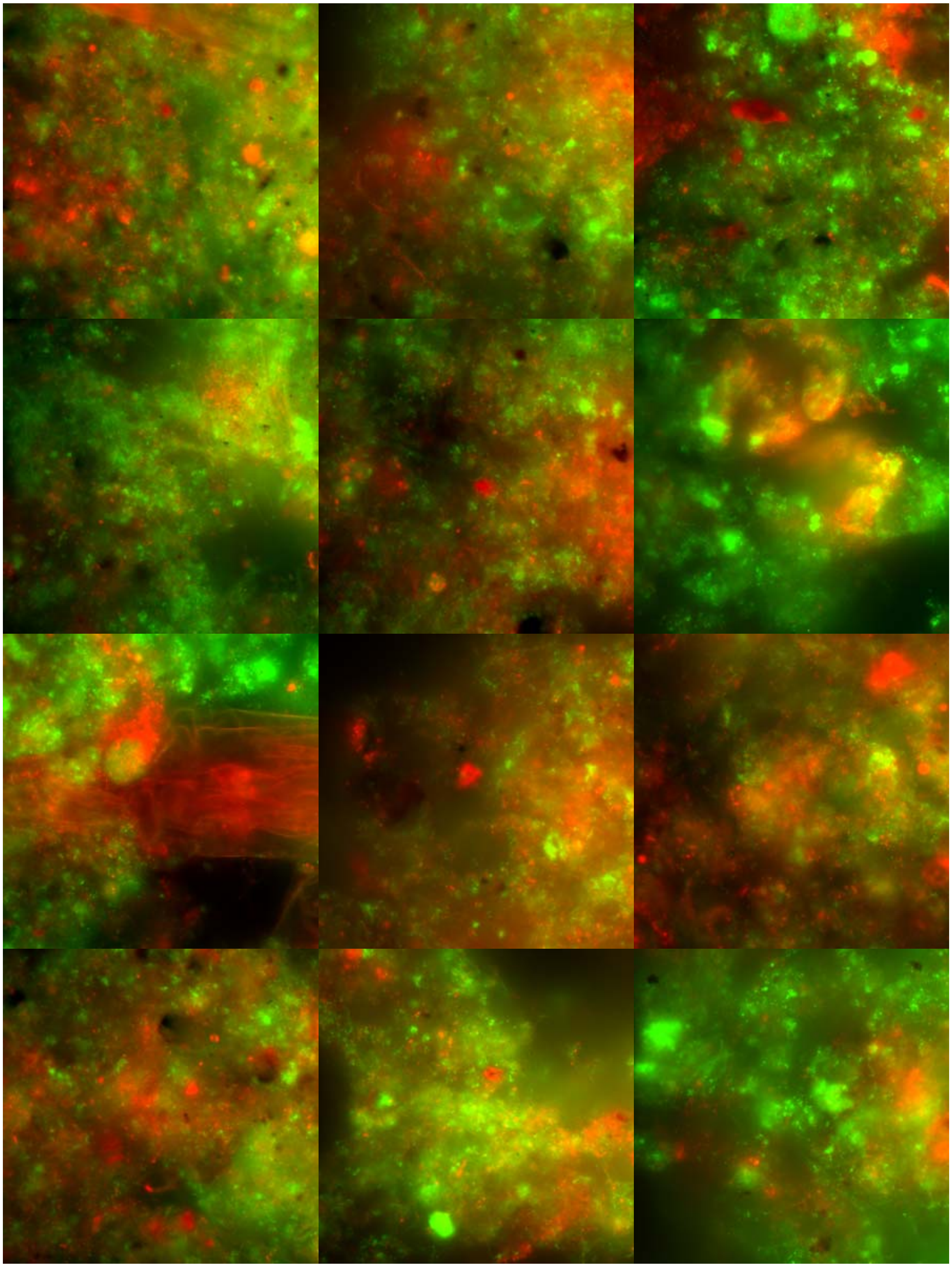
Heat Treatment, 40 Degrees Celsius, 5 Minutes



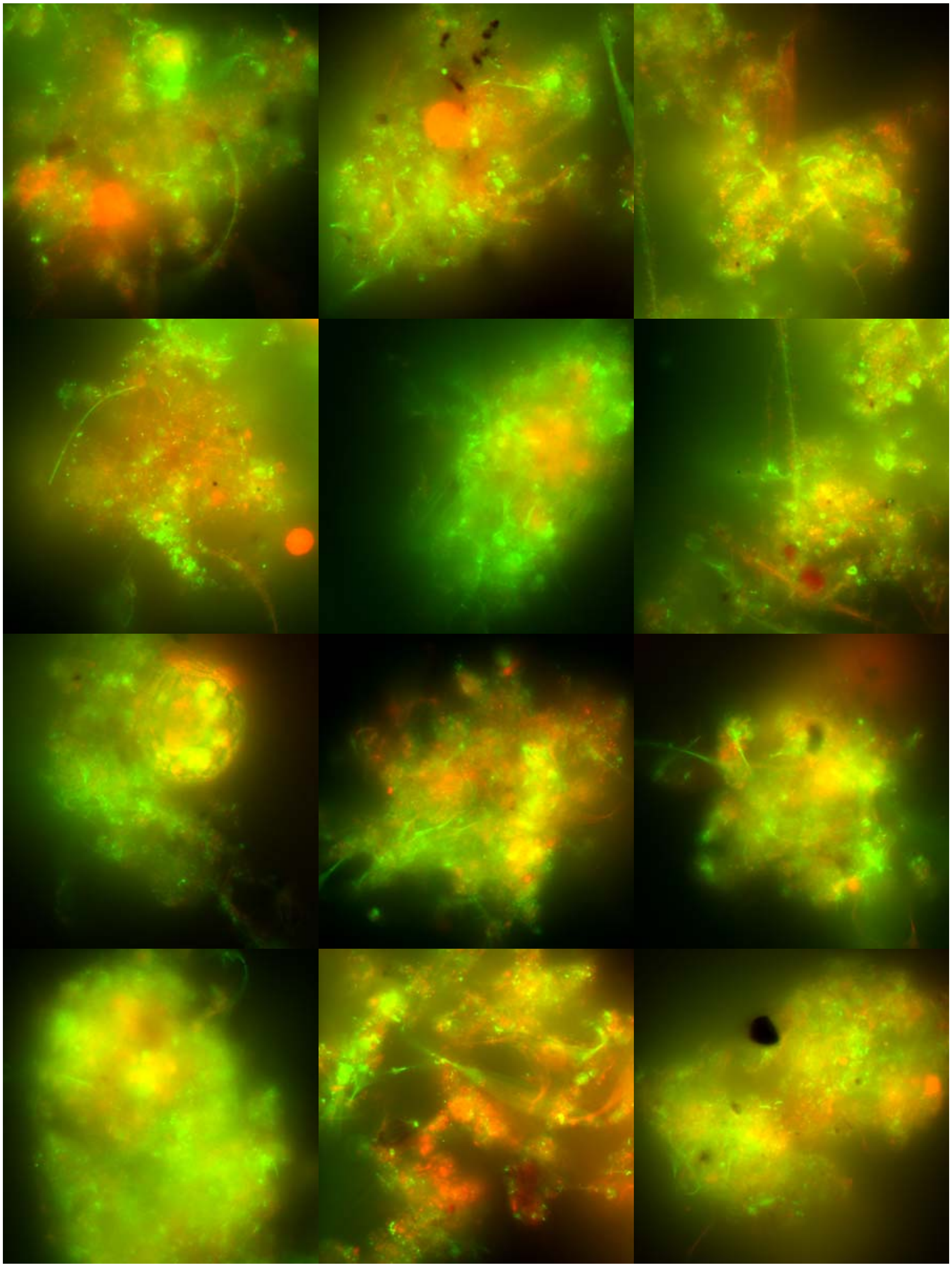
Heat Treatment, 40 Degrees Celsius, 1 Hour



