Anaerobic / Aerobic Digestion for Enhanced Solids and Nitrogen Removal

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Abstract

Anaerobic digestion of wastewater sludge has widely been in application for stabilization of sludge. With the increase in hauling cost and many environmental and health concerns regarding land application of biosolids, digestion processes generating minimized sludge with better effluent characteristics is becoming important for many public and wastewater utilities.

This study was designed to investigate the performance of anaerobic-aerobic-anaerobic digestion of sludge and compare it to anaerobic-aerobic digestion and single stage mesophilic digestion of sludge. Experiments were carried out in three stages: Single-stage mesophilic anaerobic digestion (MAD) 20d SRT; Sequential Anaerobic/Aerobic digestion (Ana/Aer); and Anaerobic/Aerobic/Anaerobic digestion (An/Aer/An). The Anaerobic/Aerobic/Anaerobic digestion of sludge was studied with two options to determine the best option in terms of effluent characteristics. The two sludge withdrawal options were to withdraw effluent from the anaerobic digester (An/Aer/An – A) or withdraw effluent from the aerobic digester (An/Aer/An – B). Different operational parameters, such as COD removal, VS destruction, biogas production, Nitrogen removal, odor removal and dewatering properties of the resulting biosolids were studied and the results were compared among different processes.

From the study, it was found that An/Aer/An – B (wastage from aerobic reactor) provided better effluent characteristics than An/Aer/An – A (wastage from anaerobic reactor), Ana/Aer or conventional MAD. The study also shows that the Anaerobic/Aerobic/Anaerobic (An/Aer/An, with wastage from the aerobic or anaerobic digester) digestion of the sludge can improve the biosolids quality by improving the
dewatering capabilities, with lower optimum polymer dose, reduced CST and increased cake solid concentration, and reduce the odor generation from the biosolids.

Both An/Aer/Ana – A and An/Aer/An – B gave 70% VS removal, compared to 50% with single MAD and 62% with only Ana/Aer. COD removal of both An/Aer/An – A and An/Aer/An – B was 70%, while it was 50% and 66% for single MAD and Ana/Aer respectively. In the aerobic reactors of Ana/Aer and An/Aer/An - B, nitrification and denitrification with removal of nitrogen was observed. The An/Aer/An – B system had more ammonia and TKN removal (70%) than Ana/Aer (64%).

The effluent from each stage was analyzed for dewatering ability, cake solid concentration and odor production potential. Compared with a single Ana/Aer system, the extra anaerobic step in An/Aer/An – A and – B reduced polysaccharides in the effluent. The Ana/Aer system released less protein than the conventional MAD system and the addition of the second anaerobic step - especially with system An/Aer/An – B (discharge from aerobic reactor) - greatly reduced protein, resulting in improved dewaterability and less polymer demand. An/Aer/An (both of the options: A and B) had lower CST than single MAD (both 15d and 20d SRT) and Ana/Aer. Compared to Ana/Aer, a reduction of 52% for An/Aer/An – A and 20% for An/Aer/An – B in polymer dose requirement was observed, indicating improved dewatering characteristics. The An/Aer/An – B has higher biosolid cake concentration than MAD or Ana/Aer. The results showed that An/Aer/An (both options: A and B) biosolid had lower odor generation potential than single MAD (15d and 20d SRT) or Ana/Aer. Of all the stages, the An/Aer/An – A and – B system, generated odor which peaked at shorter time and lasted for shorter duration of time.
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**Attribution**

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Dr. Novak served as a coauthor in Chapters 2 and 3. He helped shape these chapters with his ideas and guidance and contributed to the research as the main investigator.
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Chapter 1

LITERATURE REVIEW

Introduction

Minimization of sludge generated from WWTP is very important because of cost, health and environmental factors associated with the transport and disposal of the biosolids. Under 40 CFR 503 Part (b) sludge reuse and disposal regulations (U.S EPA 1992), certain level of treatment of the sludge is required for pathogen deactivation before its land application. Applying biosolids in land has benefits; it is economical and it increases soil productivity and recycles resources. However, with use of biosolids in land, odor emissions from the biosolids have become a major environmental and health issue for general public and wastewater utilities.

Different treatment processes for achieving Class A biosolids have been investigated by many researchers. Anaerobic digestion of wastewater sludge has widely been in application for stabilization of sludge, because of its advantage in that it produces biogas as a byproduct. Mesophilic (35°C) anaerobic digestion is more commonly used than thermophilic digestion because of its higher stability. Thermophilic digestion, because of the process instability, is not listed as one of the processes to further reduce pathogens (US EPA, 1989). Rather, according to US EPA (1992) aerobic thermophilic digestion has been included as a process to reduce pathogens further and to reduce volatile solids efficiently. But it is also associated with high energy requirement for oxygen supply as well as for temperature maintenance. In anaerobic process, the biodegradation of organic compounds forms ammonia. So although anaerobic sludge digestion produces methane that can be used to generate energy, it also produces end products with liquid and solid residual organic matter which poses problem in disposal. Odegaard, (1988) reported nitrification-denitrification post-treatment of the anaerobic sludge as a widely used option to treat ammonia. In aerobic digestion of anaerobically digested sludge, the ammonia is oxidized to nitrite and nitrate which in turn are reduced completely through
denitrification, using volatile fatty acids as carbon source (Akuna, 1995). Novak et al., (2004), they proposed that organic compounds in sludge have many fractions. They can be degradable only under aerobic conditions, only under anaerobic conditions, under both aerobic and anaerobic conditions or cannot be degraded under any condition. So each anaerobic and aerobic digestion of sludge can degrade only some fractions of the sludge. The combination of both of these types of digestion can be complementary to each other in that it can be capable of degrading more fractions in the sludge using both anaerobic and aerobic environments. Novak et al., (2004) also found that the combination of both anaerobic and pre- or post- aerobic digestion of sludge reduced more volatile solids than a single anaerobic or aerobic digestion. Anaerobically digested sludge produces unacceptable odor (Murthy et al., 2002) and the compounds associated with the generation of odor are the volatile organic sulfur compounds (VOSCs) (Forbes et al., 2003; Higgins et al., 2002). Higgins et al. (2006) and Winter and Duckham (2000) have found that liquid anaerobic digested sludges have lower volatile odor compounds than dewatered, stored sludge. Novak et al. (2004), in their study of digested sludge characteristics, found that VS destruction decreases dewatering ability of sludge, increasing the polymer dose requirement.

This study was designed to investigate the performance of anaerobic-aerobic-anaerobic digestion of sludge and compare it to anaerobic-aerobic digestion and single stage mesophilic digestion of sludge. The anaerobic/aerobic/anaerobic digestion of sludge was studied with two options to determine the best option in terms of effluent characteristics. The two sludge withdrawal options were to withdraw effluent from the anaerobic digester or withdraw effluent from the aerobic digester. The hypothesis in this study was that the anaerobic-aerobic-anaerobic digestion of sludge would produce effluent with better VS destruction, nitrogen and COD removal, better dewatering properties and less odor compounds in the biosolids, compared with single or sequential anaerobic/aerobic digestion, and the research is described in detail in subsequent chapters. The results were expected to be useful in understanding the effectiveness of single anaerobic or combination of anaerobic and aerobic digestion mechanisms, based on sludge effluent characteristics.
Anaerobic Digestion

Anaerobic digestion is a process that microbially degrades organic matter without the use of oxygen. Biodegradable organic matters, both soluble and particulate, are converted to carbon-dioxide, methane and water. The anaerobic process also reduces and inactivates pathogens (Grady et al., 1999). Anaerobic process is a popular solid stabilization process, used in municipal wastewater treatment.

\[ \text{Organic matter} + \text{H}_2\text{O} \rightarrow \text{CH}_2 + \text{CO}_2 + \text{H}_2\text{O} \]

Depending on the operating conditions, anaerobic digestion reduces volatile solids by 35 percent to 60 percent (US EPA, 1992).

Microbiology and Biochemistry of Anaerobic Digestion

A wide range of microorganisms, primarily prokaryotic, mainly bacteria and methanogens are involved in anaerobic digestion. The characteristic of the microbial community depends on the substrate with which the digester is fed. The conversions of complex organic materials into simple matter are carried out by four types of microorganism: Hydrolytic bacteria, Fermentative acidogenic bacteria, Acetogenic bacteria and Methanogens (Archer and Kirsop, 1991). These consortia of microbial community operate in synergistic relationship as shown in (fig. 1.1).

a. Hydrolysis

Hydrolytic bacteria break down large organic molecules (e.g. polysachharides, proteins) into smaller, soluble molecules (e.g. sugars, amino acids). The hydrolysis reactions are catalyzed by extracellular enzymes (cellulases, proteases) produced by the bacteria. Hydrolysis rate depends on temperature, biodegradable organic matter, biomass nature, particles size and pH (Elefsiniotis et al., 1996).
b. Acidogenesis

Fermentative acidogenesis bacteria transform sugars, amino acids, fatty acids to organic acids, alcohols and ketones, CO₂ and H₂. In an anaerobic digester, the acidogenic bacterial population is the largest, covering 90% of the total (Zeikus, 1980).

c. Acetogenesis

Fatty acids and alcohols are converted to acetate, hydrogen, and carbondioxide by acetate and H₂-producing bacteria called acetogenic bacteria. These groups of bacteria require low H₂ partial pressure for fatty acid conversions. Substrate is converted to propionic acid, butyric acid and ethanol and so acetate formation is reduced under relatively high H₂ tensions. Methanogens help in achieving low H₂ tensions by continually removing H₂ to produce methane. Thus, the H₂-producing bacteria have a symbiotic relationship with the methanogens that use the H₂ (Grady et al., 1999). The methanogens are described in detail in the next section.

d. Methanogens

(Bitton, 2005) suggests that the generation times of methanogens range from 3 days at 35°C to 50 days at 10°C and they grow slowly in wastewater. Acetate, CO₂ and H₂ formed from the acidogenesis process are used by the methanogens to produce methane gas. Methanogens are split into: Aceticlastic methanogens which convert acetate into methane and CO₂, and, H₂-oxidizing which convert hydrogen and carbodioxide into methane. In an anaerobic digestion process, among these substrates the methanogens use, about two-thirds of the methane produced is derived from acetic acid while only one-third is from H₂ and CO₂ (Gujer and Zehnder, 1983).
Figure 1.1. Schematic diagram of metabolic steps in anaerobic digestion

Complex Biodegradable Organic Particulates (Polysaccharides, proteins, fats)

HYDROLYSIS

Proteins and Carbohydrates

Lipids

Amino acids, Simple sugars

Long chain Fatty acids

Volatile Fatty acids, Organic Acids, Alcohols, Ketones, Acetic acid, Hydrogen, CO₂

FERMENTATIVE ACIDOGENESIS

ACETOGENESIS

Acetate

CO₂, H₂

Aceticlastic Methanogens

H₂ Oxidising Methanogens

METHANOGENESIS

CH₄
When the wastewater contains significant concentrations of sulfate, sulfate-reducing bacteria competes with methanogens for the same electron donors, acetate and H2. Sulfate-reducers are more versatile than methanogens and in conditions where sufficient sulfate is present, single species of these microorganisms directly degrade compounds like propionate and butyrate (Stams et al., 2005). Methanogens are out-competed by the sulfate-reducers in low acetate concentrations (Oremland, R.S., 1988; Shonheit et al., 1982; Yoda et al., 1987). In wastewater containing both sulfate and acetate, feed acetate/\(\text{SO}_4^-\) ratio can determine the amounts of acetate used by methanogens and sulfate-reducers. In higher feed acetate/\(\text{SO}_4^-\) ratios, acetate used by sulfate-reducing bacteria decreases significantly (Bhattacharya et al., 1996). Choi and Rim (1991) found that at COD/\(\text{SO}_4^-\) ratios greater than 1.7-2.7, methanogens are favoured while sulfate reducers are favoured at decreased ratios. Anaerobic digestion process has been considered as one of the processes of reducing pathogens (Dahab et al., 1996; Eliot, 2003). Ponugoti et al., (1997) and Berg, (1980) showed that indicator bacteria were reduced by 1 to 3 log due to anaerobic digestion.

In a single-stage anaerobic digestion, all the processes, hydrolysis, fermentation, acidification and methanogenesis, take place together. The production of volatile fatty acids and utilization rates are balanced in a properly working anaerobic digester. Ghosh, (1991) proposed that volatile fatty acids are produced more than its utilization at short retention times. The acetogens and methanogens in an anaerobic digester have different growth rate and are favoured by different environment. In a two-stage anaerobic digestion (2PAD), acid forming and methane forming phases are separated. In a 2PAD system, the first phase is the hydrolysis-acidogenesis with a suppressed pH and the second phase is the methanogenesis. Another two-stage anaerobic digestion is Temperature-phased anaerobic digestion (TPAD). In TPAD system, thermophilic digestion precedes mesophilic digestion. The thermophilic stage in TPAD system is 3-5 days whereas it is 15-20 days in mesophilic stage of TPAD system (Dichtl, 1997).

Mesophilic anaerobic digestion (37°C) has been more in use for sludge stabilization because of its higher process stability than thermophilic digestion (55°C). Thermophilic
digestion produces high VFA concentration (Gavala et al., 2003; Zinder, 1986) and is inhibited by higher amount of substrate or product in the digester (Lettinga, 1995).

**Aerobic Digestion**

Aerobic digestion involves the oxidation of biodegradable and microbial cellular matter by aerobic microorganisms resulting in the overall reduction in the mass of sludge and generation of finite amount of stabilized cell mass (McFarland, 2001).

In an aerobic digestion, biodegradable particulate organic matter is hydrolyzed converting it into biodegradable soluble organic matter, releasing ammonia-N and phosphate. The biodegradable soluble organic matter thus produced is then converted into water, carbon dioxide and active biomass through the action of heterotrophic bacteria. The biomass then decays and generates additional carbon dioxide and water and debris. The aerobic digestion process does not affect the non-biodegradable organic matter in the sludge. The terminal electron acceptor used for the oxidation is either dissolved oxygen or nitrate-N (Grady et al., 1999). The presence of heterogeneous population of microorganisms in an aerobic digester makes it a complex ecosystem. One microbial species can serve as a food source to other members of the population. Thus the degradable matter in the sludge is reduced. The digested product is an odorless, stable matter with good dewatering characteristics (Nevim et al., 2002).

