

**Comparative Study on Aeration Control by Automatic Oxygen
Supply Device (AOSD) and Fixed ON/OFF Time Systems in
Intermittently Aerated Activated Sludge Process for Domestic
Wastewater Treatment**

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Supply Device (AOSD) and Fixed ON/OFF Time Systems in
Intermittently Aerated Activated Sludge Process for Domestic
Wastewater Treatment**

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Mahmoud BADISS

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List of abbreviations

- AAA: Alternating Aerobic/Anoxic
- A2O/AAO: Anaerobic-Anoxic-Oxic
- AOB: Ammonia Oxidizing Bacteria
- AOSD : Automatic Oxygen Supply Device
- BNR: Biological Nutrients Removal
- BOD₅ : Biochemical Oxygen Demand at 5days
- CBOD : Carbonaceous BOD
- C:N:P : Carbon : Nitrogen : Phosphorus ratio
- COD_{Mn} : Chemical Oxygen Demand using KMnO₄ as oxidizing reagent
- COD_{Cr} : Chemical Oxygen Demand using K₂Cr₂O₇ as oxidizing reagent
- DCO₂:Dissolved CO₂
- DO : Dissolved Oxygen
- EBPR: Enhanced Biological Phosphorus Removal
- ECD : Electron Capture Detector
- F/M: Food to Microorganisms ratio
- FID : Flame Ionization Detector
- GHG: GreenHouse Gas
- HRT : Hydraulic Retention Time
- L: Liter (capacity unit)
- LDO : Luminescent Dissolved Oxygen
- mA : milliAmpere (electric current unit)
- Max : Maximum
- MBR : Membrane BioReactor
- min : minute (time unit)
- Min : Minimum
- Mm³ : Millions of cubic meters (capacity unit)

- mg : milligram (mass unit)
- mL : milliliter (capacity unit)
- MLSS : Mixed Liquor Suspended Solids
- NBOD : Nitrogenous BOD
- NH₄-N : Ammonium Nitrogen
- NO₂-N : Nitrites Nitrogen
- NO₃-N : Nitrates Nitrogen
- NO_x-N : Nitrites and Nitrates Nitrogen
- NOB : Nitritea Oxidizing Bacteria
- NPOC : Non Purgeable Organic Carbon
- ORP : Oxidation-Reduction Potential
- OUR : or OCR Oxygen Uptake (Consumption) Rate
- PTFE : PolyTetraFluoroEthylene
- Q_a : Air flow rate
- Q_i : Influent flow rate
- rpm : rotation per minute
- SBR : Sequential Batch Reactor
- S.D. : Standard Deviation
- SND : Simultaneous Nitrification-Denitrification
- SRT : Sludge Retention Time
- SS : Ssuspended Solids
- TN : Total Nitrogen
- TOC : Total Organic Carbon
- TP : Total Phosphorus
- VFA : Volatile Fat Acids
- vol ppm : parts per million in volume ratio

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Comparative Study on Aeration Control by Automatic Oxygen Supply Device (AOSD) and Fixed On/Off Time Systems in Intermittently Aerated Activated Sludge Process for Domestic Wastewater Treatment

(生活排水処理の間欠ばっ気プロセスにおける AOSD および固定時間給気制御システムの比較研究)

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Abstract

Activated sludge process (ASP) is the most widely used for wastewater treatment. It relies on microorganisms to remove pollutants. Artificial aeration is necessary to favor microbial activity and aeration time control is important not only for optimization of organic pollutants removal but also for energy saving. This study evaluates the performance of Automatic Oxygen Supply Device (AOSD) and fixed ON/OFF time aeration systems in intermittent oxic-anoxic complete mix single reactors.

Wastewater and sludge inoculum from Miho rural village treatment plant, Inashiki, Ibaraki Prefecture (Lat. +36° 0' 18.06", Long.+140° 21' 26.39") were used. Experimental device consisted of 4 Runs, each composed of a 30 L aeration tank and a clarification tank of 7.5 L working volume with stirring systems. The novel AOSD system, based on Dissolved Oxygen (DO) measurement, was designed to calculate optimal aeration time in a 120min intermittent aeration cycle. It was put to test against fixed ON/OFF aeration time sets under conditions of constant influent load, temperature and air flow rate using domestic wastewater normalized to johkasou (onsite domestic wastewater treatment facility developed in Japan) standards. Effluent was weekly collected for 24 hours. Samples were analyzed for main effluent quality parameters. DO and pH of reactors were also measured. Long monitoring periods were conducted with fixed 30/90, 40/80 and 60/60 ON/OFF aeration systems. Each aeration system featured a characteristic DO profile but despite controlled

environment, parameters and influent, seasonal fluctuations were observed in average levels of plateaus and peaks. Changes in influent microbial communities and organic matter composition are believed to be the cause. Good effluent quality in general was obtained with DO ranging from 1 to 2 mg•L⁻¹. Addition of media in a Run also operated with 30/90 setting (30/90M) resulted in improved O₂ dissolution compared to the simple 30/90. AOSD, by adjusting aeration time (ranging between 27 and 34 min with an average of 32 min) reduced the fluctuations and prevented DO peaks indicators of over aeration. Bending points of pH in reactors were correlated to DO pattern and pH average tended to stabilize at lower values with longer aeration time. Peak of pH coinciding with DO peak in 40/80 and 60/60 Runs was explained by decrease of dissolved CO₂ stripped into atmosphere under effect of increasing DO partial pressure. While pH remained close to neutral in the other Runs, it reached as low as 5.8 with 60/60 as a result of long exposition to over aeration. Greenhouse Gases (GHG) emissions were also higher with longer aeration time. AOSD achieved lower GHG emissions. The Biochemical Oxygen Demand (BOD₅) removal was over 90% under all systems except for 30/90. Although aeration time is positively correlated to BOD removal, better efficiency was obtained with shorter oxic period by O₂ recovery from denitrification process. TN removal averaged 84%, less than 75% and 45% with AOSD and 30/90M, 30/90 and 40/80, and 60/60 respectively. The AOSD system provided optimal conditions for balanced nitrification-denitrification and promoted partial simultaneous nitrification-denitrification (SND). On the other hand, average Total Phosphorus (TP) removal ranged between 36% and 46% for all Runs, which was insufficient though higher than conventional ASP. Increasing air flow rate of AOSD during the 2nd experiment period reduced risk of long term bulking occurrence and gave more stability to DO profile while 30/90 unavoidably showed symptoms of hypoxia. As for biota, AOSD favored the buildup of a diversified; more stabilized and well balanced food chain allowing lower sludge production and stable results.