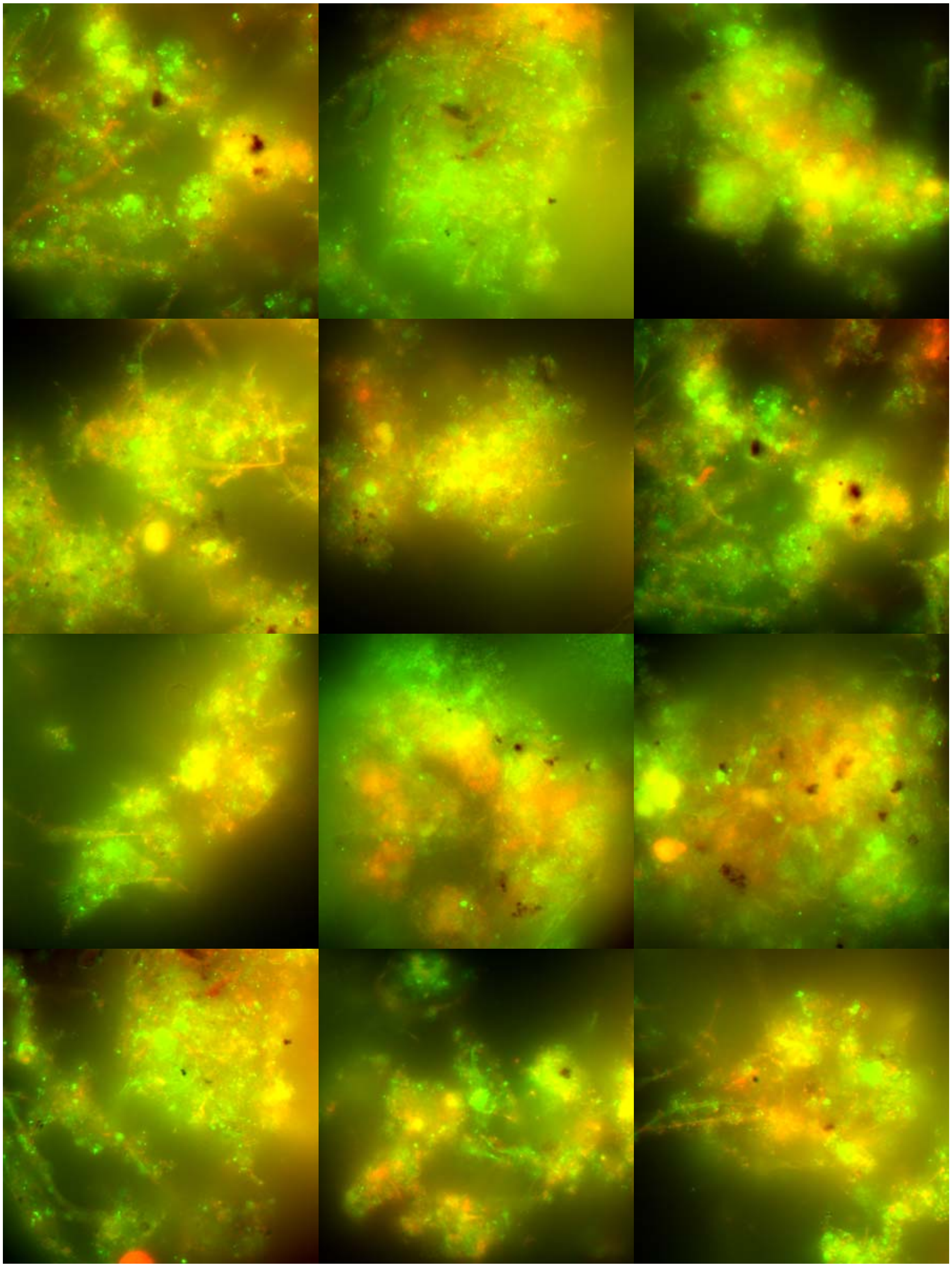
Heat Treatment, 40 Degrees Celsius, 2 Hours