The factors affecting the performance of aerobic digestion are solids retention time, temperature, pH, mixing, solids type and biosolids configuration (Grady et al., 1999). In aerobic digestion, criteria for quantifying the degree of stabilization of biodegradable organic matter are the VSS destruction efficiency and the specific oxygen uptake rate (SOUR) of the digested solids (Grady et al., 1999).

In aerobic digestion, two processes can occur, ammonification and nitrification. Soluble organic nitrogen can be mineralized to form free ammonium nitrogen through respiration of amino acids (ammonification) and if proper conditions are present, chemolithotrophic
bacteria can use the ammonium nitrogen to synthesize new cell material (Anderson and Mavinic, 1993). In nitrification, aerobic autotrophic nitrifying bacteria oxidize ammonium forming nitrate. Following nitrification, heterotrophic denitrifying bacteria can reduce nitrate, forming nitrogen gas. These steps involve two bacterial populations: ammonia oxidizers (eg. *Nitrosomonas*) and nitrite oxidizers (eg. *Nitrobacter*) (Bernet et al., 2001). Nitrification is influenced by DO concentration (Stenstrom and Poduska, 1980). When DO is not limiting, the sludge age affects the partial nitrification process and irrespective of sludge age, ammonium is completely converted to nitrite if the aeration is provided intermittently (Pollice et al., 2002). Nitrification affects the pH of the digester. The digester efficiency could be enhanced by controlling the pH between 6 and 8 (Anderson and Mavinic, 1993). The other factor affecting nitrification is temperature. Nitrification occurs at maximum rate at 30°C (Bhargava and Datar, 1989).

During denitrification, nitrate is converted to nitrous oxide (N₂O), then to nitric oxide (NO) then finally to nitrogen gas (N₂) (Madigan et al. (2003). In denitrification process, nitrate or nitrite is used as an electron acceptor and organic carbon as an electron donor. So, low levels of COD or carbon source inhibits denitrification.

The progress of aerobic digestion system depends on the properties of the raw sludge (Nevim et al., 2002). The stabilization of aerobic digestion is influenced by various factors as: temperature, SRT of sludge, oxygen uptake rate (OUR) and mixing rate (Khalil et al., 2000). Aerobic digestion has many advantages, such as, low capital cost, stabilized sludge with no odor and easy operation. Also aerobic digestion gives a higher reduction in pathogenic indicator organisms and pathogens than storage of the sludge (Rao et al., 1993). However, this process has no energy recovery and is costly regarding the energy costs associated with continued aeration (Bernard and Gray, 2000).

*Use of ORP to monitor aerobic sludge digester*

The oxidation-reduction potential (ORP) is the measure of the activity of electrons in a system. In a biological system, the observed ORP signifies the net electron activity of all
the oxidation-reduction reactions and so represents the oxidative status of the system (Kjaergaard, 1977; Koch and Oldham, 1985). Anaerobic fermentation processes such as methane production, have been monitored by ORP (Ishizaki et al., 1974; Koch and Oldham, 1985). Researchers have found that ORP is a sensitive parameter at low oxygen concentrations and it varies linearly with log of oxygen (Ishizaki et al., 1974). Results of Radjai et al., (1984) have showed that bacterial activity changes the ORP and ORP can also influence bacterial activity.

Peddie et al. (1990) demonstrated in their study that ORP can be related to the nitrate and oxygen concentrations and can be used to monitor biological systems. Similar results have been shown by Sekine et al. (1985). They showed that the nitrification rate and ORP are linearly related and ORP can be used as a control parameter for nitrification and oxygen demand. Sekine et al. (1985) also reported that the ORP varied from +300 to -300 mV. This indicates that the system was changing between aerobic and anaerobic conditions. Kim and Hao (2001) studied the effect of total cycle time and aeration ratio on the performance of an alternating anoxic and aerobic system. They found that the aerobic phase can be controlled by a control point on the pH profile (ammonia valley) and the anoxic cycle can be controlled by a point on the ORP profile (nitrate apex). The point on the pH profile indicates the end of nitrification and the point on the ORP profile indicates the end of denitrification. Peddie et al., (1990) have reported that ORP, through control of air supply in an aerobic digester, can bring about cost savings in daily operation of many WWTPs.

**Monitoring and Control of Anaerobic and Aerobic Digester**

Operation of anaerobic digestion is influenced by many factors such as SRT, Volumetric Organic Loading Rate (OLR), Total Hydraulic Loading, temperature, pH, Inhibitory and toxic materials, nutrients, mixing, waste type (Grady et al., 1999). The temperature of the digester should be kept at an optimum range and changes in temperature should be less than 1°C in a day. The optimum pH of an anaerobic digestion is between 6.8 and 7.4 (Grady et al., 1999). Lay et al., (1997) has reported that at pH lower than 6.3 and higher than 7.8, the methanogenesis rate decreases. A lower pH inhibits methanogens while a
higher pH increases release of ammonia in the reactor which causes toxicity. When methanogenesis is decreased, organic acids accumulate in the digester, decreasing pH and causing failure of the system. A pH decrease in the digester due to high organic loadings or toxic materials can be adjusted by adding alkalinity. Eldem et al., (2004a) and Eldem et al., (2004b) reported that at a constant pH, as the ammonia concentration increased, methane production decreased and free ammonia in an anaerobic digester was more toxic than the ammonium ion.

Gomec and Speece (2003), in their study of mesophilic anaerobic digestion, observed that COD and methane production were controlled by pH in both primary and secondary sludge. The total COD and biogas production were estimated using VSS destruction.

Though pH may be the major parameter to control anaerobic processes, several papers have proposed that pH may not be a good indicator of anaerobic digester performance (US EPA, 1976; US EPA, 1979). The better parameters to indicate any upsets of a digester are VFAs, alkalinity and biogas production rate. The VFA to alkalinity ratio represents the presence or absence of organic acids and buffering capacity of the digester. Increase in acidity of the digester indicates a decrease in buffering capacity and failure of the system. Biogas production in an anaerobic system is related to the amount of stabilized biodegradable organic matter and the methanogens in the reactor (Grady et al., 1999). When methanogens in a reactor are affected, methane production of the digester is also affected. Also, in a constant condition and constant-composition feed, the proportion of methane converted to COD or VS is constant. So any deviation to this proportion indicates imbalance in the digester performance.

The performance of an aerobic digester is influenced by its design. For example, in winter, the operation is affected by the heat loss. Also, an aerobic CSTR which is designed in series rather than a single CSTR can improve performance (Grady et al., 1999). Peddie et al., (1990) suggested that ORP can be used as a parameter to monitor and control aerobic sludge digestion. In their results, it is proposed that low oxygen concentrations in an aerobic reactor are indicated by ORP. As with anaerobic digester, an
aerobic digester performance is influenced by various factors. One such parameter is the maintenance of pH in the digester which can be achieved by the use of chemical pH control, aerobic-anoxic digester or auto-thermal aerobic digester. Al-Ghusain et al., (1995) have demonstrated in their results that pH-profile of an anoxic-aerobic reactor can provide different control points indicating different stages (nitrification/denitrification) in the cycles. They have also reported that pH profile is more effective than using ORP profile for monitoring and controlling digester performance.

VS Destruction and Nitrogen Removal in Anaerobic Digestion

VS destruction in an anaerobic digestion depends on various factors. Some of these are pH, temperature, SRT and the characteristics of sludge. Secondary sludge is considered to degrade slower than both primary sludge and mixture of primary and secondary sludge. Kugelman and Guida (1989) compared mesophilic and thermophilic digestion for volatile solids reduction. They found that thermophilic digestion showed poor performance in terms of process stability due to high VFA production and had poor supernatant quality. But, they also observed that the reduction of organic solids was higher in the thermophilic digester than in the mesophilic digester. Similar data showing greater VS destruction and total coliform destruction in thermophilic digestion have been reported by (Buhr and Andrews, 1977; Song et al., 2004). Garber (1982) found that at a 20-day solid retention time, the VS reduction for mesophilic anaerobic digestion was 68 % while it was 65 % for thermophilic anaerobic digestion. Song et al. (2004) also found higher specific methane yield, effluent quality and process stability in mesophilic digestion compared to thermophilic digestion.

Researchers have shown that 2PAD process (Ghosh et al., 1995) and TPAD process (Han and Dague, 1997; Inman et al., 2004; Shang and Sung, 1998; Streeter et al., 1997; Vik and Olsen, 1997) have greater VS reduction than conventional single-stage digestion. Han and Dague, (1997) have also reported that in TPAD system, the thermophilic stage offers the advantage of pathogen destruction. With TPAD, the resulting biosolids meet the requirements for Class A biosolids with respect to pathogen destruction and pathogen
vector attraction reduction criteria as defined by United States CFR 40 Part 503 regulations (Han and Dague, 1997; Streeter et al., 1997; Vik and Olsen, 1997; Vandenburgh and Ellis, 2002).

Moen et al., (2003) operated anaerobic thermophilic (55°C) and mesophilic (35°C) digesters at 15, 10, 6 and 4 day SRTs in parallel, fed with mixture of primary and secondary sludge. They found that at all SRTs, the thermophilic digesters had higher soluble COD concentrations though the volatile solids destruction efficiencies were similar. They also reported that at all SRTs; the thermophilic digestion had more protein destruction resulting in higher NH$_3$-N in the system. The protein destruction releases more sulfur based amino acids generating organic sulfur based gases in the biosolids and the high amount of ammonia in the digesters can toxify the system. These researchers found similar gas production rates and methane content in both digesters at SRTs 10 days and greater; at SRTs 6 days and less, the thermophilic digester had greater gas production with higher methane content. Volatile fatty acids were also higher in thermophilic digester at all SRTs.

In aerobic digestion, VS reduction depends on the temperature, SRT, nature of the feed sludge. Jaworski et al., (1963) proposed that VS destruction decreased at low temperature. Grady and Lim (1999) suggests that VS destruction of an aerobic digester is influenced by temperature and SRT together, and VS destruction efficiency depends on the biodegradability of the solids. At lower temperature, longer SRTs are needed while shorter SRTs are required at higher temperature for same percentage of VS destruction. Secondary sludge is more aerobically-degradable than primary sludge (Counts and Shuckrow, 1974). The fraction of biodegradable components in both of the sludges differs, which in turn is influenced by the SRT from which it came from. Sludge coming from a system with a long SRT has less biodegradable fraction and is more stable, so may not need further stabilization. Sludge coming from a short-SRT system, however, needs to be further digested and stabilized.
Combined Anaerobic and Aerobic Digestion of Sludge

Pre-Aerobic and Post-Aerobic Digestion of Anaerobic Sludge

Anaerobic or aerobic digestion of sludge can be used to remove organic matter. Anaerobic sludge processes have not been widely used in other parts of the world. One of the disadvantages of anaerobic digestion is the lack of post-treatment process to remove residual organics and nutrients from the effluent. With the view that the anaerobically digested effluent doesn’t meet common effluent standards, some researchers have focused on combinations of anaerobic and aerobic processes. The aerobic treatment of sludge after anaerobic digestion has advantages such as simple design technology and minimization of sludge production (Jenicek et al., 1999). The use of combined anaerobic and aerobic digestion of sludge can eliminate the need for a separate sludge stabilization units (Motta La et al., 2007).

In combined anaerobic/aerobic systems, the influent of aerobic reactor is pretreated in the anaerobic digester. Thus a large fraction of organic matter is already biodegraded and eliminated. So the residual organic matter from the anaerobic digester requires a lower oxidation capacity in the aerobic digester for nitrification and further degradation of the residual organics. Castillo et al., (1997) has proposed that the combined anaerobic/aerobic digestion of sludge has lower energy consumption and less excess sludge production than a single conventional anaerobic digestion.

VS and COD Removal in Combined Anaerobic and Aerobic Sludge Digestion

Pagilla et al. (1996) found that aerobic thermophilic pretreatment of anaerobic mesophilic digested sludge achieved United States 40 CFR 503 Part (b) Class A biosolids requirement for pathogen reduction with improved VS reduction. Combining anaerobic and aerobic digestion of sludge uses both of their advantages, i.e. biogas production of anaerobic and COD and VS destruction of aerobic digestion (Rous and Zupancic, 2004).
In a later study by Tapana and Pagilla (2000), it was reported that pre- and post-aerobic treatment of anaerobic sludge reduced VS up to approximately 65%, while a single mesophilic anaerobic digestion had only about 51% VS reduction, at SRT of 15 days. Similar results were obtained by Pagilla et al. (2000). In a batch experiment on digestibility of waste activated sludges, Park et al. (2006) found that combined anaerobic/aerobic or aerobic/anaerobic digestion of sludge produced more VS destruction (63%) than single anaerobic digestion processes. This implies that with single digestion (may it be anaerobic or aerobic), some degradable organic matter remains in the floc. This degradable organic fractions are further degraded when an additional digestion system is applied, which accounts for the additional VS destruction in a combined digestion. VS destruction in anaerobic digestion is affected by Fe content in the sludge (Novak et al., 2007) while in aerobic digestion, divalent cations in the sludge influenced the VS removal efficiency (Park et al., 2006). As obtained by Park et al. (2006), in anaerobic digestion, the VS reduction decreased with increase in sodium and increased with increase in iron content in the influent (Novak et al., 2007). Anaerobic digestion resulted in degradation of protein and thus reduced the iron in the sludge, accounting for the VS destruction while aerobic digestion degraded organic matter and released polysaccharides and divalent cations in the solution.

Kumar (2006) found more than 40% overall VS reduction in sequential anaerobic/aerobic digestion at 3 day SRT of the aerobic reactor even when the anaerobic digester wasn’t functioning properly. In his studies, he also demonstrated additional VS reduction of 10% to 20% when the anaerobic sludge was digested aerobically. Organic solids in mesophilic anaerobically digested sludge, with 30d SRT, are reduced by 20% through post-aerobic digestion (5d SRT, 30°C) (Parravicini et al., 2004). Later in 2006, in another study, Parravicini et al. (2006) investigated further stabilization of digested sludge under both anaerobic and aerobic conditions. They found that aerobically stabilizing/digesting the already digested sludge degraded the residual VSS significantly. In a later study by Parravicini et al., (2008), they reported that post-aerobic digestion (6d SRT, 36°C) of mesophilic anaerobic digested sludge (30d SRT) reduced additional 16% organic solids.
Tapan and Pagilla (2000), in their research, also demonstrated that the pre-and post-aerobic treatment reduced COD by 56±67% while only 44±60% reduction was obtained in single mesophilic anaerobic digestion. In sequential anaerobic/aerobic digestion of sludge, at 3 day SRT of aerobic digestion, up to 60% to 70% COD removal can be achieved (Kumar, 2006).