Beside good effluent quality and low GHG emissions, AOSD potentially saved up to 50% energy consumption compared with fixed on/off time under similar conditions. Experiments on variable parameters and conditions with different process configurations are necessary to fully assess AOSD system's capacity to adapt aeration to meet the needs of biota for optimal depollution of wastewater. Air flow rate control might be a useful improvement of AOSD.

KEYWORDS: Intermittent aeration, Automatic System, Activated sludge, Dissolved Oxygen, Nitrogen removal

Chapter 1: Generalities and literature review

1.1 Introduction

Although water covers more than 2/3 of our planet's surface, only 3% of total water volume is fresh and even less (1%) is potentially usable for human activity. In addition to that, dry climates, irregularity of rainfall, storage issues, increasing demand and other factors make fresh water a scarce resource. Wastewater has been treated for environment and health reasons. But development of advanced treatment hinted to possibilities of wastewater reclaim especially for irrigation and industry.

Morocco is a country that suffers from chronic drought. The country, in an effort to cope with the lag in drinking water and sewage networks as well as the too long neglected issue of environment pollution, has put in motion numerous national programs for water and wastewater management. The environment charter highly recommended water resources preservation, energy saving and wastewater reclaim as environment friendly actions.

Choice of advanced wastewater treatment process is of the highest importance in terms of adequacy, performance, operation management, longevity and financing.

In this study, an overview on wastewater treatment methods, present situation of water and wastewater treatment in Morocco, as well as some aeration control systems developed to improve treatment performance and save energy, will be presented. Results of experiments conducted on the novel Automatic Oxygen Supply Device and fix ON/OFF aeration time in a simple lab-scale activated sludge process will be discussed to evaluate performance in terms of sludge and effluent quality, energy saving potential, and greenhouse gases emission. Monitoring of pH and analyses of variations of nutrients concentrations in mixed liquor during one aeration/anoxia cycle

were also undertaken for a better understanding of differences between tested fixed and variable time control systems.

1.2 Generalities about Wastewater and its characteristics

Wastewater may be defined as a combination of liquid or water-carried wastes - from households, institutions, industry, commerce- and ground water, surface water as well as storm water (Metcalf & Eddy, 2003). Wastewaters can be distinguished according to usage of water. **Black water** or **septage** is wastewater from toilets or content of latrines and septic tanks. **Grey water** or **sullage**, on the other hand is wastewater collected from washing (sink, shower, bath, washing machine, etc.). Mixture of grey and black waters is known as **domestic wastewater**. Industrial wastewater may contain highly toxic, corrosive organic and inorganic components. It therefore may require specific treatment before reuse or discharge in sewers. Combined domestic, industrial and storm waters is referred to as **urban (or municipal) wastewater** (Smith, 2005).

Origin of wastewater determines types of pollutants and their concentrations as well as methods necessary for its treatment. However, all pollutants fall into one of the following groups (Wiesmann et al., 2007):

Dissolved substances: this group can be split into organic substances both biodegradable and non-biodegradable types, as well as inorganic materials that include nutrients that can be partly or totally be used by microorganisms, but also metals and heavy metals. Oxidation of dissolved organic substances causes depletion of dissolved O₂. As for inorganic compounds, nitrogen and phosphorus nutrients are the most important pollutants; they may cause eutrophication in receiving waters. Metals and heavy metals are often considered as micronutrients that should be present in very limited concentrations to prevent toxicity.

Colloids: non-settleable particles (organic or inorganic solids and small drops of fats). Oils are usually removed by decantation and emulsified oils are oxidized like the rest of organic matter. Mineral and synthetic oils are considered carcinogenic and highly persistent environment contaminants. Although edible oils are not toxic, they may affect biological wastewater treatment performance (Łobos-Moysa, 2011) and increase fouling in membrane filter systems (Marchese et al., 2000). Solid colloids are mostly a source of turbidity and cause filters fouling.

Suspended solids: this group includes organic residues and microorganisms as well as inorganic particles (sand, minerals, etc.). One of the objectives of wastewater treatment is to reduce number of bacteria, viruses, worm eggs and protozoa that are at the origin of a variety of diseases (Gerardi and Zimmerman, 2005).

Russell (2006) categorizes effects of pollutants on environment into toxicity and Dissolved Oxygen depletion in receiving waters. If interpretation of toxicity tests on organisms often proves to be difficult, DO and its consumption have more reliable evaluation methods. He considers that DO is the most important water quality parameter. Standard method for DO measurement is the Winkler test. However, DO probes, more convenient, have become of common usage. One of the parameters to measure O_2 consumption is **Biochemical Oxygen Demand** and more specifically BOD_5 that requires incubation of analyzed sample for 5 days. It allows estimation of O_2 demand for biological oxidation of organic matter (CBOD) but also for nitrification (NBOD) although the latter usually requires longer period of incubation (BOD_{10} or BOD_{20}). To ensure that only CBOD is measured, it is possible to prevent nitrification by addition of ammonium chloride (NH_4Cl). As many factors affect accuracy of this test, it is considered statistically unreliable at values below $20 \text{ mg}\cdot\text{L}^{-1}$. Long duration of the test also limits its usefulness. Therefore, **Chemical Oxygen Demand** (COD) is generally used as control parameter as results can be obtained in 3 hours. The COD value reflects chemical digestion of organic matter (Russell, 2006). Most frequently used reagent is potassium dichromate ($K_2Cr_2O_7$). It is a strong

oxidative agent and thus decomposes hardly biodegradable substances, hence the higher values of COD compared to BOD₅ (Metcalf & Eddy, 2003; Wiesmann et al., 2007). A similar method using potassium permanganate (KMnO₄), known as Permanganate Index and sometimes referred to as COD_{Mn}, is also used. It is though not recommended as standard method for wastewater (especially industrial wastewater) as complex organic waste is not oxidized by KMnO₄. Dissolved Organic Carbon (DOC) and Total Organic Carbon (TOC) also help determine pollution level of effluent.