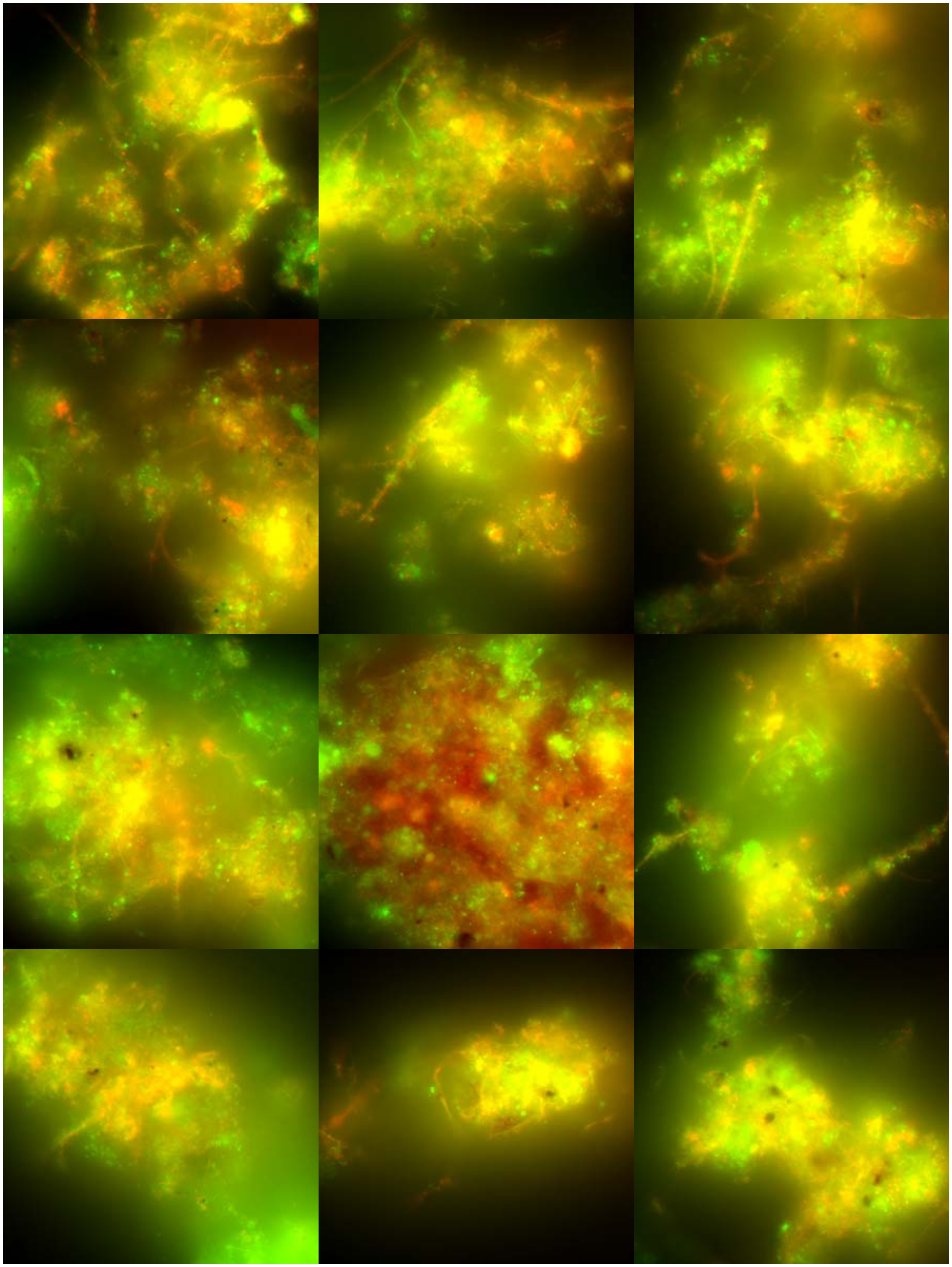
Heat Treatment, 60 Degrees Celsius, 5 minutes



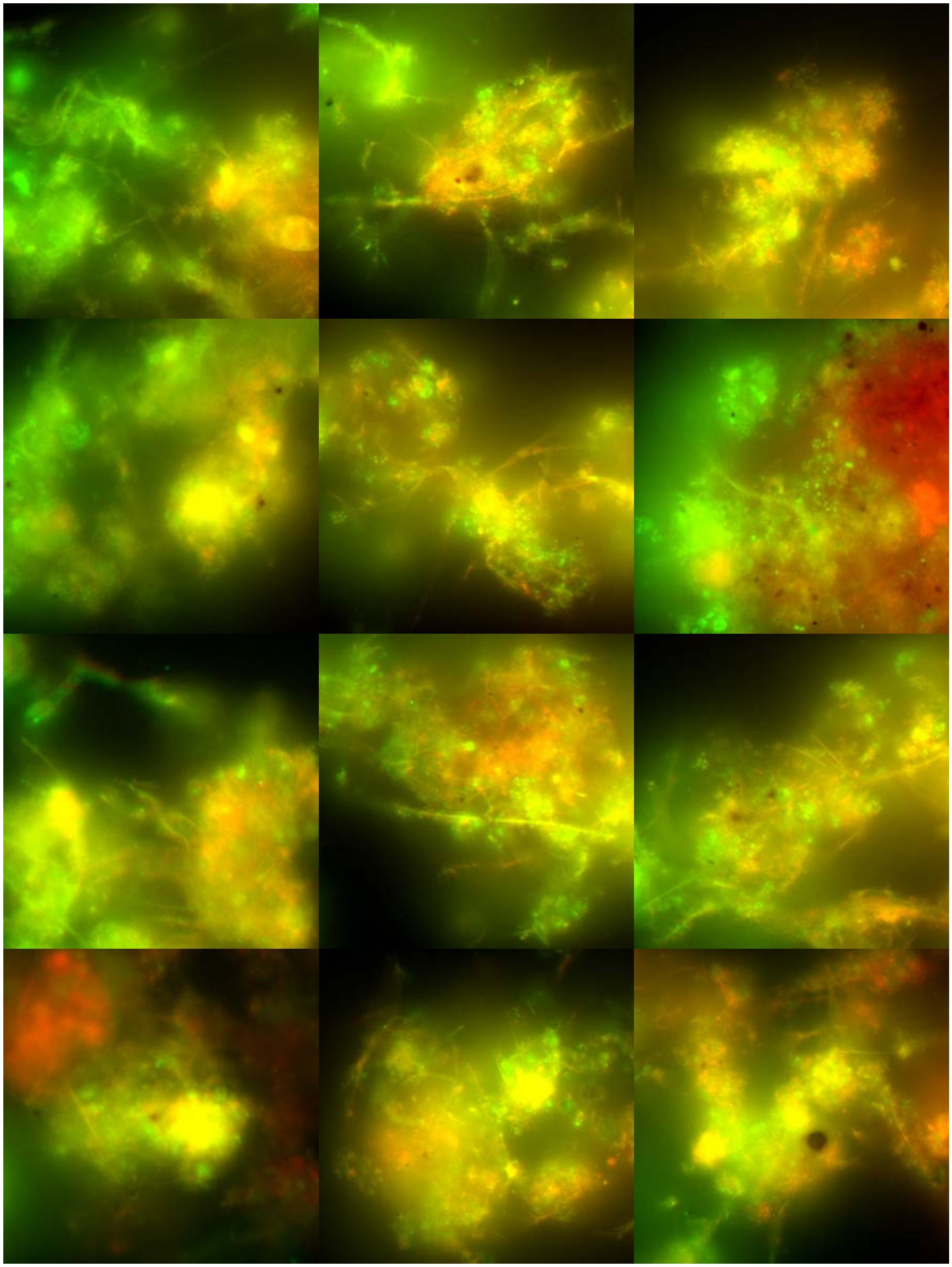
Heat Treatment, 60 Degrees Celsius, 1 Hour