**Nitrogen Removal in Combined Anaerobic and Aerobic Digestion**

Anaerobic digestion degrades protein which in turn produces a high amount of ammonium nitrogen in the digester. Fifty percent of sludge bound nitrogen is released in the form of ammonium in the digestion and centrifugation of sludge (Siegrist, 1996). Dewatering of digested sludge can produce streams with ammonium concentration up to 2 kg m$^3$ (Strous et al., 1997). When the stream is recycled to the head of the WWTP, it increases the nitrogen load to the plant. Partial removal of nitrogen is achieved by intermittent aeration of digested sludge through nitrification and denitrification. In nitrification, aerobic autotrophic nitrifying bacteria oxidize ammonium forming nitrate. Then, heterotrophic denitrifying bacteria reduce nitrate to form nitrogen gas. Optimum nitrification conditions should be maintained for nitrification to occur. A low concentration of COD in the influent can inhibit denitrification. So COD/TKN ratio is an important parameter when assessing nitrogen removal of a system. Itokawa et al., (2001) and Nagako et al., (2002) have proposed that the COD/TKN ratio should be 5.0 or above 5.0 for complete nitrogen removal, when other organic material isn’t supplied. Pollice et al., (2002) studied the nitrification process in reactors, with continuous aeration and intermittent aeration and the effect of sludge age on ammonium oxidation to nitrite. The results showed that in unlimited oxygen supply, sludge age was the critical parameter for partial nitrification. In limited oxygen supply, ammonium conversion to nitrite was complete and stable and was not dependent on the sludge age.

Parravicini et al., (2008) have reported that 45% total nitrogen removal can be obtained by intermittent aeration of the anaerobically digested sludge, with an optimum aeration/pause ratio. In their study, they found that the efficiency of NH$_4$-N removal
through nitritation and denitritation was 98%. In a study by Akuna et al., (1994), it was shown that the amount of organic matter left in the digester affects the amount of ammonia nitrogen to be nitrified. In this study, it is reported that the heterotrophic growth in an aerobic digester out-competes autotrophic nitrifiers and so completely inhibits nitrification. It is also proposed that at low aeration rate in the aerobic digester, the nitrogen loss through denitrification was significant.

Nitrogen removal in a combined anaerobic and aerobic digestion system depends on the recycle-to-influent ratio (Akuna et al., 1994). In the study by Akuna et al., (1994), complete added nitrogen removal was seen at recycle-to-influent ratios of 4 and 5. They also found that on increasing the ratio from 0 to 5, the methane production rate in the anaerobic digester decreased whereas the nitrogen gas production rate increased.

Kumar (2006) studied the combined anaerobic and post-aerobic digestion of sludge with aerobic digestion SRTs at 3, 6 and 9 days. He found that ammonia removal (80%) and TKN removal (more than 50%) were achieved when the SRT of the aerobic digester was increased from 3 to 9 days. Bernet et al. (2000) studied batch scale anaerobic-aerobic digestion of piggery wastewater with aerobic cycle of 24hr, in which the anaerobic digester was fed with raw wastewater and recycled effluent from the aerobic digester. The aerobic reactor showed nitrification and denitrification (during filling duration), while denitrification followed by methanogenesis were observed in the anaerobic reactor. They found 85% to 91% TKN removal and 81% to 91% TOC removal in the final effluent and the removal efficiency depended on the recycle-to-influent ratio. Lower the ratio, lower was the removal percentage and partial denitrification in the aerobic digester increased the nitrogen removal efficiency.
VFA Formation in Sludge Digestion

Volatile fatty acids (VFAs) are produced during anaerobic processes of methane fermentation. Anaerobic digester with high VFA production causes decrease in pH which ultimately leads to digester failure. The efficiency of the digester can be measured by regulating VFA concentrations. The VFA produced from the degradation of the biodegradable compounds are mainly acetic acid, propionic acid and butyric acid. According to Buyukkamaci and Filibeli (2004), the VFA concentrations are less in the top of the digester and increases from top to bottom. A higher COD in the influent produces more VFAs. Also based on VFA and pH measurements, they have concluded that methanogens are more active in the upper part while acetogens have higher activity on the bottom. The concentrations of acetate, propionate and butyrate are lower in mesophilic anaerobic sludge digestion than in thermophilic digestion (Meon et al., 2003).

Fothergill and Mavinic, (2000) found that VFA production in an autothermal thermophilic aerobic digestion (ATAD) increases with decrease in aeration and retention time. They also observed increased accumulation of VFA when the mixture of primary sludge and secondary sludge was used as feed to the digester along with increased release of phosphorous and ammonia nitrogen. Similarly, Chu et al., (1994) found that propionate concentration in an ATAD changes with increase or decrease of aeration rate.

Biosolids and Odor

Murthy et al., (2002) has reported that anaerobically digested biosolids have the potential to produce unacceptable odors. Volatile organic sulfur compounds (VOSCs) are the primary group of compounds associated with odor from anaerobically digested biosolids (Forbes et al., 2003; Higgins et al., 2002). Methanethiol (MT) and dimethyl sulfide (DMS) are the major odor compounds associated with dewatered sludge cakes (Novak et al., 2006). DMS forms under both aerobic and anaerobic digestion of sludge from the
degradation of sulfur-containing compounds while MT forms under anaerobic conditions (Bremner and Banwart, 1976; Lomans et al. 1999; Lomans et al. 2001). Verma, (2005) and Higgins et al., (2008) found that iron content of the sludge is correlated with the generation of odor from the dewatered sludge cake. Iron binds the protein in the biosolids, so more iron in the sludge cake will decrease the availability of bound-protein (Higgins et al., 2008). Hydrogen sulfide is present in the raw sludges. If iron is present in the treatment units, H₂S precipitates out as FeS (Novak et el., 2006). So in wastewater with low iron concentrations, H₂S will be a problem. In contrast to these results, Novak et al. (2007) found increasing VOSCs with increase in iron content of the sludge. In their method, the sludge was first dewatered centrifugally, and then dewatered again using press. The shearing in centrifuge may have released more previously-undegraded iron in the solid cake and thus the VOSCs generation from the cake increased. So odor generation in a cake solid also depends on the method and extent of dewatering and shearing.

Witherspoon et al. (2004) noted that biodegradable matter, some proteins, present in the primary sludge are broken down by the diverse microbial community in the secondary sludge which is why primary and secondary sludge mixture have emission of odorous compounds before and after digestion. Higgins et al., (2008) found that production of odorous VSCs was affected by the concentration of methionine, a sulfur-containing amino acid.

Forbes et al. (2003) and Higgins (2002) have found that during biosolids cake storage, these VOSCs increase in concentration, and then decrease gradually to below detection limits. The major volatile sulfur compounds (VSCs) are hydrogen sulfide (H₂S), methanethiol (MT), dimethyl sulfide (DMS), dimethyl disulfide (DMDS), dimethyl trisulfide (DMTS) and carbon disulfide (CS₂).
Indole production from the sludge cakes are associated with pumping and storage of the cakes and it is very persistent in its odor potential (Novak et al., 2006). There has not been much research done on the processes of odor formation from the digested and dewatered biosolids cake. But research related to VSC production in freshwater sediments (Bak et al., 1992) and human oral cavity (Persson, 1992; Persson et al., 1990) by oral bacteria in periodontal diseases, has led to a great deal of understanding possible mechanisms for VOSC production and their degradation.

H₂S and MT can occur from the anaerobic biodegradation of L-cysteine and L-methionine, both sulfur containing compounds (Persson, 1992; Persson et al., 1990). Proteins are degraded to form peptides, which are broken down to amino acids. The amino acids are then degraded, forming VOSCs. The substrate for the reaction is protein which is available in biosolids, with protein content present up to 50% (Higgins et al., 2006). Higgins et al., (2006) observed increase in the peak concentration of MT with the increase in the mass of methionine suggesting the possibility that methionine concentration might determine the odor production potential of biosolids cake. The other pathway for VOSC production is methylation of H₂S and MT. Methylation of H₂S to form MT and methylation of MT to form DMS in freshwater sediments, soils and water by anaerobic bacteria have been found to occur (Bak et al., 1992; Drotar et al., 1987; Lomans et al., 1997 and 2001). Persson et al., (1990) notes that methionine and cysteine degrade only to MT or H₂S respectively, which rules out the production of DMS from these amino acids. Experiments conducted by Higgins et al., (2006) show the presence of DMS in headspace of biosolids sample. From these researches, it is shown that methylation of H₂S and MT occurs to form DMS. When syringate, a methyl group is added to the biosolids cake, the DMS production is increased (Higgins et al., 2006). Oxidation of MT to DMDS occurs readily in the presence of oxygen (Chin and Lindsay, 1994; Fritz and Bachofen, 2000; Kelly and Smith, 1990; Parliament et al., 1982; Tulio et al, 2002). Higgins et al. (2006) found that DMDS production from a serum bottle stopped, with increase in MT concentrations, when the oxygen concentration was exhausted.
VOSCs production and degradation are balanced in system. In freshwater sediments, because of this balance, little VOSCs are emitted (Lomans et al., 1999, and 2001). In anaerobically digested biosolids also, VOSCs (MT, DMS and DMDS) are only emitted when the methanogens in the solid cake are disturbed such as when there are toxic conditions in digesters or following high-sheared dewatering and increased polymer dosing (Iranpour et al., 2003; Zitomer and Speece, 1995). The degradation of VOSCs is carried out by methanogens (Higgins et al., 2006). Lomans et al., (1999) have also shown that DMS and methanethiol are degraded by methanogens to sulfides. Chen et al., (2005) and Higgins et al., (2006) performed an experiment to determine the role of methanogens in degrading VOSCs. They added bromoethanesulfonic acid (BESA) to the dewatered biosolids. The BESA is a methanogenic inhibitor. The results showed that when BESA was added to the biosolids cake, the production of MT and DMS increased. This demonstrates that due to the inhibition of methanogens by BESA, the methanogens were incapable of cycling and degrading the VOSCs. Tepe et al., (2008) observed that with bio-augmentation of biosolids cake with strains of *Bacillus*, *Pseudomonas*, and *Actinomycetes* and various micronutrients, methanogenesis and odor control was enhanced. According to Higgins et al. (2006), the odor potential of a cake solid is the amount of TVOSC that can be produced by a sludge cake and is the concentration of TVOSC peak measured with addition of BESA.

Kumar (2006) studied sequential anaerobic-aerobic digestion of sludge. He found that increase in anaerobic digestion SRT decreased odor generation. Similar results have been obtained by Verma, (2005). Kumar (2006) also observed that aerating the digested sludge produced biosolids with less odor generation.

Tapana and Pagilla (2000) operated lab-scale experiments with aerobic thermophilic pretreatment and aerobic thermophilic post treatment of mesophilic anaerobic digestion at 15 days and 15.5 days SRTs. They found that the average H$_2$S concentration of the aerobic pretreatment was significantly lower than those of aerobic post treatment and the single mesophilic anaerobic sludge.
Dewatering and Biosolid Odors and Factors affecting sludge characteristics

As stated in Novak (2006), dewatering is affected by the type of the dewatering equipment and the type of sludge to be dewatered. Liquid anaerobic digested sludges have lower volatile odor compounds than dewatered, stored sludge (Higgins et al., 2006; Winter and Duckham, 2000). The cause for this is hypothesized to be the imbalance in the production and degradation rates of VOSCs by dewatering. Dewatering equipment, polymer dose, cake handling and transport can affect VOSC production after dewatering (Higgins et al., 2006). Muller et al. (2004) found that more shear is created with high-solid centrifuge producing higher cake solids. In another experiment, Murthy et al. (2003) observed increased odor production from sludge cakes dewatered using high-solids centrifuges than cakes dewatered using medium-solids centrifuges. The high-solid centrifugation process has also been shown to break up the sludge floc more and thus produce greater VOSCs (Higgins et al., 2002; Murthy et al., 2003). High-solids centrifuge produces higher shearing and higher cake solid due to their high speeds. As stated in Novak (2006), cake solids made from high-solid centrifuge dewatered sludge produces more odor compounds than that made from low-solid centrifuge or belt filter dewatered cakes.

Dewatering process that generates greater shear breaks up the floc more which releases more EPS-bound protein. Due to the shear, the methanogens in the cake undergo cell lysis and damage, thus inhibiting their activity. This would eventually increase VOSC concentrations as methanogens would be unable to degrade the odor compounds. Higgins et al., (2002) also found increase in VOSCs with increase in polymer dose due to the increase in available protein in the sludge cake. Muller et al., (2004), also measured VSC production from the sludge cake in which shear was applied with no polymer addition. They found that there was no significant odor compounds produced without polymer addition. Polymer binds to the EPS protein released from the floc by shear and makes it bioavailable. The VOSCs production rate is also affected by the mechanism of transportation of the sludge cake. Murthy et al., (2002) found that transporting the cake in high-shear conveyance increased the production rate of VOSCs.
Novak et al. (2007), in their study of centrifugally dewatered sludge cakes, found that Al, Ca, Mg, monovalent to divalent ratio had no correlation with odor generation. They recommended in their results that it is not possible to predict odor generation from the VS reduction from anaerobically digested sludge. Novak et al., (2007) proposed that aluminium content in the sludge had no correlation with odor generation. But Adams et al., (2007) observed decrease in TVOSCs with increase in aluminium in the sludge. They proposed that aluminium could bind organics in the cake and make them unavailable for degradation thus causing decrease in VOSC generation. So odor generation of a cake solid depends on type of sludge and dewatering method, the equipment used for shearing and the extent of shearing.

**Dewatering and Polymer Conditioning of Digested Sludge, Role of Cations and Biopolymers**

In waste activated sludge, dewatering is promoted with the addition of polymers. Novak et al., (2003) has proposed that waste activated sludges have two types of biopolymers. In one fraction of biopolymer, calcium and magnesium ions bind polysachharides and proteins while the other fraction has iron and aluminium binding protein, polysachharides and humic acids (Park et al., 2002). Novak et. al., 2001, found more protein released by anaerobic digestion than by aerobic digestion. In their study, the anaerobically digested sludge had a reduced dewatering capability than the aerobically digested sludge, shown by the increasing Capillary Suction Time (CST). Later, in another study by Novak et al. (2003), and Novak and Park (2004), it has been shown that when waste activated sludge is digested anaerobically, floc destruction occurs, iron is reduced and biocolloids are released in the solution. The un-degraded fraction of biocolloids in the solution reduces its dewatering capability contributing to increase in polymer dose (Bivins and Novak, 2001). Novak et al., (2003) also found that during aerobic digestion, along with protein and polysaccharides, calcium and magnesium were also released (Novak et al., 2003). These researchers have also shown that for anaerobic digestion, the biocolloid that was responsible was protein while polysaccharides were the primary biocolloids in aerobic digestion.
Novak et al., (2004) studied variety of digested sludge and its dewatering characteristics. They found that as solid destruction increases due to digestion, dewatering ability decreases and polymer conditioning requirements increases. They have also proposed that cation content of the sludge may determine biopolymer content of the sludge and cation affects solids destruction which influences the dewaterability of the digested sludge. Novak et al. (2001) demonstrated that iron reduction and solubilisation in sludge reduces its floc strength and decreases the dewatering ability.