Nitrogen, a major element in metabolism for all organisms, exists in wastewater under various forms. **Total Nitrogen** (TN) is an important effluent quality parameter. Organic nitrogen is measured by Kjeldahl method. Total Kjeldahl Nitrogen (TKN) comprises organic and ammonia nitrogen. Inorganic forms of nitrogen -**ammonia** and **nitrite** which can be highly toxic for fish and **nitrate** which favors eutrophication and may cause infant methemoglobinemia- are also very important to measure for they are indicators of biological treatment performance. Phosphorus is another essential element to all organisms and the first responsible of eutrophication. **Total Phosphorus** comprises **orthophosphates**, immediately available for metabolism, **polyphosphates** only available after slow hydrolysis, and **organic phosphates** of very little importance in domestic wastewater (Metcalf & Eddy, 2003). Many other parameters of water quality may be measured such as **Suspended Solids**, indicator of solid-liquid separation and turbidity, **pH** along with temperature, as an important factor affecting reactions and equilibrium of the medium, **odor**, source of discomfort and sometimes indicator of toxic substances inhaled, **metals concentrations**, which may be highly toxic at very low levels or after long period of exposure, **pathogens enumeration**, etc. (Metcalf & Eddy, 2003; Russell, 2006; Smith, 2005; Wiesmann et al., 2007).

Characteristics of wastewater depend on its origin and standards for effluent quality also vary from a country to another. A number of treatment methods were developed to help achieve those standards.

1.3 Wastewater treatment process types

Cheremisinoff (2002) breaks water and wastewater treatment methods into following types:

Chemical Process: based on addition of chemical reagents in order to oxidize pollutants (such as chlorine, iodine, silver and potassium permanganate) or precipitate undesired elements by coagulation and flocculation (like phosphorus removal using multivalent metal ions).

Physical Process: heat, distillation, reverse osmosis, filtration and adsorbing substances (sand, active charcoal...) are the main methods based on physical principle to treat water.

Energy Intensive Process: this type includes UV light and ozone disinfection methods.

If those methods can be used for both drinking water and wastewater treatment, the same author (1996) reports that **Biological Process**, relying on microorganisms to remove organic matter, is the most widely used for wastewater treatment. This process can be achieved by aerobic or by anaerobic oxidation of dissolved and colloidal pollutants to produce energy for the cell, to synthesize cell material and for endogenous respiration. Biological treatment can thus be classified depending on type of oxidation (aerobic/anaerobic) and on whether biomass is fixed on support media (carrier), for wastewater to flow around it, or suspended in mixed liquor.

Population growth of microorganisms in biological process depends on medium characteristics such as pH, temperature, concentration of nutrients and their quality,

availability and strength of oxidizer, presence of toxic agents, accumulation of byproducts, and quality of mixing (Cheremisinoff, 1996).

Wastewater treatment facilities usually include a combination of these process types. First biological processes known and documented date back to middle of the 19th century and first experiment on activated sludge was conducted in 1913 (Wiesmann et al., 2007). They are considered less energy consuming, use less chemicals and produce less solid waste (Metcalf & Eddy, 2003) and are therefore at the core of majority of municipal and some industrial wastewater plants. Activated sludge is the most used process in low to moderately polluted wastewater.

Typical activated sludge wastewater treatment plant process can be divided into three main steps (**Figure 1-1**).