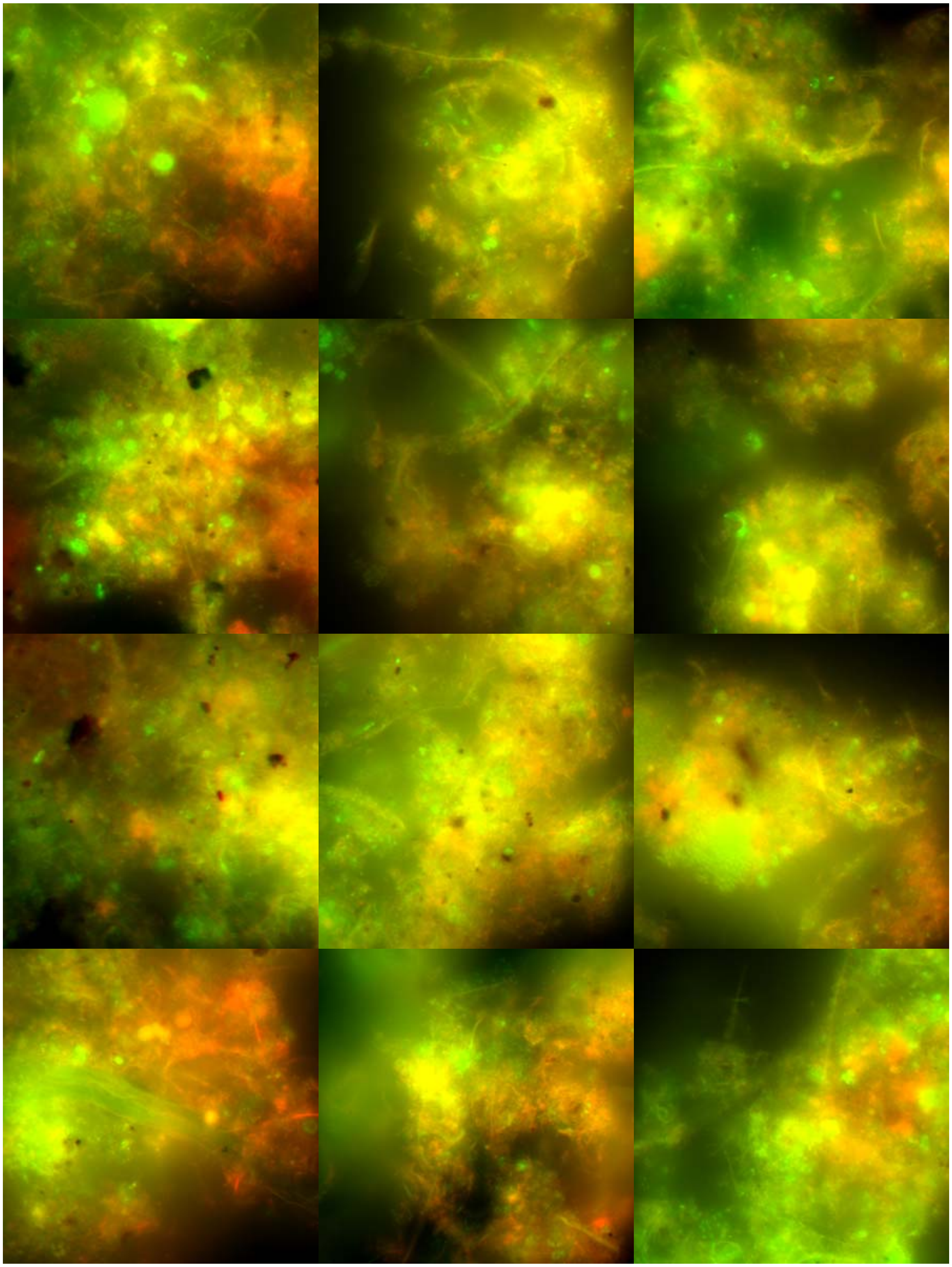
Heat Treatment, 60 Degrees Celsius, 2 Hours



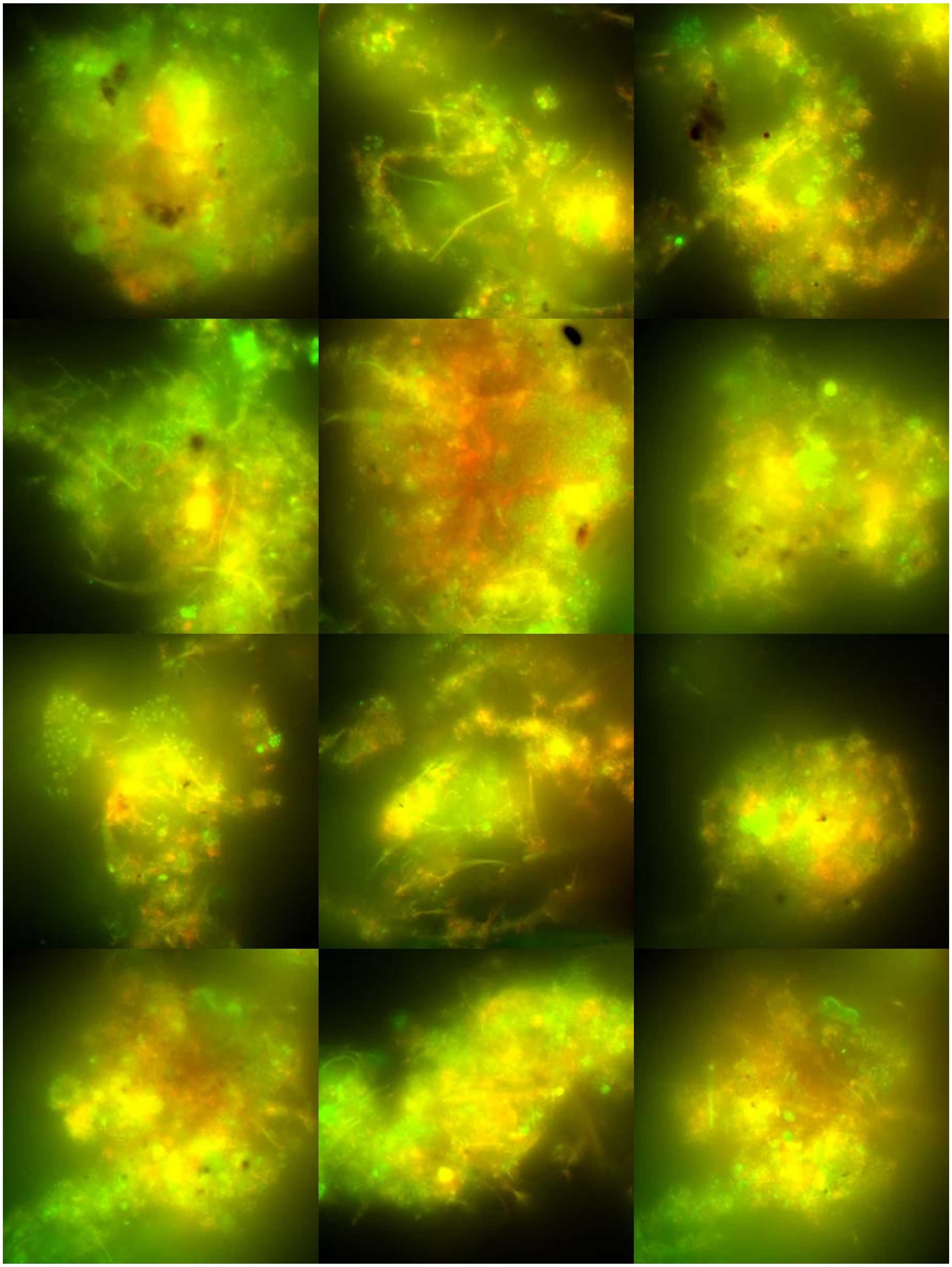
Heat Treatment, 80 Degrees Celsius, 5 Minutes