Murthy and Novak (1999) studied the effect of adding divalent cations to the performance of aerobic digestion of waste activated sludge. They found that higher calcium and magnesium content in the sludge showed better effluent quality, in terms of lower polymer dose requirement, better dewatering, better floc, and lower soluble EPS. The divalent cations were involved in bridging proteins and polysaccharides in the floc together while monovalent cations were released in the solution along with protein and polysaccharides and were not able to bind the floc. Murthy and Novak (1999) and Sobeck and Higgins (2002) have proposed that sludge with high monovalent cations (mainly sodium) have poor settling and dewatering properties. Higgins and Novak (1997), in their study of activated sludge characteristics, found that monovalent to divalent (M/D) ratio and specific resistance to filtration (SRF) of sludge were positively related. They found that as the M/D ratio increased above 2, the dewatering ability of the sludge decreased. The divalent cations within the floc are replaced by the monovalent cations, thus influencing the floc characteristics and dewaterability. So cation – monovalent and divalent – influences the digestion of sludge.

Erdinçler et al., (2001) has found that low energy-level blending of the sludge-polymer reduces the polymer dose. Intra-cellular and extracellular polymers are released with blending leading to an increase in the flocculation rate. Erdinçler et al. (2001) also proposed that polymer at low energy levels causes a considerable reduction the polymer dosage required. Mikkelsen and Keiding (2001) found that with increase in turbidity of the sludge and solids concentration, the optimum polymer dosage also increases. The
polymer dose of sludge is also influenced by Gt value of the shearing equipment, where G is the mean velocity gradient (s⁻¹) while t is the time of shear (sec). Lynch and Novak (1991) have found that Gt value of high-solid centrifuge ranges from 100,000 to 120,000 and it produces high shearing due to their high speed and also increases the optimum polymer dose of the sludge.

Kugelman and Guide (1989) reported that thermophilic sludge had a higher polymer demand than mesophilically digested sludge. In a similar study by Reusser and Zelinka (2004), it was found that the thermophilic sludge and TPAD sludge had respectively 3.1 times and 1.8 times more polymer demand than that of mesophilic sludge.

Bivins and Novak, (2001) studied dewaterability characteristics of waste activated sludge (WAS) and found that TPAD system had higher polymer demand than mesophilic digestion. The higher amount of protein and polysachharides released from thermophilic digestion caused higher polymer demand.

Regarding the pre- treatment and post- treatment of anaerobically digested sludge, Tapana and Pagilla (2000) has demonstrated that the pre- and post-aerobic treatment of mesophilic anaerobic sludge produced digested sludge with better dewaterability characteristics than the single mesophilic anaerobic sludge. Subramaniam (2005) found that sequential anaerobic/aerobic produces sludge with lower CST, lower polymer dose and lower bound-water content than single anaerobically digested sludge. Kumar (2006) studied sequential anaerobic-aerobic digestion of sludge. He found that the 50% biopolymers were removed in the final effluent and it had lower polymer dose requirement that single-digestion sludge with improved dewatering characteristics.
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Chapter 2

Enhanced Solid and Nitrogen Removal in Anaerobic/Aerobic/Anaerobic Digestion of Sludge

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Chapter 2

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Abstract

Combination of both anaerobic and pre- or post- aerobic digestion of sludge reduces more volatile solids than a single anaerobic or aerobic digestion. With an objective for efficient VS and nitrogen removal, digestion of sludge (mixture of primary and secondary sludge in 1:1 ratio by weight) was performed using Conventional MAD, Sequential Anaerobic/Aerobic (Ana/Aer) and Anaerobic/Aerobic/Anaerobic (An/Aer/An) digestion and compared for efficient organic carbon and nitrogen removal. An/Aer/An sludge digestion was investigated with two phases: An/Aer/An – A (Wastage from Anaerobic Reactor) and An/Aer/An – B (Wastage from Aerobic Reactor). All of the phases were studied using lab-scale reactors. The anaerobic reactors in all the phases were operated in mesophilic (35°C) conditions while the aerobic reactors were operated under room temperature (20°C). Different operational parameters, such as COD removal, VS destruction, biogas production and Nitrogen removal were studied and results were compared among different processes. The study found that An/Aer/An – B (wastage from aerobic reactor) provided better effluent characteristics than An/Aer/An – A (wastage from anaerobic reactor), Ana/Aer or conventional MAD. Both An/Aer/Ana – A and An/Aer/An – B gave 70% VS removal, compared to 50% with single MAD and 62% with only Ana/Aer. COD removal of both An/Aer/An – A and An/Aer/An – B was 70%, while it was 50% and 66% for single MAD and Ana/Aer respectively. In the aerobic reactor of An/Aer/An – A and – B, and Ana/Aer stages denitrification followed by nitrification was observed. An additional removal of carbon and nitrogen occurred in the aerobic reactor. Nitrogen removal data showed that with An/Aer/An – A (wastage from anaerobic reactor), 43% TKN removal was obtained, while it was almost 70% for
An/Aer/An – B (wastage from aerobic reactor). The An/Aer/An – B system was better than Ana/Aer (with 64% TKN removal) in TKN removal. An/Aer/An system (both A and B) proved better than single anaerobic digestion or sequential Ana/Aer system in VS and COD reduction. An/Aer/An system with wastage from the aerobic side proved best in the effluent characteristics.

**Keywords** – Mesophilic Anaerobic Digestion, Aerobic Digestion, VS reduction, Nitrogen removal, Nitrification, Denitrification, Organic Carbon removal

**INTRODUCTION**

Sludge produced from a wastewater treatment plant should be stabilized and minimized before it is disposed or used for land application. Minimization of sludge generated from WWTP is very important because of cost, health and environmental factors associated with the transport and disposal of the biosolids. Under 40 CFR 503 Part (b) sludge reuse and disposal regulations (EPA 1992), certain level of treatment of the sludge is required for pathogen deactivation before its land application. To reduce the overall adverse environmental and health effect from the disposal of the biosolids, the desired treatment system produces minimum sludge mass with reduced pathogen and odor, less energy requirements and more stability.

Different treatment processes for achieving Class A biosolids have been investigated by many researchers. Anaerobic digestion of wastewater sludge has widely been in application for stabilization of sludge, because of its advantage in that it produces biogas as a byproduct. Mesophilic (35°C) anaerobic digestion is more commonly used than thermophilic digestion because of its higher stability. But since mesophilic digestion is not considered efficient in reducing pathogens and does not produce class A biosolids, thermophilic digestion has been gaining interest. However, thermophilic (55°C) digestion has not only been associated with higher metabolic rate of microorganisms, higher pathogen destruction and higher methanogenic potential at lower HRT (Aoki and Kawase, 1991; Fang and Chung, 1999; Maibaum and Kuehn, 1999; Zabranska et al.,
2000) but also with poor process stability and poor supernatant quality (Rimkus et al., 1982) and poor dewatering quality of the effluent (Fang and Chung, 1999; Maibaum and Kuehn, 1999; Kim et al., 2002). Thermophilic digestion efficiency also depends on temperature, organic loading rate (OLR), and the feed and higher energy is required for its operation (Kim et al., 2002; van Lier, 1996). Another concern for thermophilically digested biosolids was the recurrence of fecal coliforms (Iranpour and Cox, 2006). Jolis (2006) observed positive relationship in temperature and recurrence of fecal coliforms in biosolids. Because of the process instability, thermophilic digestion is not listed as one of the processes to further reduce pathogens (US EPA, 1989). Rather, according to US EPA (1992) aerobic thermophilic digestion has been included as a process to reduce pathogens further and to reduce volatile solids efficiently. But it is also associated with high energy requirement for oxygen supply as well as for temperature maintenance.

In anaerobic process, the biodegradation of organic compounds forms ammonia. So although anaerobic sludge digestion produces methane that can be used to generate energy, it also produces end products with liquid and solid residual organic matter which poses problem in disposal. Odegaard, (1988) reported nitrification-denitrification post-treatment of the anaerobic sludge as a widely used option to treat ammonia. In aerobic digestion of anaerobically digested sludge, the ammonia is oxidized to nitrite and nitrate which in turn are reduced completely through denitrification, using volatile fatty acids as carbon source (Akuna, 1995).

Knudsen et al., (2000) observed in their research that the anaerobically digested sludge had higher amount of organic pollutants remaining than aerobically digested sludge. This may be because the organic matter could be degraded under aerobic conditions and not in anaerobic conditions. Later Novak et al., (2004) also found that the combination of both anaerobic and pre- or post- aerobic digestion of sludge reduced more volatile solids than a single anaerobic or aerobic digestion. Previous research by Akunna et al., (1994) showed that coupled anaerobic and aerobic filters removed nitrogen and carbon effectively. Later, Parravicini et al. (2004) studied post-aerobic digestion of anaerobic sludge. They observed that digesting the anaerobic sludge (30d SRT, 38°C) aerobically (5d SRT, 30°C)
stabilized the sludge with an additional 20% solids reduction. Intermittent aeration of the sludge enables it to go through nitrification and denitrification thereby reducing additional total nitrogen. In a later study by Parravicini et al. (2008), they reported that post-aerobic digestion (6d SRT, 36°C) of mesophilic anaerobic digested sludge (30d SRT) reduced organic solids an additional 16%. This has also been demonstrated by Akuna et al., (1994). Tapana and Pagilla (2000), in their research, also demonstrated that the pre-and post-aerobic treatment reduced COD by 56±67% while only 44±60% reduction was obtained in single mesophilic anaerobic digestion. In sequential anaerobic/aerobic digestion of sludge, at 3 day SRT of aerobic digestion, up to 60% to 70% COD removal can be achieved (Kumar, 2006). Kumar (2006) studied the combined anaerobic and post-aerobic digestion of sludge with aerobic digestion SRTs at 3, 6 and 9 days. He found that ammonia removal (80%) and TKN removal (more than 50%) were achieved when the SRT of the aerobic digester was increased from 3 to 9 days.

During sequential anaerobic and aerobic treatment of sludge, if the influent has high nitrogen content, the intermediate metabolic compounds of anaerobic treatment, such as ammonia, might be inhibitory or toxic to the methanogenesis process. Decreased activity in methanogens decreases the efficiency of the treatment process which can affect the downstream aerobic treatment by increasing the organic load. When the activity of methanogens decreases, the efficiency of the treatment also decreases. Novak et al., (2004), they proposed that organic compounds in sludge have many fractions. They can be degradable only under aerobic conditions, only under anaerobic conditions, under both aerobic and anaerobic conditions or cannot be degraded under any condition. So each anaerobic and aerobic digestion of sludge can degrade only some fractions of the sludge. The combination of both of these types of digestion can be complementary to each other in that it can be capable of degrading more fractions in the sludge using both anaerobic and aerobic environments.
RESEARCH OBJECTIVES

Previous research, as mentioned earlier, focuses on sequential anaerobic-aerobic digestion of sludge but a review of the literature did not show much research on anaerobic-aerobic-anaerobic digestion of sludge. Some work regarding the use of anaerobic-aerobic-anaerobic processes for treating wastewater has been found but there is none on sludge. This study was designed to investigate the performance of anaerobic-aerobic-anaerobic digestion of sludge and compare it to anaerobic-aerobic digestion and single stage mesophilic digestion of sludge. The anaerobic/aerobic/anaerobic digestion of sludge was studied with two options to determine the best option in terms of effluent characteristics. The two sludge withdrawal options were to withdraw effluent from the anaerobic digester or withdraw effluent from the aerobic digester. The hypothesis in this study was that the anaerobic-aerobic-anaerobic digestion of sludge would produce effluent with better VS destruction, nitrogen and COD removal, better dewatering properties and less odor compounds in the biosolids, compared with single or sequential anaerobic/aerobic digestion.

The study was conducted in Virginia Tech laboratories and the digesters were operated for a year. This research was designed to:

- Determine the effect of Anaerobic-Anaerobic-Aerobic digestion of sludge on the characteristics of the effluent based on Nitrogen and COD removal.
- Quantify the VS reduction in Anaerobic-Aerobic-Anaerobic digestion of sludge, Anaerobic-Aerobic digestion of sludge and Single conventional digestion of sludge.
- Demonstrate the biogas production capability of the anaerobic digesters.
METHODS AND MATERIALS

Experimental Approach
The study was divided into three phases. The three phases are: Mesophilic Anaerobic Digestion of sludge, Sequential Anaerobic/Aerobic Digestion of sludge and Anaerobic/Aerobic/Anaerobic Digestion of sludge. These are described in detail in the following sections.

I. Mesophilic Anaerobic Digestion of Sludge
The mesophilic anaerobic digester (35°C) was operated as completely mixed reactor (25 L nominal volume and 20 L active volume). This digester served as process control and is termed as MAD 20d SRT.

Fig: 2.1. Digestion configuration of Mesophilic Anaerobic Digestion (MAD) of sludge with arrows representing direction of mass flow (feed and waste).
II. Sequential Anaerobic / Aerobic Digestion

The mesophilic anaerobic digester (35°C) was operated as completely mixed reactor (25 L nominal volume and 15 L active volume). The waste sludge from the anaerobic digester was then digested aerobically. The aerobic digester was 9L nominal volume with 5L active volume. The system is termed as Ana/Aer. The mesophilic digester in the system is termed as MAD 15d SRT.

Fig. 2.2. Digestion configuration of Sequential Ana/Aer digestion of sludge. The mass flow through the digesters is given by the arrows.