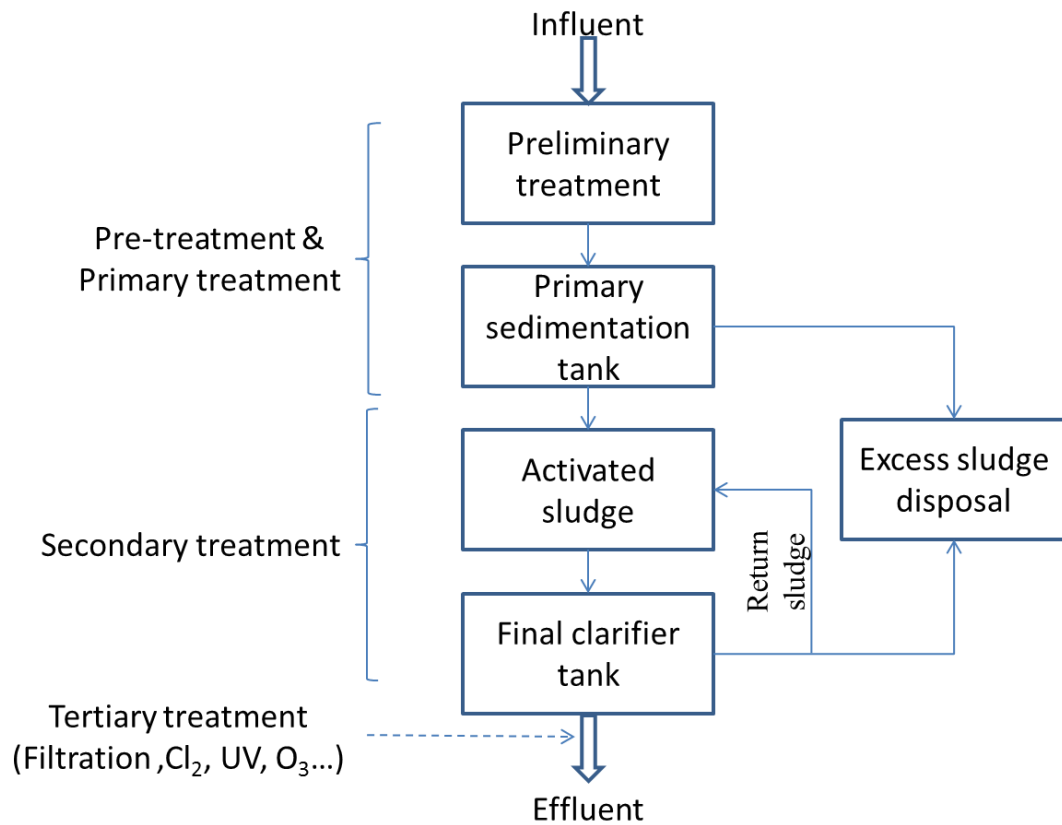


Figure 1-1 Block diagram of typical activated sludge wastewater treatment plant

Pre-treatment and primary treatment: this step generally includes screening to remove big solids, gritting to eliminate sand and stones as well as a primary settling tank in which influent is decanted to separate oils and settleable solids from wastewater.

Secondary treatment: it is the main step of the process. Wastewater containing only dissolved and small suspended particles is mixed with activated sludge in aerator. Many variants exist for this step regarding to aeration system, to whether media are used for sludge fixation or not and characteristics of those media, as well as number of tanks and sequences of aeration-anoxia-anaerobia as well as their distribution in space and time are also characteristics of systems.

Tertiary treatment: this step is usually added if effluent quality does not meet requirements (extremely low levels of pollutants and pathogens targeted or as remediation to a problem when necessary). This optional phase rather uses chemicals, UV and filtration.

According to Smith (2005), activated sludge process has advantages such as compactness (it takes for example seven times less land than a trickling filters system) and less odor but requires skilled operators and operating costs are higher than in some extended treatment processes such as lagooning. Wang et al (2009) actually recommend use of extended land systems when possible due especially to energy cost.

1.4 Examples of activated sludge systems

A standard activated sludge, for BOD removal only, also known as plug flow system is composed of a long and deep rectangular aerated reactor. Part of settled sludge in final clarification tank is recycled by reintroducing it at the head end of aeration tank together with influent (Cheremisinoff, 1996; Wang et al., 2009). In order to adapt this system to (i) influent quality, (ii) volumes to be treated and (iii) effluent

target, a variety of configurations of the reactor has been developed. Some of them are exposed below:

Oxidation ditch: The reactor is built in a ring, oval or horseshoe shape (**Figure 1-2**) with single or multiple channels to increase SRT and thus improve BOD removal (USEPA, 2000).

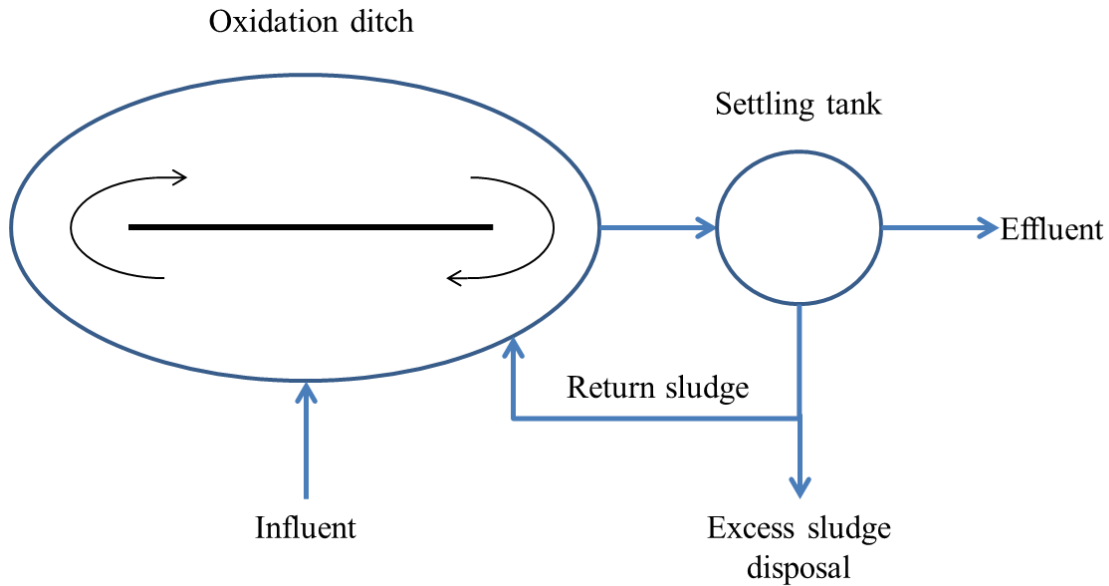


Figure 1-2 Block diagram of oxidation ditch

It is a rather efficient system in terms of energy and sludge production but requires more land than some other systems.

Modified Ludzack-ettinger system: In order to improve nitrogen removal, an anaerobic period is necessary. This system (**Figure 1-3**) proposes to mix Return Sludge and recycled Mixed Liquor with influent upstream of an anoxic compartment that is added before aeration tank (USEPA, 2000).

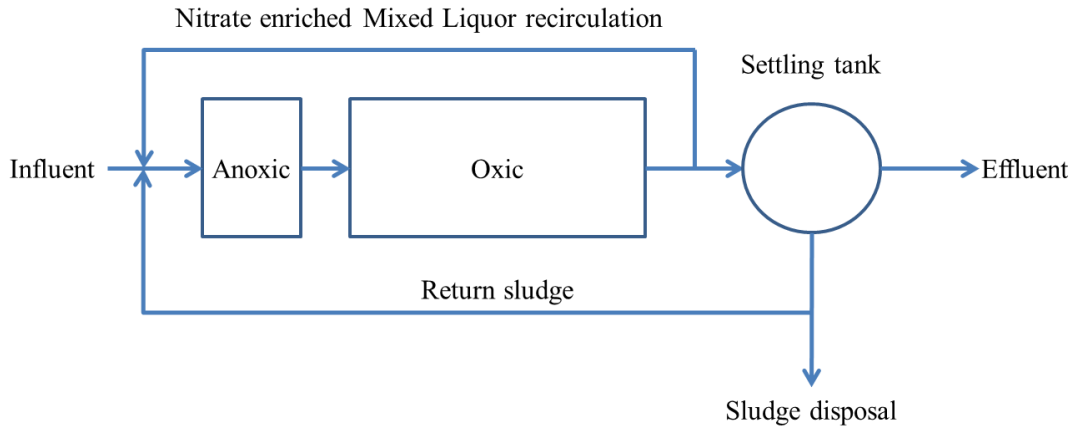


Figure 1-3 Block diagram of modified Ludzack-ettinger process (MLE)

This process improves nitrogen removal by favoring conditions of denitrification.