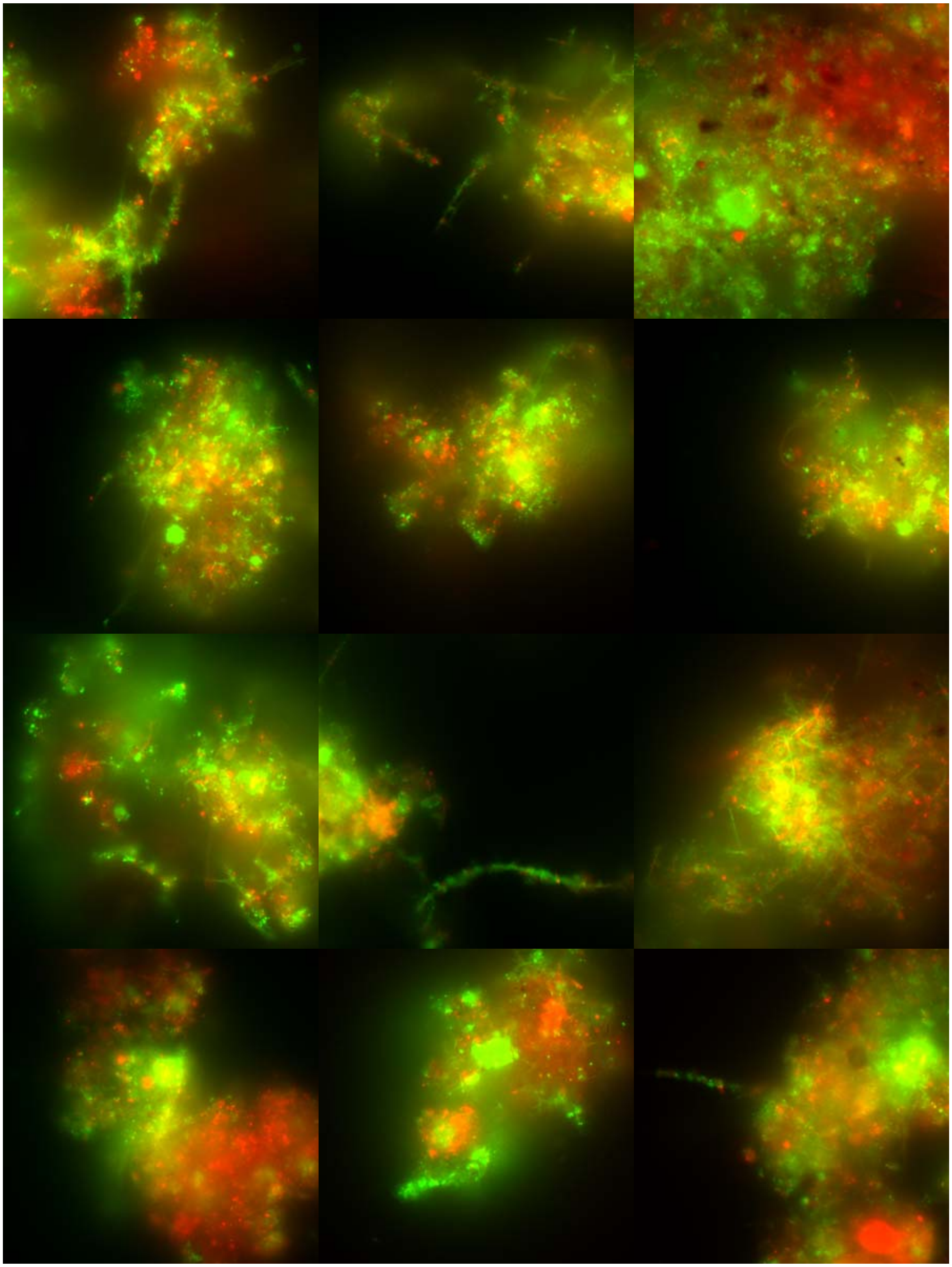
Heat Treatment, 80 Degrees Celsius, 1 Hour