III. Anaerobic/Aerobic/Anaerobic Digestion of Sludge

The mesophilic anaerobic digester (35°C) was operated as completely mixed reactor (35 L nominal volume and 30 L active volume). The waste sludge from the anaerobic digester was digested aerobically, then aerobic sludge was centrifuged, the centrate was
discarded while the pellet was re-suspended in raw sludge. The mixture was then fed to the anaerobic digester. The aerobic digester was 9L nominal volume with 5L active volume. This scheme was studied with two options:

- Option A: Waste from Anaerobic Digester (termed as An/Aer/An – A)
- Option B: Waste from Aerobic Digester (termed as An/Aer/An – B)

**Option A: Waste from Anaerobic Digester**

![Diagram of Anaerobic/Digestion Configuration](image)

Fig. 2.3. Digestion configuration of An/Aer/An – A (Wastage from Anaerobic Digester), with arrows showing the direction of mass flow through the anaerobic and aerobic digester.
Option B: Waste from Aerobic Digester

Fig. 2.4. Digestion configuration of An/Aer/An – B (Wastage from aerobic digester), with arrows representing the mass flow through the anaerobic and aerobic digester.

The phases were named as shown in table 2.1. The SRTs for all the digesters for all the phases were also calculated and are provided in table 2.2.
Table 2.1. Names of digesters used in the study

<table>
<thead>
<tr>
<th>Schemes</th>
<th>Acronyms</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional Mesophilic Anaerobic Digestion (served as process Control)</td>
<td>MAD (MAD 15d SRT and MAD 20d SRT)</td>
</tr>
<tr>
<td>Sequential Anaerobic-Aerobic Digestion</td>
<td>Ana/Aer</td>
</tr>
<tr>
<td>Anaerobic-Aerobic-Anaerobic Digestion</td>
<td>Option A: Anaerobic Waste</td>
</tr>
<tr>
<td>Option B: Aerobic Waste</td>
<td>An/Aer/An - A</td>
</tr>
<tr>
<td>An/Aer/An - B</td>
<td>An/Aer/An - B</td>
</tr>
</tbody>
</table>

**SRTs of the System:**

The SRTs for the conventional and anaerobic/aerobic reactors was based on the hydraulic detention time. For the recycle streams, the overall system SRT was calculated based on the solids in the system divided by the wastage rate. The values are shown in Table 2.2.

Table 2.2. SRTs of the anaerobic and aerobic digesters in the systems.

<table>
<thead>
<tr>
<th>System</th>
<th>System SRT</th>
<th>Anaerobic SRT</th>
<th>Aerobic SRT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional MAD</td>
<td>20 d</td>
<td>20 d</td>
<td>----</td>
</tr>
<tr>
<td>Sequential Ana/Aer</td>
<td>20 d</td>
<td>15 d</td>
<td>5 d</td>
</tr>
<tr>
<td>Ana/Aer/Ana Anaerobic Waste</td>
<td>35 d</td>
<td>15 d</td>
<td>5 d</td>
</tr>
<tr>
<td>Ana/Aer/Ana Aerobic Waste</td>
<td>35 d</td>
<td>15 d</td>
<td>2.5 d</td>
</tr>
</tbody>
</table>

All of the digesters were kept in a constant temperature room to maintain the temperature. Plastic, egg-shaped fermenters supplied by Hobby Beverage Equipment Company, were used as anaerobic digesters. A stainless steel thermometer was placed at
the side of the digester. For aerobic digester, 9 L glass digesters (Fisher Scientific) were used. Bubble diffusers were used for maximum oxygen transfer and a compressor was used to supply oxygen. The aeration cycle in the aerobic reactors was 24hr with continuous feed and wasting. Dissolved oxygen (measured before feeding) in the reactor was kept within 2.5 - 3.0 ppm. Distilled water was added each day to counter any water loss due to evaporation in the aerobic reactor before wasting from it.

For mixing of gas in the anaerobic digesters, peristaltic pumps (Cole Parmer 6-600 rpm) were used and the gas was recirculated from the headspace to the bottom of the digesters using Cole Parmer Masterflex Tygon LFL-18 pump tubing. The pumps were operated at 50% of their maximum possible speed. To ensure greater mixing of the digesters before and after feeding, gas recirculation in the digesters was increased by increasing the speed of the pumps to 100%, 10 minutes before sampling and also for 10 minutes after feeding.

The anaerobic digesters were seeded with mesophilic anaerobically digested sludge taken from Pepper’s Ferry Regional Wastewater Treatment Facility, Radford, Virginia. No feeding or wastage was done on the seeded anaerobic digesters for one week to ensure that the microbial communities are acclimatized to the digester environment. After a week, feeding and wasting from the reactor was done. The digesters were monitored for steady-state. Biogas production, pH and sampling and analysis were done only after the determination of the steady-state condition.

The feed for the anaerobic digester was a mixture of primary and secondary sludge (gravity thickened sludge and air flotation thickened waste activated sludge), 1:1 by weight. The primary and secondary sludge were supplied weekly by DCWASA Blue Plains Advanced Wastewater Treatment Facility and shipped overnight. Total solids percentages of both the sludge were measured and a mixture of 1:1 by weight of the sludges with total solid percentage of 5% was made by dilution. The sludge was blended and was stored in a 4°C room until used. To maintain the SRT of both the anaerobic and aerobic digesters, constant volume was maintained and same amount of sludge was fed and wasted daily from the digesters. The daily biogas production by the anaerobic
digesters was measured by using RebelTM wet-tip gas flow meters (invented by Dr. R.E. Speece and manufactured by Rebel Point Wet TipGas Meter Co., Nashville, TN, USA). The Rebel wet-tip gas meter ‘tips’ when a known volume of gas passes through. Each tip is recorded by an internal magnetic counter. To calculate the total volume, total number of recorded tip counts is multiplied by the volume per tip.

Analytical Study

Total solids (TS) and volatile solids (VS) on the samples (feed, anaerobic effluent and aerobic effluent) were measured twice a week according to Standards Methods for the Examination of Water and Wastewater (APHA, 1999). Volatile solids destruction was determined by the formula (VS_{initial}-VS_{final})/VS_{initial}. The pH was measured on the fresh samples daily by pH probe. Total Kjeldahl Nitrogen (TKN) and total ammonia were measured by methods described in Standards Methods (APHA, 1999). For total COD, samples were acidified using concentrated H_{2}SO_{4} to lower the pH to less than 2 before measuring COD. Acidifying the sample fixes the carbon. COD measurements were conducted by the closed reflux method (APHA 1995). For volatile fatty acids (VFA), cations, anions, proteins and polysaccharides, samples were centrifuged at 10,000 for 30 min and filtered through 1.5 um pore size cellulose filters (Type 935-AH, Whatman), followed by filtration through 0.45 um microfibre filters (nitrocellulose disc filters, Fischer Scientific). The filtered samples were kept frozen prior to analyses, so as to inhibit any biological activity affecting the VFA concentrations. When analyzing the VFA, the frozen samples were thawed and acidified using phosphoric acid.

Solution cations and anions were analyzed using a Dionex DX-120 Ion Chromatograph (IC). Ions were separated and eluted with AutoSuppression technology and a PC with Peaknet Software was used for integration. The gas volume was measured daily and a Shimadzu GC14-A gas Chromatograph (Shimadzu Scientific Instruments, Columbia, MD) with a thermal conductivity detector (TCD) was used to measure methane and carbon-dioxide content in the biogas. The carrier gas used in the GC14-A was Helium with flow of 17 mL/min.
VFA was measured using a Shimadzu GC-14A Gas Chromatograph with Nukol Column and flame ionization detector (FID). The carrier gas used was Helium with flow rate of 17 mL/min and gas pressure (at the tank) of 25 psi. Other gases and their flow rates were: Nitrogen (13 mL/min), Hydrogen (45 mL/min) and Air (450 mL/min). The VFA analyses were done by a Shimadzu CR501 Chromatopak computer integrator. The VFA analyzed were acetic acid, butyric acid, isobutyric acid, heptanoic acid, propionic acid, valeric acid, isovaleric acid and caprionic acid. Standards for the measurement were supplied from Supelco. Dissolved Oxygen in the aerobic digesters was measured by DO probe. An ORP probe (Model 96-78-BN) was used to measure ORP of the aerobic digesters.

RESULTS AND DISCUSSION

Digesters Performance

Lab-scale anaerobic and aerobic digestions of sludge were operated to determine the performance of anaerobic digestion followed by aerobic digestion. The analyses were performed after determination of steady-state conditions and were evaluated by monitoring pH, biogas production and solids removal. Different performance parameters such as volatile solids destruction, COD removal, nitrogen removal, biogas production and VFA production and destruction were measured. All the digesters performed well during the steady-state phases and the analyses are presented below.

pH

The pH is considered as one of the factors that influences the operation and performance of anaerobic and aerobic digesters (Grady et al., 1999). Grady et al. (1999) has reported that the optimum pH of an anaerobic digestion is between 6.8 and 7.4. At a pH lower than 6.3 and higher than 7.8, the methanogenesis rate decreases (Lay et al., 1997). Lay et al. (1997) has also proposed that lower pH inhibits methanogens while a higher pH increases
the concentration of unionized ammonia, causing toxicity in the reactor. When methanogenesis is decreased, organic acids accumulate in the digester, decreasing pH and causing failure of the system.

The pH of the anaerobic digesters through a period of steady state is given in Figure 2.5. It shows that the anaerobic digesters operated within the range needed for proper solids destruction, with the pH varying between 7.2 and 7.8. At times just after starting feeding, the pH of the system varied and sometimes decreased below 6.5. The pH was adjusted by adding sodium bicarbonate in the digester, after which it became steady.

In an aerobic system, nitrification and denitrification tends to change the pH of the system. Alkalinity with its buffering capacity affects the tendency of pH changes in the system. Metcalf and Eddy (1991) has given optimum pH ranges for both nitrifying and denitrifying activities: 7.5 to 8.5 (no activity below 6 - 6.5 and above 10) for nitrification activity and 6 – 8 (most favorably 7 – 8) for denitrification activity. Nitrification consumes alkalinity (Grady et al., 1999) and decreases pH; it converts ammonium to nitrite and nitrate in the presence of oxygen and high DO conditions. Denitrification reduces nitrate to nitrogen gas under reducing or low DO conditions; nitrate serves as the terminal electron acceptor. When both nitrification and denitrification are occurring, all the ammonium is converted to nitrogen gas, as given by the equation from Grady et al., (1999): 

\[4C_3H_7O_2N + 23O_2 \rightarrow 20CO_2 + 2N_2 + 14H_2O\]

Alkalinity destruction or pH changes do not occur in these cases and there is no need for pH control since simultaneous nitrification and denitrification occurs. In our study, the pH range for the aerobic reactors in both the An/Aer/An – B and Ana/Aer system shows that the reactors operated within the range for nitrification and denitrification to occur and pH control was not necessary. High pH of an aerobic system can cause ammonia stripping and can also indicate the failure of the system. The aerobic reactors, with pH within range of 6-7.5, were considered stable and working well.
Fig. 2.5. pH of the mesophilic anaerobic digesters through steady state

Fig. 2.6. pH of the digesters through steady state
**Volatile Solids Reduction (VSR)**

In a digestion system incorporating an aerobic stage after anaerobic digestion, large fraction of organic matter is already biodegraded and eliminated before the sludge reaches the aerobic digester. Therefore, the residual organic matter from the anaerobic digester requires a lower oxidation capacity in the aerobic digester for nitrification and further degradation of the residual organics. Studies performed by Park et al. (2006) and Kumar (2006) showed more VS reduction in combined anaerobic/aerobic or aerobic/anaerobic sludge digestion process than for a single digestion process with the same overall detention time. In another study by Parravicini et al. (2006), residual VSS was found to be degraded significantly when anaerobic sludge was further stabilized by aerobic digestion. Later Parravicini et al., (2008), reported that post-aerobic digestion (6d SRT, 36°C) of mesophilic anaerobic digested sludge (30d SRT) reduced the organic solids an additional 16%. These studies show that with anaerobic digestion, some organic fraction in the sludge remains undegraded, which are available for further degradation under aerobic conditions.

The data of VS removal as shown in Figures 2.7 and 2.8 show that An/Aer/An – A and An/Aer/An – B gave about 70% VS removal, compared to 62% for Ana/Aer and 50% for both MAD 50d and 15d SRTs. Compared with single-stage mesophilic anaerobic digestion, the enhancement for VS reduction with combined anaerobic and aerobic digestion (anaerobic digestion followed by aerobic digestion) was about 13%. Both the An/Aer/An – A (wastage from Anaerobic digester) and An/Aer/An – B (wastage from Aerobic digester) had a VS reduction enhancement of almost 20% compared with MAD and 8% compared with only Ana/Aer digestion.
Fig. 2.7. Volatile Solids Reduction (%) of MAD 15d SRT and MAD 20d SRT. The average VSR for both systems is also included.

Fig. 2.8. Volatile Solids Reduction (%) in Ana/Aer, An/Aer/An – A, and An/Aer/An – B through steady-state period. The average VSR for MAD 20d SRT is also included.
**COD Removal**

In a study by Tapana and Pagilla (2000) it was shown that pre-and post-aerobic treatment reduced COD by 56±67% while only 44±60% reduction was obtained in a single mesophilic anaerobic digestion. Kumar (2006) achieved up to 60% to 70% COD removal by sequential anaerobic/aerobic digestion of sludge, at 3 day SRT of aerobic digestion.

The COD removal data show 50% removal for MAD 20d and 15d SRT, while an aerobic digestion following the MAD (each of the options: Ana/Aer, An/Aer/An – A and An/Aer/An – B) show 70% COD removal. So, an additional 20% COD destruction was achieved with an aerobic digestion after anaerobic digestion. Fig. 2.9 and Fig. 2.10 show the COD removal (%) for the options, through a steady-state time period.