Anaerobic-Anoxic-Oxic (AAO or A2O) system: As shown in **Figure 1-4**, an anaerobic compartment is used for influent and return sludge mixing. It is an adaptation of Anoxic/Oxic (A/O) and MLE processes (Smith, 2005).

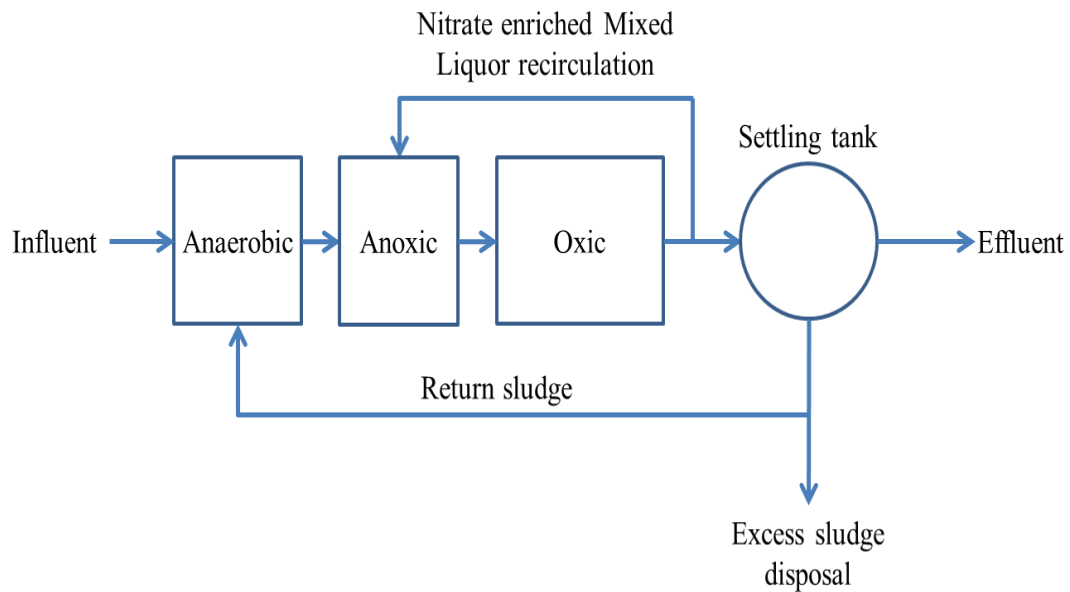


Figure 1-4 Block diagram of Anaerobic-Anoxic-Oxic system (AAO)

Absence of DO in the first compartment favors phosphorus uptake by phosphate-accumulating organisms (PAOs) while the anoxic compartment allows denitrification.

Sequential batch reactor (SBR) system: In this process (**Figure 1-5**), similar to modified Ludzack-ettinger system, all steps occur in a single tank and are therefore separated in time instead of in space (USEPA, 1999a).

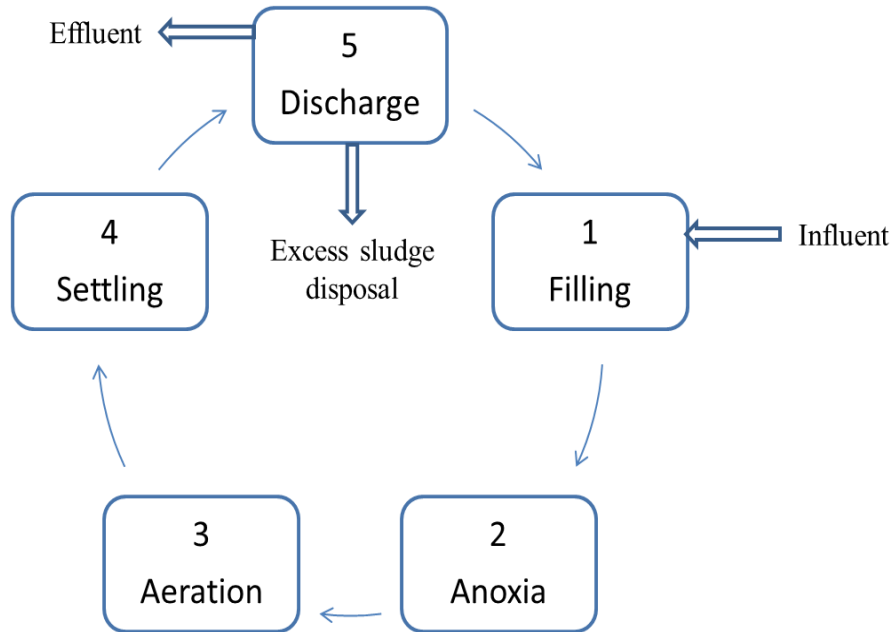


Figure 1-5 Block diagram of sequencing batch reactor system (SBR)

This system offers flexibility in operation and control and potential investment cost saving but is more demanding in automation systems and maintenance in addition to higher risk of sludge washout during draw phase.

Bardenpho system:

Nitrification and BOD removal occur mainly in the bigger oxic compartment. The anoxic zone next to it ensures denitrification of the non-recycled mixed liquor while the last compartment is a re-aeration to freshen effluent (**Figure 1-6**). A 5-stage bardenpho system, by addition of anaerobic compartment at the head end of the tank, improves phosphorus removal (Smith, 2005).