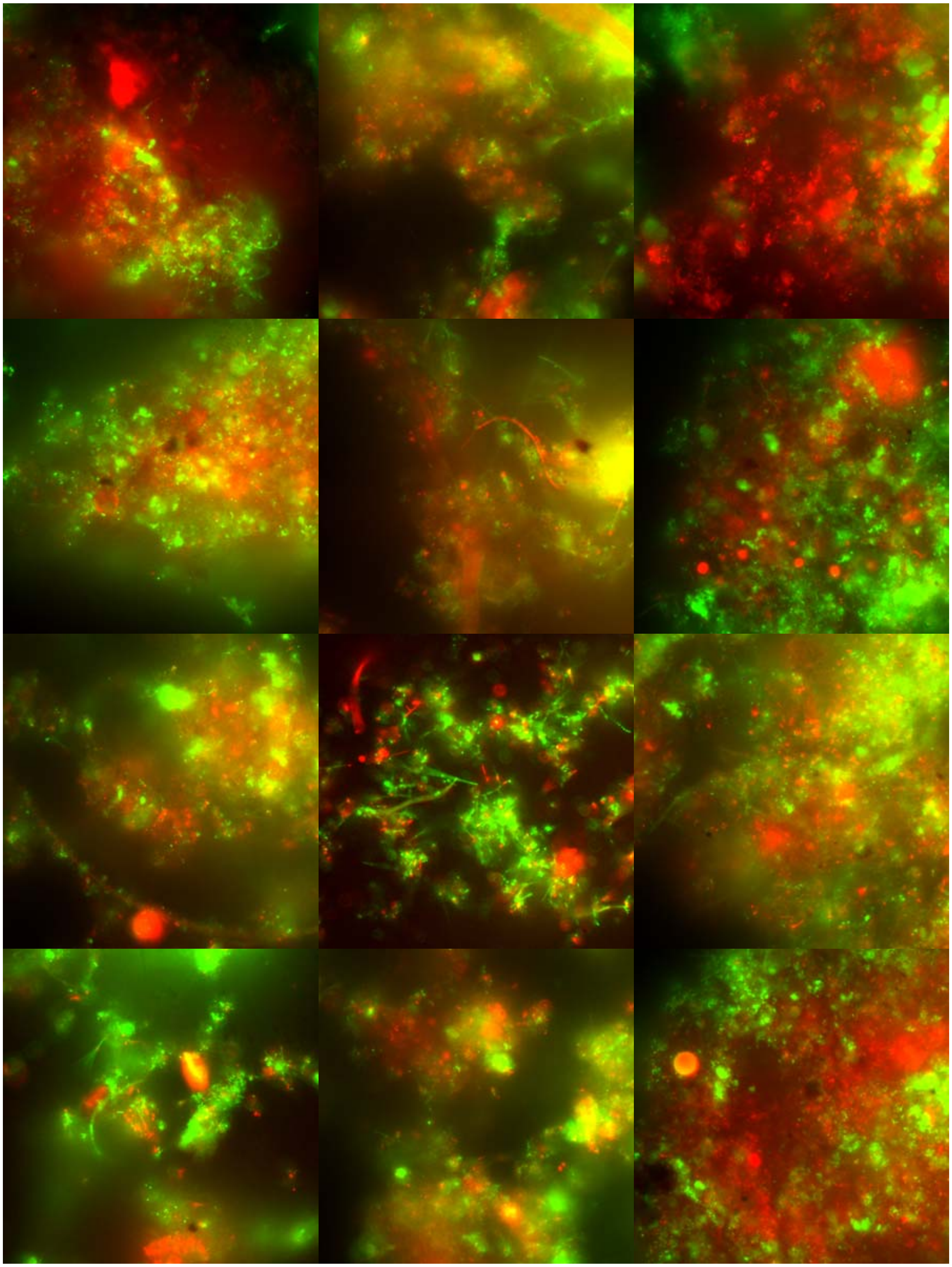
Heat Treatment, 80 Degrees Celsius, 2 Hours