![Fig. 2.9. COD removal (%) in MAD 15d SRT and MAD 20d SRT. The average COD removal for both systems is also included.](image-url)
Biogas Production and Composition

The main by-product of anaerobic digestion is biogas, with methane and carbon-dioxide as major components. Other gases, such as nitrogen, constitute smaller fractions. Methane content in the biogas indicates the stability and performance of the digester and it depends on the fraction of organic matter degraded. In a stable condition, a fixed amount of methane per unit of COD or VS fed, is produced (Grady et al., 1999). So, variations in the methane to COD or VS ratio over time can indicate the decreasing performance of the digester. The CH4 and CO2 percentages in the anaerobic digesters are shown in table 2.3; the table shows the systems are stable.
Table 2.3. Gas composition of the Anaerobic Digesters

<table>
<thead>
<tr>
<th>Sample</th>
<th>Methane Content (% by volume)</th>
<th>CO2 Content (% by volume)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MAD (20d SRT)</td>
<td>61 ± 2.1 %</td>
<td>35 ± 1.2 %</td>
</tr>
<tr>
<td>MAD (15d SRT)</td>
<td>57 ± 3.1 %</td>
<td>39 ± 0.4 %</td>
</tr>
<tr>
<td>An/Aer/An - A</td>
<td>64 ± 1.1 %</td>
<td>33 ± 0.7 %</td>
</tr>
</tbody>
</table>

**Nitrogen Removal, ORP and DO**

In an anaerobic sludge digestion, high amount of ammonium nitrogen is produced. Digestion and centrifugation of sludge can release up to 50% of sludge bound nitrogen (Siegrist, 1996) which increases the nitrogen load to the plant when the centrate is recycled. Intermittent aeration of the anaerobically digested sludge can remove some fractions of nitrogen. Nitrification occurs in aerobically while denitrification and methane production occur in an anaerobic stage, with VFAs, protein and polysaccharides serving as carbon sources. Akuna et al., (1994) proposed that recycle-to-influent ratio influenced the nitrogen removal in combined anaerobic-aerobic digestion. In the study, they also found that with increased recycle-to-influent ratio, the methane production rate in the anaerobic digester decreased whereas the nitrogen gas production rate increased. Kumar (2006) studied the combined anaerobic and post-aerobic digestion of sludge with aerobic digestion SRTs at 3, 6 and 9 days. He found that ammonia removal (80%) and TKN removal (more than 50%) were achieved when the SRT of the aerobic digester was increased from 3 to 9 days. Parravicini et al., (2008) found 45% total nitrogen removal with intermittent aeration of the anaerobically digested sludge, with optimum aeration/pause ratio.

Nitrogen analyses were performed on the anaerobic and aerobic sludge in all the three phases. TKN and ammonia measurement throughout a steady-state period is shown in Figures 2.11 - 2.15.
Fig. 2.11. Nitrogen (TKN and NH$_3$) present in the influent and effluent in MAD 15d SRT.

Fig. 2.12. Nitrogen (TKN and NH$_3$) present in the influent and effluent in MAD 20d SRT.
Fig. 2.13. Nitrogen (TKN and NH₃) present in the influent and effluent in Ana/Aer.

Fig. 2.14. Nitrogen (TKN and NH₃) present in the influent and effluent in An/Aer/An - A
Fig. 2.15. Nitrogen (TKN and NH₃) present in the influent and effluent in An/Aer/An - B

Table 2.4. TKN and NH₃ in and out of the Digestion processes

<table>
<thead>
<tr>
<th>Digestion Process</th>
<th>TKN in (mg/d / L feed)</th>
<th>TKN out (mg/d / L feed)</th>
<th>NH₃ in (mg/d / L feed)</th>
<th>NH₃ out (mg/d / L feed)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MAD 20d SRT</td>
<td>2996.5</td>
<td>2839.5</td>
<td>420.5</td>
<td>1426.8</td>
</tr>
<tr>
<td>MAD 15d SRT</td>
<td>3744.6</td>
<td>3284.9</td>
<td>914.7</td>
<td>1762.6</td>
</tr>
<tr>
<td>Ana/Aer</td>
<td>3744.6</td>
<td>1339.4</td>
<td>914.7</td>
<td>282.3</td>
</tr>
<tr>
<td>An/Aer/An – A</td>
<td>2780.2</td>
<td>1558.3</td>
<td>700.0</td>
<td>778.7</td>
</tr>
<tr>
<td>An/Aer/An - B</td>
<td>3152.2</td>
<td>946.4</td>
<td>440.4</td>
<td>417.2</td>
</tr>
</tbody>
</table>
Fig. 2.16. TKN removal (%) of Ana/Aer, An/Aer/An – A and An/Aer/An – B. An/Aer/An – B shows higher percentage (70%) of TKN removal

The table 2.4 shows that the TKN in the An/Aer/An – B effluent is lower than the TKN in Ana/Aer or An/Aer/An – A. The variance in influent TKN and NH₃ amount can be attributed to the change in feed characteristics supplied from DCWASA. Figure 2.16 shows TKN removal in the sequential anaerobic/aerobic digestion and anaerobic/aerobic/anaerobic (with wastage from anaerobic or aerobic reactor) digestion. It shows that when aerobic digestion is used after anaerobic digestion, the TKN removal enhancement can be up to 64% while An/Ana/An – A (wastage from anaerobic digester) has 44% TKN removal. Higher TKN removal, of almost 70%, can be obtained from An/Aer/An – B (wastage from aerobic digester).

Nitrate and nitrite was also measured in the aerobic reactors of Ana/Aer, An/Aer/An – A and An/Aer/An – B. It showed no detectable amount of nitrate or nitrite, measured at the end of the 24hr cycle. This can be explained by simultaneous nitrification and denitrification occurring at the end of the cycle in the aerobic digesters. Measurement of ORP was performed on the aerobic digesters to analyze the nitrification and denitrification processes. In one study by Sekine et al. (1985), nitrification rate and ORP
have been found to be linearly related and ORP can be used as a control parameter for nitrification and oxygen demand. Kim and Hao (2001) found that the end of nitrification and denitrification can be indicated by points in pH and ORP profiles respectively. The ORP measurement shows oxidizing and reducing conditions occurring in the aerobic digester, through the 24hr cycle. It shows that immediately before feeding the digester with raw sludge, oxidizing conditions were present. After addition of sludge, conditions gradually became reducing. Bernet et al. (1996) has showed that the increase in redox potential during the reducing environment is due to the denitrifying activity. As shown in Figure 2.17 and Figure 2.18, the ORP profiles of the aerobic digesters, over the last 6 hours of the 24 hr cycle, the conditions again became oxidizing. This interchanging of oxidizing and reducing conditions produced both nitrification and denitrification in the aerobic digesters. With low or no detection of nitrite and nitrate in the digester, removal of nitrogen seen in the aerobic digester compared with the anaerobic digester (in aerobic stage of Ana/Aer system and An/Aer/An – B, as shown in the figures) suggests the occurrence of nitrification and denitrification.

Figure 2.19 shows the dissolved oxygen (DO) concentration in the aerobic digesters. The figures show that the DO decreases gradually after feeding and it increases at the end of the 24hr cycle. The anaerobic feed has high oxygen demand which is supplied by the dissolved oxygen present in aerobic digester. This causes the DO to decrease.
Fig. 2.17. Oxygen Reduction Potential (mV) in the aerobic reactor of the system Ana/Aer through the 24hr cycle

Fig. 2.18. Oxygen Reduction Potential (ORP) (mV) in the aerobic reactor of the system An/Aer/An - B through the 24hr cycle
Volatile Fatty Acid production

In an anaerobic digestion, VFA are formed as intermediates and increased VFA accumulation in the digester indicates its decreased stability (Grady et al., 1999). Methanogens use acetic acid and H₂ to form CH₄. When VFA production rate is greater than its use by the methanogens, VFA accumulation occurs. This is indicated decrease in pH and alkalinity and this in turn inhibits activities of methanogens. Thus, VFA analyses are considered important for monitoring the performance of an anaerobic digester.

VFAs in both the anaerobic, as well as aerobic digesters, of all the stages were measured. The average steady-state VFA concentrations in the digesters are given in table 2.5. Figures 2.20 and 2.21 give the acetic and propionic acid concentrations through steady-state. In all the digesters, Acetic acid was present in greater amount than other acids. The observed increase in acetate concentration with decreasing propionate concentrations indicates that degradation of propionic and other higher molecular weight VFA results in
simultaneous production of acetic acid. The concentrations of VFA fractions in the anaerobic digesters showed that the systems were stable and the hydrolysis and acidification process occurred in proper rate. In aerobic digesters of Ana/Aer and An/Aer/An – B, the VFA from the anaerobic effluent were removed. Thus, the data show that in the aerobic digestion process, the short chain VFAs were used as carbon source for denitrification. The An/Aer/An – B showed the lowest total VFA concentrations.

Figure 2.20. Comparison of acetic acid concentrations in the digesters, through steady-state.

Figure 2.21. Comparison of propionic acid concentrations in the digesters, through steady-state.

69
Table 2.5. Average steady-state VFA concentrations in the digesters. The VFA concentrations are given in mg/L as HAc.

<table>
<thead>
<tr>
<th>VFA</th>
<th>MAD 20d</th>
<th>MAD 15d</th>
<th>Ana/Aer</th>
<th>An/Aer/An-A</th>
<th>An/Aer/An-B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetic</td>
<td>201.29</td>
<td>175.80</td>
<td>25.74</td>
<td>276.94</td>
<td>22.23</td>
</tr>
<tr>
<td>Butyric</td>
<td>0.94</td>
<td>1.01</td>
<td>0.41</td>
<td>6.43</td>
<td>0.35</td>
</tr>
<tr>
<td>Propionic</td>
<td>73.35</td>
<td>40.08</td>
<td>0.55</td>
<td>50.79</td>
<td>0.59</td>
</tr>
<tr>
<td>Isobutyric</td>
<td>5.71</td>
<td>9.20</td>
<td>0.78</td>
<td>14.71</td>
<td>5.53</td>
</tr>
<tr>
<td>Hexanoic</td>
<td>0.20</td>
<td>0.70</td>
<td>5.87</td>
<td>0.26</td>
<td>0.83</td>
</tr>
<tr>
<td>Isovaleric</td>
<td>0.12</td>
<td>0.30</td>
<td>0.62</td>
<td>0.73</td>
<td>0.28</td>
</tr>
<tr>
<td>Valeric</td>
<td>0.55</td>
<td>0.20</td>
<td>0.53</td>
<td>0.48</td>
<td>1.06</td>
</tr>
<tr>
<td>Isocaproic</td>
<td>0.42</td>
<td>2.64</td>
<td>1.55</td>
<td>0.16</td>
<td>0.30</td>
</tr>
<tr>
<td>Heptanoic</td>
<td>BDL</td>
<td>2.50</td>
<td>BDL</td>
<td>BDL</td>
<td>0.12</td>
</tr>
<tr>
<td>Total VFA</td>
<td>282.58</td>
<td>229.92</td>
<td>36.04</td>
<td>350.50</td>
<td>31.18</td>
</tr>
</tbody>
</table>

CONCLUSIONS

This study was performed with an objective to investigate the performance of anaerobic-aerobic-anaerobic digestion of sludge and compare it to single conventional digestion and sequential anaerobic-aerobic digestion of sludge. To determine the quality of effluent characteristics, the anaerobic/aerobic/anaerobic digestion of sludge was studied with two options: wastage from anaerobic digester and wastage from aerobic digester. Different operational parameters, such as COD removal, VS destruction, biogas production and Nitrogen removal were studied and the results were compared among different processes.

From the study, it was found that An/Aer/An – B (wastage from aerobic reactor) provided better effluent characteristics than An/Aer/An – A (wastage from anaerobic reactor), Ana/Aer or conventional MAD.
Both An/Aer/Ana – A and An/Aer/An – B give 70% VS removal, compared to 50% with single MAD and 62% with only Ana/Aer.

COD removal for both An/Aer/An – A and An/Aer/An – B was 70%, while it was 50% and 66% for single MAD and Ana/Aer, respectively.

Nitrogen removal data provided much insight into the comparison of An/Aer/An – A and An/Aer/An – B. It showed that with An/Aer/An – A (wastage from anaerobic reactor), 43% TKN removal was obtained, while it was almost 70% for An/Aer/An – B (wastage from aerobic reactor). The An/Aer/An – B system was better than Ana/Aer in TKN removal. Ana/Aer provided about 64% TKN removal.

The study of effluent characteristics such as VS reduction, COD removal and nitrogen removal, the An/Aer/An system with wastage from the aerobic side proved best.

REFERENCES


Chapter 3

Dewatering characteristics and Odor Reduction in Anaerobic/Aerobic/Anaerobic Digestion of Sludge

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Chapter 3

Dewatering characteristics and Odor Reduction in Anaerobic/Aerobic/Anaerobic Digestion of Sludge

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Abstract

Anaerobic and aerobic digestion of sludge brings about changes in polymer dose conditioning and dewatering characteristics of the digested effluent. These changes are thought to be correlated with the biopolymer, protein and polysaccharides that are released into the solution during digestion. Previous research has shown that the biodegradation of the proteins in biosolids of the digested sludge produces odor compounds or Volatile Organic Sulfur Compounds (VOSCs), which are the major compounds linked with odors produced from the biosolid cake. The objectives of this study were to investigate the effectiveness of digestion processes on the effluent quality, based on its biopolymer release and availability, dewatering ability, cake solids concentration and odor generation potential. Three different lab-scale phases were operated: MAD (with 15d SRT and 20d SRT), sequential Anaerobic/Aerobic (Ana/Aer) and Anaerobic/Aerobic/Anaerobic digestion (An/Aer/An; with An/Aer/An – A, wastage from Anaerobic reactor and An/Aer/An – B, wastage from Aerobic reactor). The study demonstrated that both An/Aer/An – A and – B digested effluent had better dewatering characteristics with lower optimum polymer dose, low CST, increased cake solid concentration and reduced odor production (low peaks and odor generation lasting for a shorter duration) from the biosolids, than Ana/Aer or MAD (15d or 20d SRT).

Keywords: Mesophilic Anaerobic Digestion, Aerobic digestion, Dewatering, Capillary suction time (CST), Cake solid concentration, Biosolids, Odor generation, volatile organic sulfur compounds (VOSCs)
INTRODUCTION

Land application of biosolids is becoming an acceptable practice but before its disposal or use, it has to meet certain criteria under 40 CFR 503 Part (b) on sludge reuse and disposal regulations (EPA, 1992) that regulates pathogen reduction and pathogen vector attraction reduction. Applying biosolids in land has benefits; it is economical and it increases soil productivity and recycles resources. However, with use of biosolids in land, odor emissions from the biosolids have become a major environmental and health issue for general public and wastewater utilities. To overcome all the concerns regarding biosolids and odor, comprehensive research have been done on odor generation, organisms and mechanisms responsible for it and ways to minimize or manage odor from the biosolids.