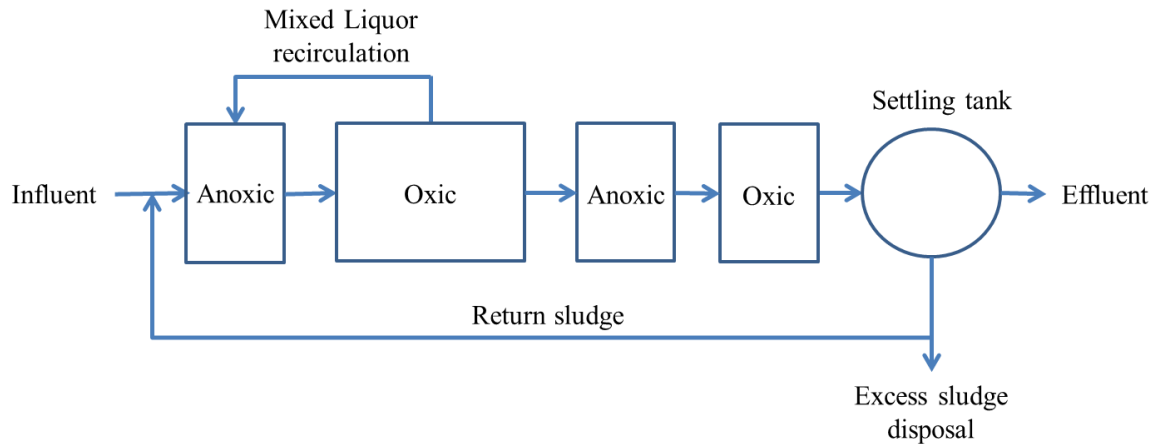


Figure 1-6 Block diagram of bardenpho system

The 5-stage system might be considered as one of the most efficient systems for nutrients removal as it achieves more than 90% removal for both nitrogen and phosphorus.

Membrane bioreactor (MBR) system:

Development of membrane filtration in combination with biological treatment helped overcome many of the issues encountered with conventional systems. Membrane system can be external from bioreactor or simply submerged in aeration tank (**Figure 1-7**). As permeate is free of suspended solids (usually microfiltration is performed in wastewater processes), settling tank is not necessary and neither is filtration of tertiary treatment (USEPA, 2007).

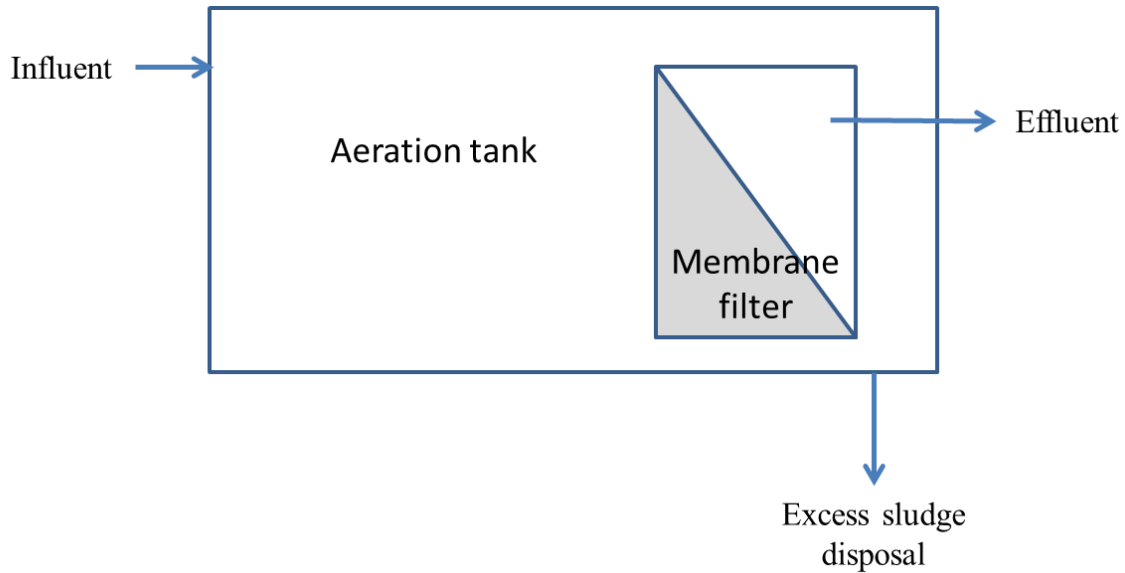


Figure 1-7 Conceptual view of membrane bioreactor (MBR)

This system allows higher sludge concentrations and therefore treatment of higher loads of influent with smaller footprint and produce high effluent quality in return of high operation, maintenance and energy costs.

Numerous wastewater treatment processes exist, with many variants, and are being used depending on wastewater quality, objective from treatment, and affordability. Activated sludge is the most wide-spread and oldest among them. It consists of using microorganisms to oxidize organic matter. The bioreactor is a complex ecosystem in which populations of various species grow and interact under the influence of environment factors. Control of this ecosystem is the key to steady and high performance of wastewater plants. Biological flocs that are more efficient in removing pollutants present the downfall of poor settling and vice versa (Metcalf & Eddy, 2003; Stypka, 1998; Wang et al., 2009; Yiannakopoulou, 2010). It is therefore important to find optimum operation parameters, continually adjust to variations and quickly remedy to occurring problems. In fact, very few plants manage to meet the more and more exacting effluent standards during all year (Richard, 2003). It is even more challenging to do so at low operation cost.