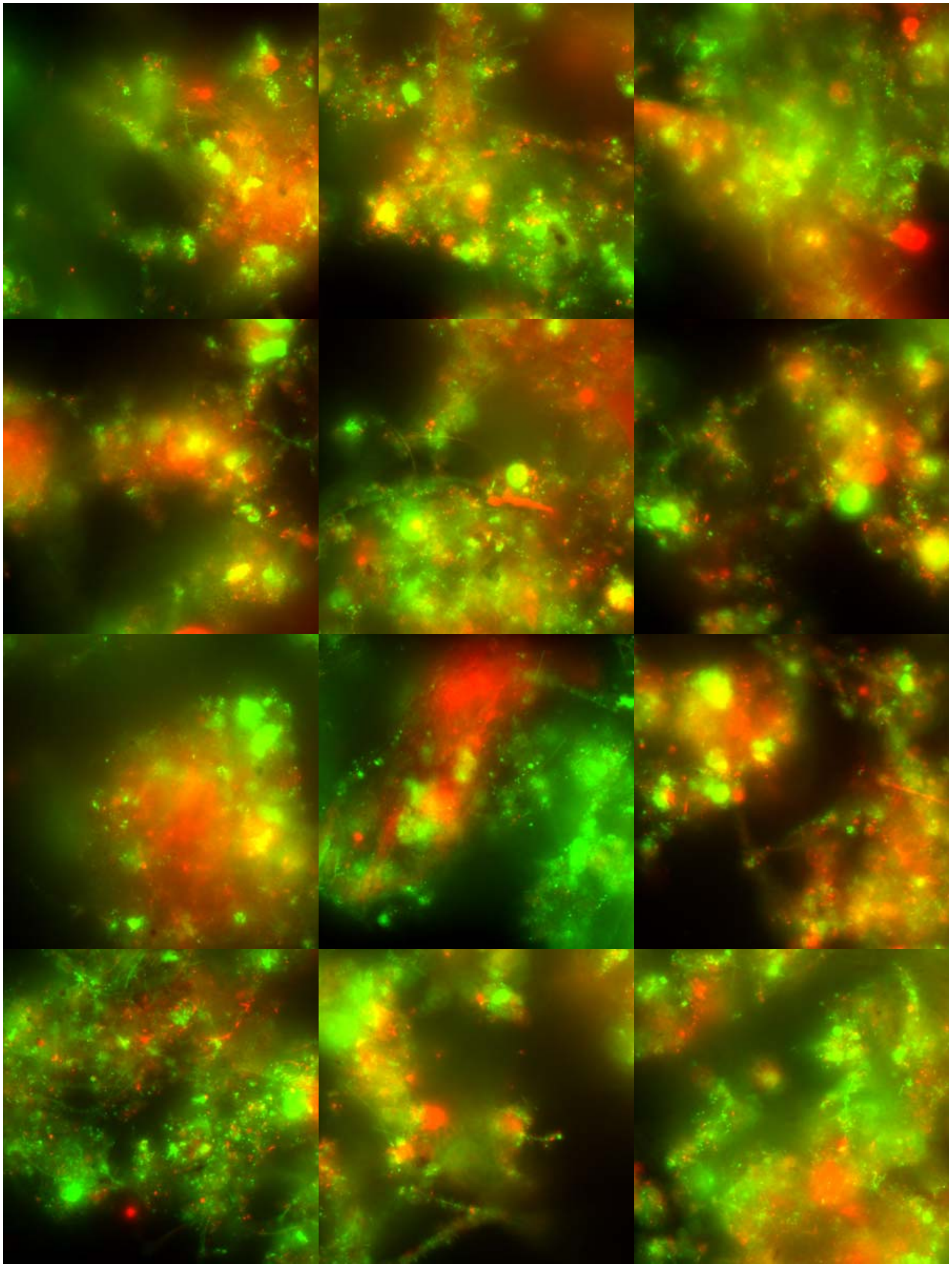
Control, Sonication



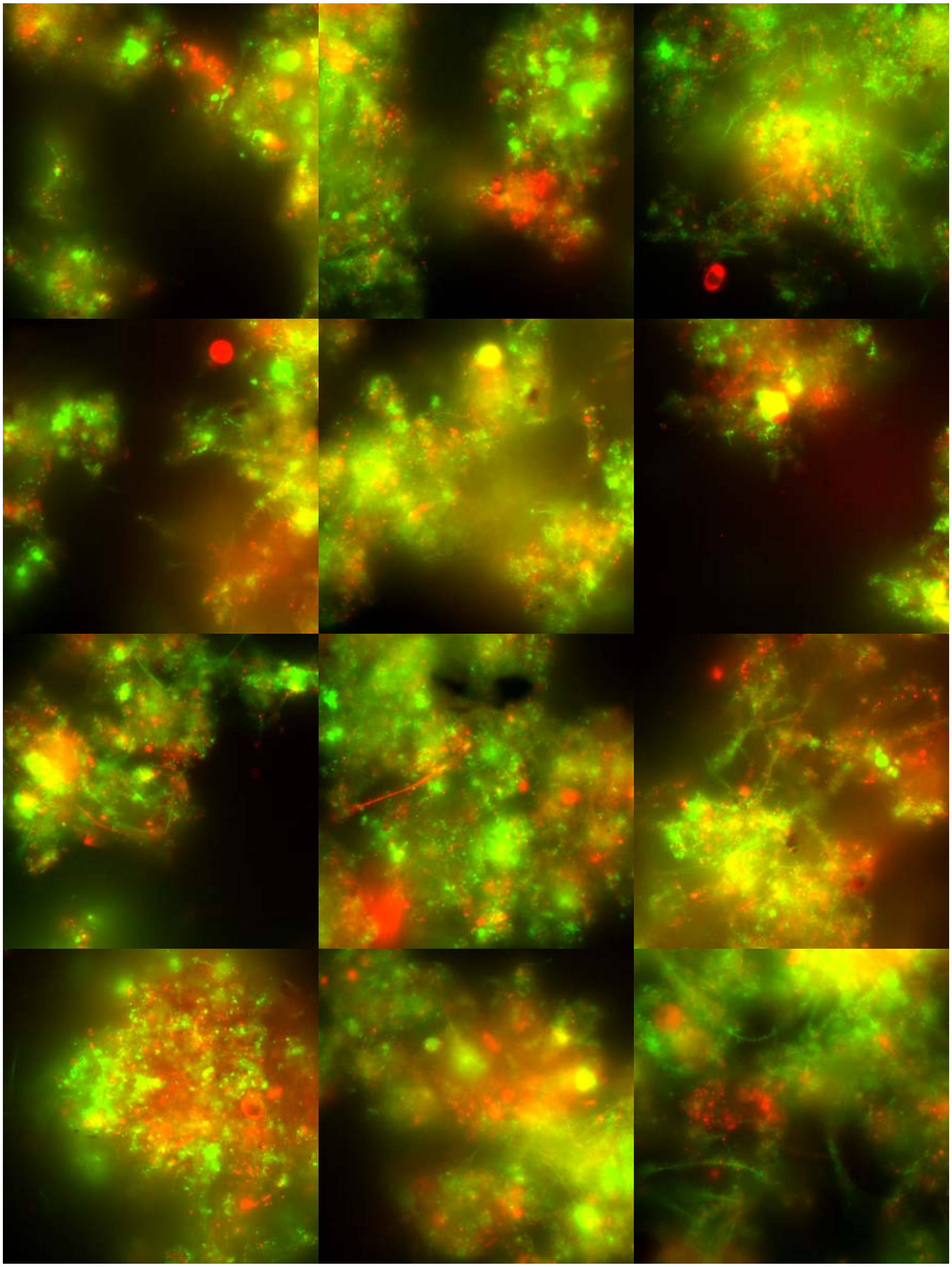
Sonication, 20 kHz, 50 Watts, 1 Minute



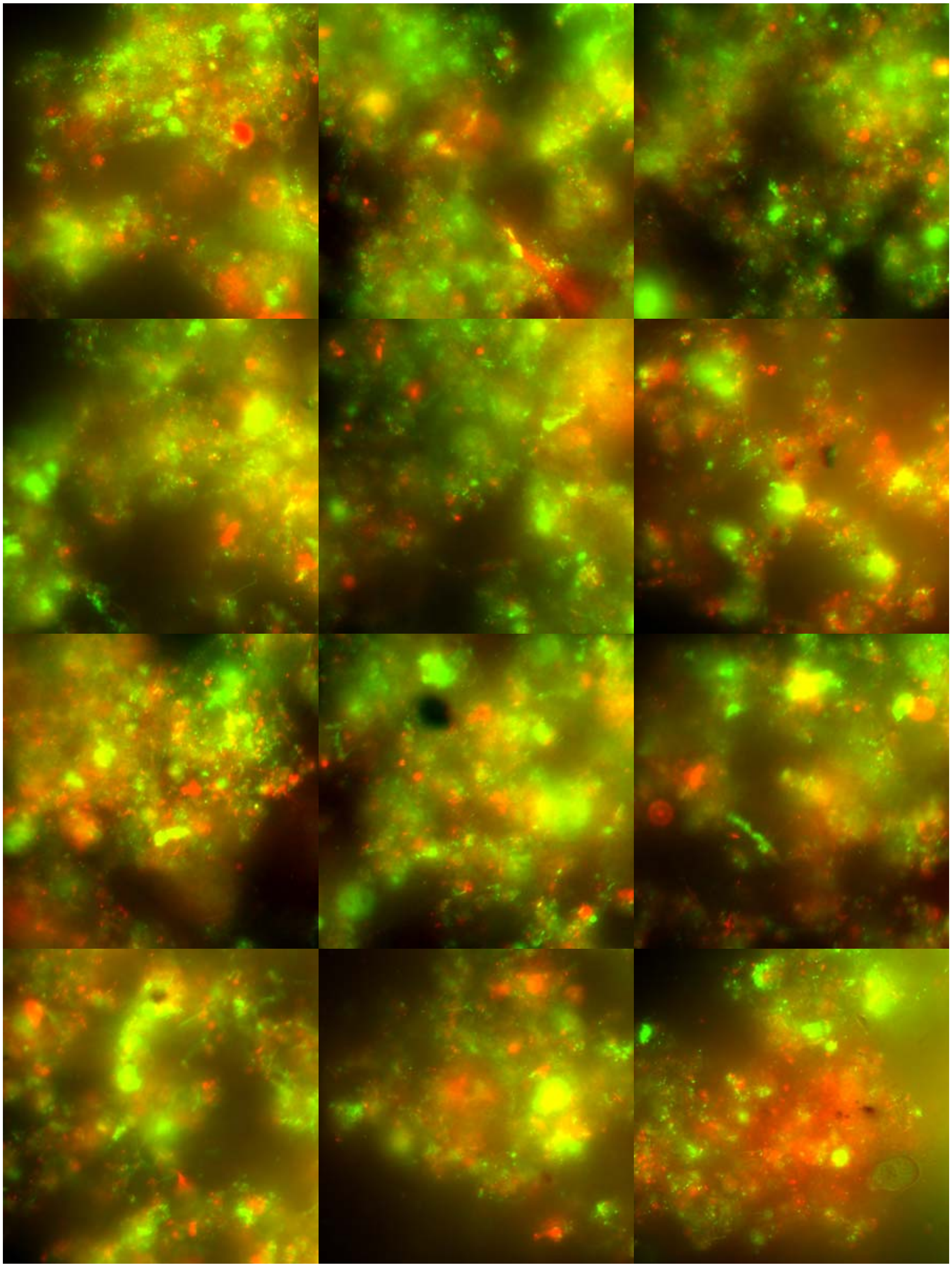
Sonication, 20 kHz, 50 Watt, 2 Minutes