Anaerobically digested sludge produces unacceptable odor (Murthy et al., 2002) and the compounds associated with the generation of odor are the volatile organic sulfur compounds (VOSCs) (Forbes et al., 2003; Higgins et al., 2002). The VOSCs in digested sludge are emitted when only methanogens in the cake solid are inhibited, for e.g. by the use of toxic materials, high-sheared dewatering or increased polymer dose (Iranpour et al., 2003; Zitomer and Speece, 1995). Forbes et al. (2003) and Higgins et al. (2002) have also shown that when methanogens are not disturbed in their activity, the VOSCs in cake solid first increase in concentration, and then decrease gradually to below detection limits. Novak et al. (2006) proposed that Methanethiol (MT) and dimethyl sulfide (DMS) were mainly associated with odor in dewatered sludge cakes. Results from studies have shown that adding a methanogenic inhibitor, BESA, to cake solid increases odor generation (Chen et al., 2005; Higgins et al., 2006) which suggests that degradation of VOSCs is carried by methanogens.

Dewatering of sludge before it is disposed or used in land minimizes sludge volume which ultimately reduces cost in handling and transporting of the waste. Higgins et al. (2006) and Winter and Duckham (2000) have found that liquid anaerobic digested sludges have lower volatile odor compounds than dewatered, stored sludge. Dewatering
process generates shear which breaks up the floc releasing more EPS-bound protein (Higgins et al., 2002; Murthy et al., 2003). The shear causes cell lysis and cell damage in the methanogens, thus rendering them unable to degrade the odor compounds and increasing the VOSCs. Adding polymer to the sludge disintegrates the EPS-bound protein from the floc and makes it bioavailable. Muller et al. (2004), found no presence of odor compounds in the sludge cake which was sheared with no polymer addition. Odor generation from cake solid is also found to be associated with Fe content of the sludge (Verma, 2005) and Higgins et al. (2008) has proposed that adding iron in sludge can reduce odor production from the sludge cake.

Novak et al. (2004) studied variety of digested sludge and its dewatering characteristics. They found that VS destruction decreases dewatering ability of sludge, increasing the polymer dose requirement. This poor dewaterability occurs as proteins and polysaccharides are released into the solution during digestion (Novak et al., 2003). Novak et al. (2001) have proposed that during anaerobic digestion, more protein is released while during aerobic digestion, polysaccharides are higher in solution. Park et al. (2006), in a study on waste activated sludges, proposed that with single digestion (anaerobic or aerobic), some degradable organic matter remains in the floc. The remaining organic matter is further degraded when it is digested further. So a combined anaerobic-aerobic digestion will have more VS destruction. Park et al. (2006) found that VS destruction in anaerobic digestion was caused by protein degradation while divalent cations, Ca and Mg, influenced aerobic digestion. Tapana and Pagilla (2000) showed that pre- and post-aerobic treatment of mesophilic anaerobic sludge produced digested sludge with better dewaterability characteristics than the single mesophilic anaerobic sludge. Subramaniam (2005) found that sequential anaerobic/aerobic produced sludge with lower CST, lower polymer dose and lower bound-water content than single anaerobically digested sludge. Kumar (2006) studied sequential anaerobic-aerobic digestion of sludge. He found that increase in anaerobic digestion SRT decreased odor generation. Similar results have been obtained by Verma (2005). Kumar (2006) also observed that aerating the digested sludge produced biosolids with less odor generation with bettering dewatering properties.
RESEARCH OBJECTIVES

Review of literature showed that some research has been done on the use of sequential anaerobic-aerobic digestion of sludge for odor and biosolid management. Limited information was available on anaerobic-aerobic-anaerobic digestion of sludge. This study was undertaken to determine the effectiveness of anaerobic-aerobic-anaerobic digestion of sludge in comparison to anaerobic-aerobic and conventional mesophilic anaerobic digestion. The anaerobic/aerobic/anaerobic digestion of sludge was studied with two options: effluent from anaerobic digester, and, effluent from aerobic digester. The hypothesis in this research was that anaerobic/aerobic/anaerobic digestion of sludge could produce effluent with better characteristics, in terms of less odor generation and better dewatering.

The objectives of the study were:

- To study the effect of anaerobic-aerobic-anaerobic digestion on the dewatering properties of the sludge.
- To study the effect of anaerobic-aerobic-anaerobic digestion on the odor generation potential of the resulting biosolid.
- To determine the biopolymer and cation content of the digested biosolids.
METHODS AND MATERIALS

Experimental Approach
The study was divided into three phases. The reactor and process description drawings are presented in the following sections.

I. Mesophilic Anaerobic Digestion of Sludge

The mesophilic anaerobic digester (35°C) was operated as completely mixed reactor (25 L nominal volume and 20 L active volume). It served as process control. It is termed as MAD 20d SRT.

Fig: 3.1. Digestion configuration of Mesophilic Anaerobic Digestion (MAD) of sludge with arrows representing direction of mass flow (feed and waste).
II. Sequential Anaerobic / Aerobic Digestion

The mesophilic anaerobic digester (35°C) was operated as completely mixed reactor (25 L nominal volume and 15 L active volume). The waste sludge from the anaerobic digester was then digested aerobically. The aerobic digester was 9L nominal volume with 5L active volume. The system is termed as Ana/Aer. The mesophilic digester in the system is termed as MAD 15d SRT.

Fig. 3.2. Digestion configuration of Sequential Ana/Aer digestion of sludge. The mass flow through the digesters is given by the arrows.

III. Anaerobic/Aerobic/Aerobic Digestion of Sludge

The mesophilic anaerobic digester (35°C) was operated as completely mixed reactor (35 L nominal volume and 30 L active volume). The waste sludge from the anaerobic digester was digested aerobically, then aerobic sludge was centrifuged, the centrate was
thrown while the pellet was re-suspended and mixed with raw sludge. The mixture was then fed to the anaerobic digester. The aerobic digester was 9L nominal volume with 5L active volume. This scheme was studied with two options:

- Option A: Waste from Anaerobic Digester (termed as An/Aer/An – A)
- Option B: Waste from Aerobic Digester (termed as An/Aer/An – B)

**Option A: Waste from Anaerobic Digester**

![Diagram of Anaerobic/Aerobic Digestion Process]

Fig. 3.3. Digestion configuration of An/Aer/An – A (Wastage from Anaerobic Digester), with arrows showing the direction of mass flow through the anaerobic and aerobic digester)
Option B: Waste from Aerobic Digester

The phases were named as shown in table 2.1. The SRTs for all the digesters for all the phases were also calculated and is provided in table 2.2.

Fig. 3.4. Digestion configuration of An/Aer/An – B (Wastage from aerobic digester), with arrows representing the mass flow through the anaerobic and aerobic digester.
Table 3.1. Names of digesters used in the study

<table>
<thead>
<tr>
<th>Schemes</th>
<th>Acronyms</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional Mesophilic Anaerobic Digestion (served as process Control)</td>
<td>MAD (MAD 15d SRT and MAD 20d SRT)</td>
</tr>
<tr>
<td>Sequential Anaerobic-Aerobic Digestion</td>
<td>Ana/Aer</td>
</tr>
<tr>
<td>Anaerobic-Aerobic-Anaerobic Digestion</td>
<td>Option A: Anaerobic Waste</td>
</tr>
<tr>
<td></td>
<td>An/Aer/An - A</td>
</tr>
</tbody>
</table>

**SRTs of the System:**

The SRTs for the conventional and anaerobic/aerobic reactors was based on the hydraulic detention time. For the recycle streams, the overall system SRT was calculated based on the solids in the system divided by the wastage rate. The values are shown in Table 3.2.

Table 3.2. SRTs of the anaerobic and aerobic digesters in the systems.

<table>
<thead>
<tr>
<th>System</th>
<th>System SRT</th>
<th>Anaerobic SRT</th>
<th>Aerobic SRT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional MAD</td>
<td>20 d</td>
<td>20 d</td>
<td>----</td>
</tr>
<tr>
<td>Sequential Ana/Aer</td>
<td>20 d</td>
<td>15 d</td>
<td>5 d</td>
</tr>
<tr>
<td>Ana/Aer/Ana Anaerobic Waste</td>
<td>35 d</td>
<td>15 d</td>
<td>5 d</td>
</tr>
<tr>
<td>Ana/Aer/Ana Aerobic Waste</td>
<td>35 d</td>
<td>15 d</td>
<td>2.5 d</td>
</tr>
</tbody>
</table>

All of the digesters were kept in a constant temperature room to maintain the temperature. Plastic, egg-shaped fermenters supplied by Hobby Beverage Equipment Company, were used as anaerobic digesters. To maintain constant temperature, a stainless steel thermometer was placed at the side of the digester. For aerobic digester, 9 L glass digesters (Fisher Scientific) were used. Bubble diffusers were used for maximum oxygen
transfer and a compressor was used to supply oxygen. The aeration cycle in the aerobic reactors was 24 hr with continuous feed and wasting. Dissolved oxygen (measured before feeding) in the reactor was kept within 2.5 - 3.0 ppm. Distilled water was added each day to counter any water loss due to evaporation in the aerobic reactor before wasting from it.

For mixing of gas in the anaerobic digesters, peristaltic pumps (Cole Parmer 6-600 rpm) were used and the gas was recirculated from the headspace to the bottom of the digesters using Cole Parmer Masterflex Tygon LFL-18 pump tubing. The pumps were operated at 50% of their maximum possible speed. To ensure greater mixing of the digesters before and after feeding, gas recirculation in the digesters were increased by increasing the speed of the pumps to 100% 10 minutes before sampling and also for 10 minutes after feeding.

The anaerobic digesters were seeded with mesophilic anaerobically digested sludge taken from Pepper’s Ferry Regional Wastewater Treatment Facility, Radford, Virginia. No feeding or wastage was done on the seeded anaerobic digesters for one week to ensure that the microbial communities are acclimatized to the digester environment. After a week, feeding and wasting from the reactor was done. The digesters were monitored for steady-state, in terms of biogas production and pH and sampling and analysis were done only after the determination of the steady-state condition.

The feed for the anaerobic digester was a mixture of primary and secondary sludge (gravity thickened sludge and air flotation thickened waste activated sludge), 1:1 by weight. The primary and secondary sludge were supplied weekly by DCWASA Blue Plains Advanced Wastewater Treatment Facility overnight shipment. Total solids percentages of both the sludge were measured and a mixture of 1:1 by weight of the sludges with total solid percentage of 5% was made by dilution. The sludge was blended and was stored in a 4°C room until used. To maintain the SRT of both the anaerobic and aerobic digesters, constant volume was maintained and same amount of sludge was fed and wasted daily from the digesters. The daily biogas production by the anaerobic digesters was measured by using RebelTM wet-tip gas flow meters (invented by Dr. R.E.
Speece and manufactured by Rebel Point Wet Tip Gas Meter Co., Nashville, TN, USA). The Rebel wet-tip gas meter ‘tips’ when a known volume of gas passes through. Each tip is recorded by an internal magnetic counter. To calculate the total volume, total number of recorded tip counts is multiplied by the volume per tip.

**Analytical Method**

**Cations, Proteins and Polysaccharides:**

For soluble cations ($\text{Ca}^{2+}$, $\text{Mg}^{2+}$, $\text{Na}^{+}$, $\text{NH}_4^{+}$ and $\text{K}^+$), protein and polysaccharides, samples were at 10,000 for 30 min and filtered through 1.5 um pore size cellulose filters (Type 935-AH, Whatman) followed by filtration through 0.45 um microfibre filters (nitrocellulose disc filters) (Fischer Scientific). The samples were kept frozen until analyses. The frozen samples were thawed at the time of analysis. Soluble cations and anions were analyzed using a Dionex DX-120 Ion Chromatograph (IC). For anions, the eluent was a mixture of sodium carbonate and sodium bicarbonate with flowrate of 1.5 mL/min. For cations, the eluent was methanesulfonic acid with flowrate of 1 mL/min.

Soluble proteins were measured using the Hartree (1972) modification of the Lowry et al. (1951) method, using bovine serum albumin (BSA) as the standard. Polysaccharides were measured using the Dubois et al. (1956) method, using glucose as the standard.

**CST and Optimum Polymer Dose**

The optimum polymer dose was the dose that had the lowest CST. For CST measurement, Cationic polymer (Clarifloc 3275, 1% w/w) was added to 100mL of digested sludge and sheared in a Waring blender at G of 10,000 (Muller et al., 2004) for 30 seconds. The time to dewater in seconds was then recorded using both Triton 304-M and Triton 165 CST apparatus and a Whatman 17-CHR chromatography paper. The process was repeated for different dose of polymer. The polymer dose corresponding to the lowest CST was the optimum polymer dose for the sludge.
**Odor Analysis**

The lab-scale odor generation from the biosolid cake was studied by simulating the conditions of a full-scale biosolids production, using a high-solid centrifuge-dewatering process. The odor sample preparation consisted following methods:

**Centrifugation and Dewatering**

After the CST determination, 400 mL of the sludge was conditioned using the optimum polymer dose of the cationic polymer. The conditioned sludge, in a 400 ml centrifuge bottle, was centrifuged in Beckman-Coulter Avanti-JE centrifuge for 15 minutes at 10,000 G at 25°C. The pellet was then further dewatered to achieve solid concentration as achieved by high-solid centrifuge system. The further dewatering consisted of pressing the sludge using hydraulic piston press; pressure applied at 39 psi for 10 minutes, using Whatman 41 filter paper as the media. The sludge cakes were then cut into small pieces for efficient gas transfer inside the bottle.

**Cake Solids Concentration**

Total solids and volatile solids in the cake solids were measured by the method described in Standard Methods for the Examination of Water and Wastewater (APHA 1999). The cake solids were then used to make samples for odor analysis.

**Odor bottle preparation and Incubation**

Bottles for odor analyses were made using 5 grams of wet cake solids in each 50 mL glass bottle. Triplicate samples were made for each sample. In a full-scale biosolids storage facility, there is no gas transfer inside it and the system is anaerobic. So to make the bottle anaerobic in the lab-scale study, the bottles were sealed with screw caps and Teflon-lined rubber septa and they were then incubated at a constant temperature (at 25°C) during the whole experiment. For samples where BESA was to be added, 3-4 squirts of 0.127 mM bromoethanesulfonic acid (BESA) was added in the biosolid cake before sealing and incubating the bottle.
Headspace odor measurement

The headspace gas concentrations were measured daily for 2 weeks and once in 2 days in the third week, so as to produce a TVOSC profile for each sample. The gases measured were hydrogen sulfide (H$_2$S), Methanethiol (MT), dimethyl sulfide (DMS) and dimethyl disulfide (DMDS). The odor compounds were measured with HP chemstation integration software. All the sulfur compounds mentioned above were then summed up and reported together as total volatile sulfur compounds (TVSC) in ppmV sulfur per gram of volatile solids of the solid cake. The total volatile organic sulfur compounds (TVOSC) were identified as TVSC without concentrations of H$_2$S as H$_2$S was usually present at concentrations low enough to be contributing to TVSC.