1.5 Aeration control systems

Treatment cost depends on various factors (Vanrolleghem et al., 1996). As far as operation costs are concerned, aeration represents 45 to 60% (Dotro et al., 2011) and in some cases up to 80% of energy consumption which itself usually represents around 30% of total operation cost (Duchène and Cotteux, 2002). A number of actions may be taken to optimize energy consumption in a wastewater plant such as energy audits and installations improvement by novel technology systems (USEPA, 2006). Intermittent aeration; studied since 1976 (Hanhan et al., 2011), is a mean of saving energy by decreasing aeration time. Automatic control of aeration is a further improvement of the system. Technical choices during design of a wastewater treatment plant (dimensions and shape of tanks, mixing system, bubble size...) have a very important effect on treatment performance, but control of aeration undoubtedly improves efficiency. by recovering oxygen from advanced denitrification, and reduces energy cost (Battistoni et al., 2003; Dotro et al., 2011). Therefore, methods and systems of aeration control were investigated by researchers. More recently developed accurate and reliable sensors helped in obtaining better performances (Vanrolleghem et al., 1996).

Automatic devices are based on one or more physico-chemical control parameters and on a specific functioning principle (Dotro et al., 2011). The simplest aeration control system, **the Open Loop control**, consists of setting a clock manually or by a cyclic mode predetermined program to operate blowers at specific moments for specific durations. The second type of systems is known as **Closed Loop control system**. Signal from sensor of a variable parameter is compared to fix values stored in automat and aeration is controlled accordingly. A third type, the **Proportional-Integral-Derivative (PID) control system**, consists of adjusting flow rate (by speed variation of the engine) according to optimal value of a determined parameter (Deronzier et al., 2002; Kuo and Golnaraghi, 2003).

Some aeration control systems belonging to closed loop control method were developed and patented. Tsumura et al. (1994) for example patented a control system based on ORP measurement and used in a double tank intermittent aeration activated sludge process. Saito et al. (1984) proposed a system that controls both aeration and return sludge flow rates using CO₂ and N₂O sensors. The Greenbasstm system (Hazard and Descamps, 2011, 2009; SUEZ, n.d.) claims to be adapted to any aerobic treatment process and relies on NH₄-N and NO₃-N monitoring. More recent researches investigate complex systems including PID and predictive algorithms. Works of Wahab et al. (2009) and Cristea et al. (2011) are examples of this trend.

Functioning principle of the device (monitoring values and bending points of DO, ORP, pH, nutrients, etc.), position of sensors but also diversity of treatment systems (sequence batch reactors, membrane bioreactor, AAO, etc.), size, wastewater characteristics (synthetic, domestic or industrial), main operation conditions (temperature, HRT, MLSS...) and targeted results offer a large number of scenarios and possibilities of development.

The AOSD system is mainly based on DO and temperature measurement to adjust aeration time according to theoretical O₂ transfer and biota requirements in oxygen within a fix 120 min oxic-anoxic cycle in order to provide optimal conditions for advanced nitrogen removal.

1.6 Situation of wastewater treatment in Morocco

Located at the end North-West of the African continent, bounded east by Algeria and to the South by Mauritania, Morocco stretches on an area of more than 700,000 km², great part of which is covered by the Rif, the middle and High Atlas mountain ranges. Altitude in these areas varies between 2000 and 4000 m with Toubkal Mountain culminating at 4.165 m, in the High Atlas. Morocco has over 3500 km of coasts (around 500 km in the North on Mediterranean Sea and approximately 3000 km on Atlantic Ocean on West side).

- **Hydrography**

Arid and semi-arid climates in three-quarters of the territory, limits Morocco's hydrographic network to only a dozen streams of more than 200 km. The Moulouya River, 600 km, is the only one to flow into the Mediterranean in the extreme north-east of Morocco, on the border with Algeria. All other streams of some importance end up into the Atlantic. The Draa is the longest with 1200 km but it is only a "Wadi" on the last two thirds of its course before reaching Tan Tan in Northern Sahara, South-West of Morocco. The Sebou, with its 600km, is one of the most important water resources of the country. Its estuary is situated north of the administrative capital, Rabat, and is strongly subject to industrial and household discharges. Oum Rbiaa, 600 km flows into the Atlantic, South of the economic capital, Casablanca. Key stone of the hydroelectric network and irrigation of Morocco, it is staked of dams (HCP, 2006).

- **Urbanization and water quality**

The Moroccan population has been multiplied by 2.5 in the last 50 years from 12 million in 1960 to 33 million nowadays, despite a slowdown in population growth since the 1970s (**Figure 1-8**). Like in the rest of the world, Morocco is rapidly urbanizing. Urban areas which represented 30% in 1960 concentrate now nearly 60% of the population (HCP, n.d.).

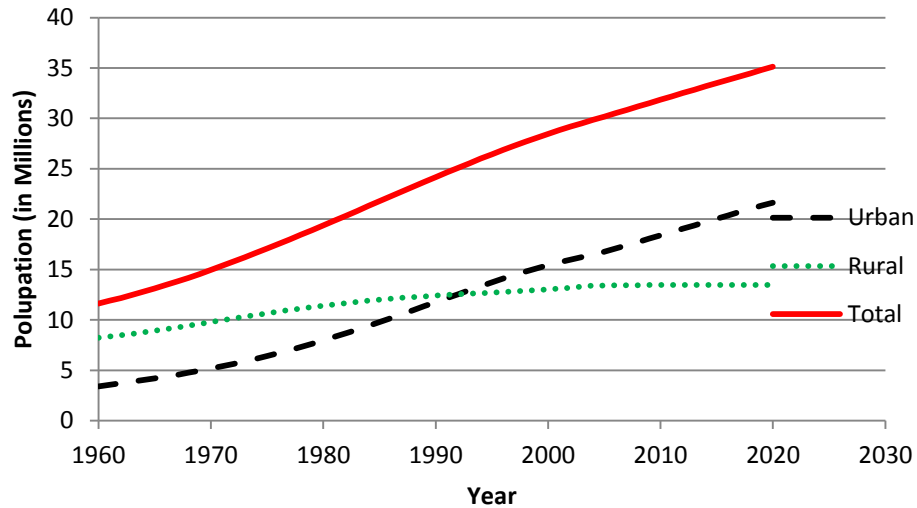


Figure 1-8 Population of Morocco

Urbanization was naturally accompanied by an increase in the needs to drinking water and wastewater treatment. Indeed wastewater volumes estimated to 470 Mm³ in 1994 are projected to attain 900 Mm³ by 2020 (**Figure 1-9**) (Jemali and Kerfati, 2002)