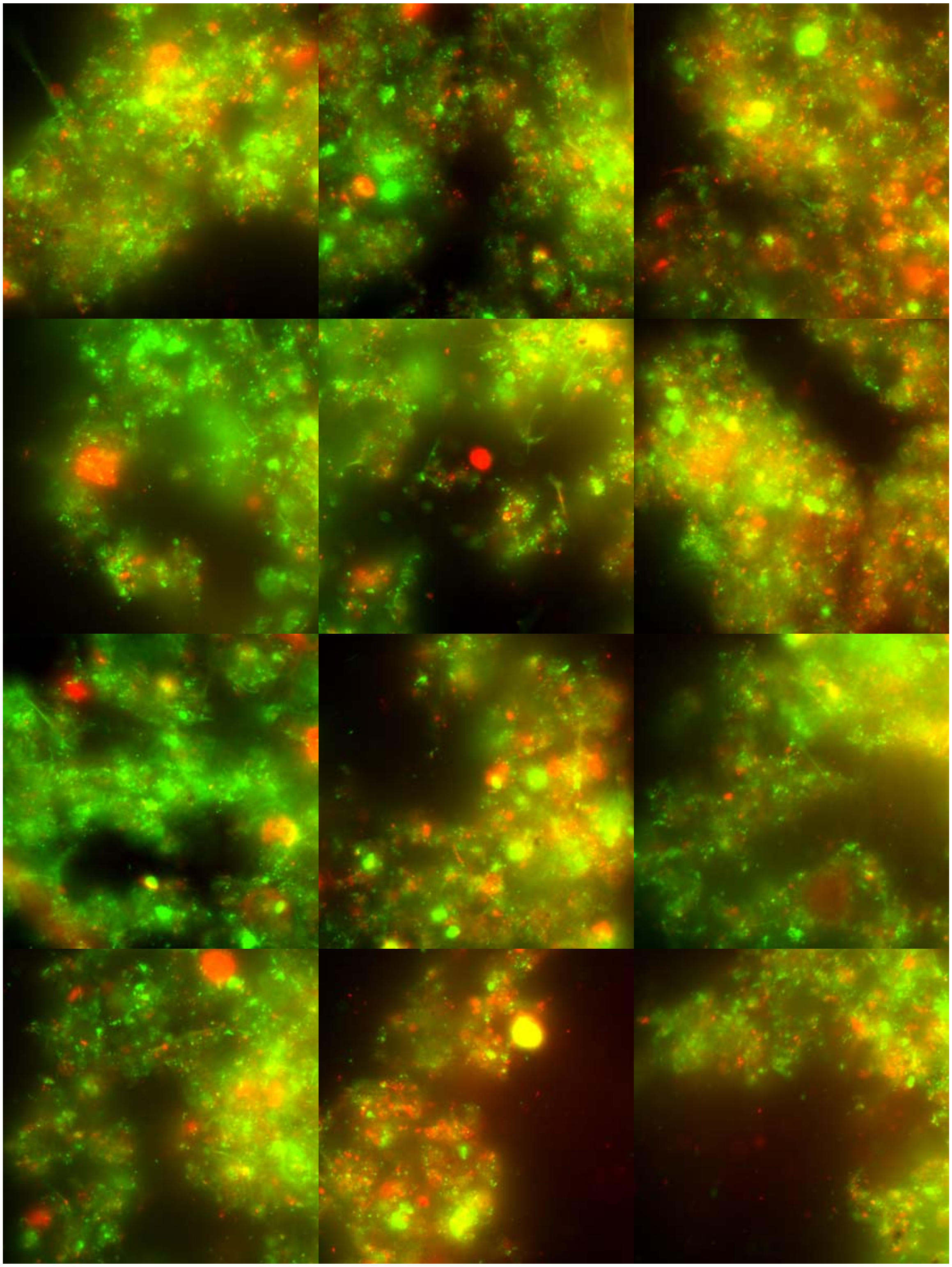
Sonication, 20 kHz, 50 Watts, 5 Minutes



Sonication, 20 kHz, 160 Watts, 1 Minute



Sonication, 20 kHz, 160 Watts, 2 Minutes



Sonication, 20 kHz, 160 Watts, 5 Minutes