The headspace odor compounds were analyzed by cryotrapping and gas chromatography / mass spectrometry (GC/MS) (GC 5890, MSD 5970), with column of Supelco Equity-5, 30 m x 0.25 mm capillary column, of film thickness of 1.0 μm. 100 μl gas from the incubated bottle was injected into the inlet column with a gastight syringe. Liquid nitrogen was used as a cyrotrap to trap the analytical compounds being injected and obtain narrow peaks with maximum separation of compounds.

RESULTS AND DISCUSSION

Dewatering, CST, Polymer dose requirement and Cake Solids Concentration

Figures 3.5 and 3.6 show the optimum polymer dose and the CST at optimum polymer dose. The Ana/Aer has higher polymer dose requirement and higher CST than conventional single digestion or An/Aer/An – A and An/Aer/An – B. The results show that An/Aer/An – A (wastage from anaerobic digester) has the lowest CST and optimum polymer dose and therefore has the better dewatering characteristics of all the other options. An/Aer/An – B has a lower CST and optimum polymer dose than Ana/Aer. The solids concentration of the dewatered sludge cake was measured. The cake solid concentration results in Figure 3.7 show that An/Aer/An – B (wastage from aerobic
digester) has the highest solids concentration, (almost 29%), higher than that of Ana/Aer and An/Aer/An – A.

![Graph showing CST values for different digestion processes](image)

**Figure 3.5.** Capillary suction time (CST) for the digested sludge. All the digestion processes are included with CST values (sec).

![Graph showing optimum polymer dose](image)

**Figure 3.6.** Optimum Polymer Dose requirement for the digested sludge. All the digestion stages are included.
Figure 3.7. Cake solids concentration of the digested sludge. The values in percentage are also included.

**Cation and Solution Biopolymer (protein and polysaccharides)**

Novak et al., (2003) has proposed that waste activated sludges have two types of biopolymers. In one fraction of biopolymer, calcium and magnesium ions bind polysachharides and proteins while the other fraction has iron and aluminium binding protein, polysachharides and humic acids (Park et al., 2002). In another study by Novak et al. (2003) and Novak and Park (2004), it has been shown that when waste activated sludge is digested anaerobically, floc destruction occurs, iron is reduced and biocolloids are released in solution. The un-degraded fraction of biocolloids in the solution reduces its dewatering capability contributing to an increase in the required polymer dose (Bivins and Novak, 2001). Novak et al., (2003) also found that during aerobic digestion, along with protein and polysaccharides, Ca and Mg were also released (Novak et al., 2003). These researchers have also shown that for anaerobic digestion, the biocolloids that were responsible for polymer conditioning demand were primarily protein while polysaccharides were the primary biocolloids in aerobic digestion. Murthy and Novak (1999) studied the effect of adding divalent cations to the performance of aerobic
digestion of waste activated sludge. They found that a higher Ca and Mg content in the sludge resulted in better effluent quality, in terms of lower polymer dose requirement, better dewatering, better floc, and lower soluble EPS. The divalent cations were involved in bridging proteins and polysaccharides in the floc while monovalent cations were released into the solution along with protein and polysaccharides and were not able to bind the floc. Murthy and Novak (1999) and Sobeck and Higgins (2002) have proposed that sludge with high monovalent cations (mainly sodium) have poor settling and dewatering properties. Higgins and Novak (1997), in their study of activated sludge characteristics, found that monovalent to divalent (M/D) ratio and specific resistance to filtration (SRF) of sludge were positively related.

Cations and solution biopolymers (protein and polysaccharides) were measured in the digested effluent of each stage, as shown in Table 3.3 and Figures 3.8, 3.9 and 3.10. The analyses showed that aerobic digestion of sludge released Ca and Mg and caused accumulation of polysaccharides in the digester. This suggests that the release of Ca, Mg and polysaccharides is correlated and that the post-aerobic digestion degrades an additional amount of solids from the anaerobic effluent. The correlation between the solution biopolymer concentration and optimum polymer dose for the effluent digested with Ana/Aer, An/Aer/An – A and An/Aer/An – B is shown in Figure 3.11. The graph shows that the polymer dose of sludge is influenced by the biopolymer amount present in the solution. Sludge from the mesophilic anaerobic digester (MAD 15d and 20d SRT) showed high amount of protein and a high amount of ammonium ions due to degradation of protein. The biopolymer in the sludge (mainly protein) results in decreased dewatering ability and an increased polymer dose. Compared with a single Ana/Aer system, the extra anaerobic step in An/Aer/An – A and – B reduced polysaccharides. The Ana/Aer system released less protein than the conventional MAD system and the addition of the second anaerobic step - especially with system An/Aer/An – B (discharge from aerobic reactor) - greatly reduced protein. The results also show that for anaerobic digestion, the biopolymer important for affecting the polymer demand is protein, while it is the polysaccharides in the solution for the aerobic digestion process. The An/Aer/An – A and – B system had less biopolymer in the solution than both Ana/Aer and MAD 15d or 20d.
SRT; thus, resulting in improved dewaterability and less polymer demand. An/Aer/An – A and – B both released higher amounts of Ca and Mg and had better dewatering characteristics and lower polymer dose requirement than Ana/Aer. The results show that An/Aer/An – A and – B both have lower M/D ratio than conventional MAD or Ana/Aer and have better settling characteristics.

Table 3.3 – Cation concentration and Monovalent to Divalent ratio of cations.

<table>
<thead>
<tr>
<th></th>
<th>$Ca^{2+}$</th>
<th>$Mg^{2+}$</th>
<th>$K^+$</th>
<th>$Na^+$</th>
<th>$NH_4^{+}$ – $N$</th>
<th>M/D ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed</td>
<td>86.71</td>
<td>54.12</td>
<td>76.77</td>
<td>140.17</td>
<td>701.67</td>
<td>2.54</td>
</tr>
<tr>
<td>MAD 15d SRT</td>
<td>22.06</td>
<td>16.32</td>
<td>162.49</td>
<td>18.85</td>
<td>370.84</td>
<td>14.39</td>
</tr>
<tr>
<td>MAD 20d SRT</td>
<td>35.52</td>
<td>20.18</td>
<td>112.27</td>
<td>19.25</td>
<td>319.15</td>
<td>8.09</td>
</tr>
<tr>
<td>Ana/Aer</td>
<td>110.69</td>
<td>124.51</td>
<td>96.58</td>
<td>30.92</td>
<td>81.17</td>
<td>0.89</td>
</tr>
<tr>
<td>An/Aer/An - A</td>
<td>186.72</td>
<td>153.59</td>
<td>102.26</td>
<td>20.64</td>
<td>156.47</td>
<td>0.82</td>
</tr>
<tr>
<td>An/Aer/An - B</td>
<td>165.95</td>
<td>150.83</td>
<td>33.46</td>
<td>15.3</td>
<td>70.3</td>
<td>0.38</td>
</tr>
</tbody>
</table>

Figure 3.8. Cation concentrations (mg/L) of feed and effluent of all the stages.
Figure 3.9. Protein and Polysaccharide concentration of effluent of all the stages.

Figure 3.10. Solution Biopolymer concentration of effluent of all the stages.
Figure 3.11. Polymer dose versus solution biopolymer (protein + polysaccharides). The graph shows the data for Ana/Aer, An/Aer/An – A and An/Aer/An – B.

Table 3.4. Summary of VS destruction, solution biopolymer and optimum polymer dose of the effluent of all the stages.

<table>
<thead>
<tr>
<th></th>
<th>VS Destruction (%)</th>
<th>Protein (mg/L)</th>
<th>Polysaccharides (mg/L)</th>
<th>Solution Biopolymer (mg / L)</th>
<th>Optimum polymer dose (mg / L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MAD 15d SRT</td>
<td>49.9</td>
<td>1318.5</td>
<td>362.82</td>
<td>1681.32</td>
<td>1150</td>
</tr>
<tr>
<td>MAD 20d SRT</td>
<td>50</td>
<td>1440.7</td>
<td>467.62</td>
<td>1908.32</td>
<td>1000</td>
</tr>
<tr>
<td>Ana/Aer</td>
<td>62</td>
<td>247.52</td>
<td>1012.14</td>
<td>1259.66</td>
<td>1350</td>
</tr>
<tr>
<td>An/Aer/An – A</td>
<td>69</td>
<td>325.4</td>
<td>444.95</td>
<td>770.35</td>
<td>950</td>
</tr>
<tr>
<td>An/Aer/An – B</td>
<td>70</td>
<td>157.77</td>
<td>757.7</td>
<td>915.47</td>
<td>1200</td>
</tr>
</tbody>
</table>
Odor analyses

Murthy et al., (2002) found that anaerobically digested biosolid followed by centrifugation produces high odor levels and in another study, Forbes et al. (2003) and Higgins et al. (2002) reported VOSCs to be associated with odor from biosolids. Methanogens play an important part in VOSC generation and Iranpour et al. (2003) have proposed that VOSCs from biosolids are generated only when the methanogens in the cake are disturbed. VOSC generation in dewatered sludge cakes is balanced, with its generation and then degradation by methanogens. Methanogens can be inhibited by bromoethanesulfonic acid (BESA) and in a biosolid cake receiving BESA, cycling and degrading of VOSCs is inhibited (Chen et al., 2005 and Higgins et al., 2006). The TVOSC peak measured with addition of BESA can be considered to be the odor generation potential of the cake (Higgins et al., 2006). VOSCs of the biosolids from MAD 20d SRT, MAD 15d SRT, Ana/Aer, An/Aer/An – A and An/Aer/An – B were measured with and without the addition of BESA and are shown in Figures 3.12, 3.13, 3.14, 3.15, 3.16 and 3.17. The odor generation potential of the biosolids was determined from the VOSC measurement. The results show that An/Aer/An – A and – B both have lower TVOSCs generation than conventional MAD and Ana/Aer. The biosolid cake from Ana/Aer effluent produces a higher TVOSC peak than that of An/Aer/An – A and – B. An/Aer/An – A and- B biosolid produces peak at shorter time than Ana/Aer and conventional MAD. The samples with added BESA showed greater TVOSC generation lasting for more days. This can be explained by the fact that methanogens are inhibited by BESA, allowing TVOSC to accumulate. It should also be noted that the TVOSC degraded even with BESA present. This shows that BESA could not absolutely inhibit methanogenic activity.
Figure 3.12. Comparison of total volatile organic sulfur concentration in MAD (15d and 20d SRT); the sludge cakes are measured without BESA and also amended with BESA.

Figure 3.13. Comparison of total volatile organic sulfur concentration in Ana/Aer, An/Aer/An – A, An/Aer/An - B; the sludge cakes are measured both without BESA and amended with BESA.
Figure 3.14. Peak total volatile organic sulfur compounds for MAD (15d and 20d SRT), with and without BESA; sludge cakes with BESA represent the odor potential of the samples. Samples are also measured without BESA.

Figure 3.15. Peak total volatile organic sulfur compounds for Ana/Aer, An/Aer/An – A, and An/Aer/An – B; sludge cakes with BESA represent the odor potential of the samples. Samples are also measured without BESA.
Figure 3.16. Comparison of total volatile organic sulfur compound concentrations, for the samples, with BESA. An/Aer/An – A and – B show lower TVOSC peaks, peaks obtained at shorter duration of incubation time than MAD (15d and 20d SRT) and Ana/Aer.

Figure 3.17. Comparison of total volatile organic sulfur compound concentrations, without BESA, showing the odor potential for all the samples. An/Aer/An – A and – B show lower TVOSC peaks, peaks obtained at shorter duration of incubation time than MAD (15d and 20d SRT) and Ana/Aer.
CONCLUSIONS

The An/Aer/An system was studied to investigate the odor removal and dewatering properties of the resulting biosolids and to compare them to sequential Ana/Aer and conventional mesophilic anaerobic digestion. The study shows that the Anaerobic/Aerobic/Anaerobic (An/Aer/An, with wastage from the aerobic or anaerobic digester) digestion of the sludge can improve the biosolids quality by improving the dewatering capabilities, with lower optimum polymer dose, reduced CST and increased cake solid concentration, and reduce the odor generation from the biosolids.

- The An/Aer/An – A and – B sludge digestion has higher cake solid concentration (%) than Ana/Aer and single MAD. The An/Aer/An – B biosolid cake concentration was observed to have increased, by approximately 30%, compared with MAD 20d SRT. Also, almost 17% increase in solid concentration with respect to Ana/Aer was observed in An/Aer/An – B.

- An/Aer/An (both of the options: A and B) has lower CST than single MAD (both 15d and 20d SRT) and Ana/Aer. Compared to Ana/Aer, a reduction of 52% for An/Aer/An – A and 20% for An/Aer/An – B in polymer dose requirement was observed, indicating improved dewatering characteristics.

- The results show that An/Aer/An (both options: A and B) biosolid has lower odor generation potential than single MAD (15d and 20d SRT) and Ana/Aer. For An/Aer/An – B, the odor generation potential reduced by, 69% compared to 15d SRT, 58% compared to MAD 20d SRT and 70% compared to Ana/Aer.

- Without addition of BESA also, the odor production decreases in the case of An/Aer/An (both options: A and B), when compared with single MAD and Ana/Aer. It was found that An/Aer/An – A generated 78% less odor than that at MAD 15d SRT, 80% less than that at MAD 20d SRT and 85% less odor compared to Ana/Aer.
- Of all the stages, the An/Aer/An – A and – B system, generated odor which peaked at shorter time and lasted for shorter duration of time. The odor generation also lasted for shorter duration time and peaked at shorter time.

REFERENCES


Figure A1. Total Solid Removal (TSR, %) of the digestion stages. An/Aer/An – A and –B have higher TSR than MAD 20d SRT, MAD 15d SRT and Ana/Aer.

Figure A2. COD/VS ratio of the digestion stages.
Figure A3. Amount of TKN (mg/d / L feed) in the effluent of the digestion stages, throughout a steady-state period.

Figure A4. Amount of NH₃ (mg/d / L feed) in the effluent of the digestion stages, throughout a steady-state period.
Figure A5. VOSC production and degradation in MAD 20d SRT biosolid.

Figure A6. VOSC production and degradation in MAD 15d SRT biosolid.
Figure A7. VOSC production and degradation in Ana/Aer.

Figure A8. VOSC production and degradation in An/Aer/An – A
Figure A9. VOSC production and degradation in An/Aer/An – B