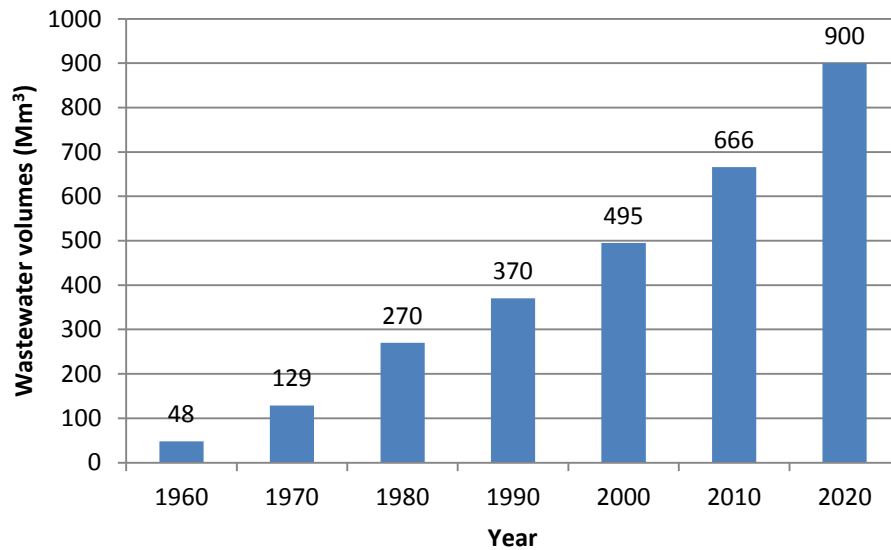


Figure 1-9 Evolution of water usage volumes

If total population growth increased volumes of wastewater, urbanization explained difference in wastewater quality as shown in **Table 1-1** (ONEP and GTZ, 1998)

Table 1-1 Wastewater quality

Parameters	Agglomeration's population (x1000)			National average
	< 20	20 - 100	>100	
BOD₅ (mg•L⁻¹)	400	350	300	350
COD (mg•L⁻¹)	1000	950	850	900
TSS (mg•L⁻¹)	500	400	300	400

The National Program of remediation (Programme National d'Assainissement) put in place in 2005 has been revised in 2008 with the following objectives:

- Achieve a rate of global connection to sewerage in urban areas by 75% in 2016, 80% in 2020 and 100% in 2030;
- Reach a 40% volume of treated wastewater by 2016, 80% in 2020 and 100% in 2030;
- Treat up to the tertiary level wastewater and reclaim 50% of it by 2020.

In 2013, some milestone objectives have been achieved. Now, 90 % of the rural population has access to drinking water, 1/3 of which is by network, the rest is still by faucets at specific points of villages. In urban areas the rate is close to 100%. Connection to the sewerage network is achieved up to 70% in urban areas and 50% in rural.

However, as far as treatment of wastewater itself is concerned, achievements have been far from satisfactory. Only 13% of the 550 Mm³ produced wastewater in 2004 was reported to be treated. Nowadays, while the volume of wastewater has increased by more than 30%, reaching 750 Mm³, the rate of treatment would be, according to a study by the Moroccan Ministry of Environment, less than 10%. The national office for drinking water ONEP (Office National de l'Eau Potable) announces a daily treatment volume of 160,000 m³ Day⁻¹. Cities of Meknes and Marrakech have

wastewater treatment plants with board management. Unfortunately all existing stations suffer from industrial discharges of toxic elements that disrupt their operation.

The situation of coastal areas is particularly alarming due to absence of wastewater treatment plants in most major urban agglomerations. They concentrate more than 60% of the urban population and more than 80% of the industries. The four main agglomerations, from North to South, are Tangier-Tetouan-Fnideq area with 1.5 million inhabitants, the agglomeration of the administrative capital, Rabat, which extends about 60 km with total of about 3 million inhabitants, the agglomeration of economic capital Casablanca, stretching almost uninterruptedly on nearly 75 km and includes an urban population that is close to 4 million and finally Agadir, a large tourist city of about 1 million inhabitants.

All wastewater of these coastal cities, not to mention agglomerations of lesser importance, is released directly into the marine environment. Only pretreatment stations remove coarse waste before wastewater is discharged through pipes, at 2 to 3 km off the shore.

- **Management of water resources**

In regards to freshwater, Morocco lies among the countries that have been relatively successful at managing a resource scarce and poorly distributed in time and space.

However the difficulties persist and can be enumerated as follows.

Surface water volumes may vary by tenfold between drought and rainy years. In addition, half of these volumes are concentrated on only 7% of the territory. Decline in rainfall, coupled with population growth, therefore resulted in a significant deterioration of the ratio of water resources per capita.

Scarcity and irregular surface water availability have led to overexploitation of groundwater, especially from the 1980s. Hydro-agricultural development is operated on insufficiently renewed resources, due to drought. The groundwater level has considerably decreased and numerous sources, and even lakes, have dried up, especially in regions with strong agricultural growth such as Agadir, West of Morocco.

Water resources are, in addition, distributed very unevenly in the territory. Ratio is almost 1 to 20 between the arid areas of southern and Northern wetlands. The latter areas, however, also suffer from water stress due to population growth and concentration of economic activities. By 2030, without significant measures, the deficit should reach 2 billion m³ per year.

As mentioned before, 90% of wastewater is discharged into natural environment (sea or rivers) without prior treatment. In addition to this, uncontrolled landfills contribute to the pollution not only of these rivers but also of the groundwater by infiltration. Anarchic exploitation and use of fertilizers and pesticides worsen the problem.

In 2008 40% of surface waters, on the whole Moroccan territory, could be classified as bad, or very bad.

- **Water and agriculture in Morocco**

The useful agricultural area (UAA) is approximately of 9 million hectares half of which receive an annual rainfall below 400 mm per year. Cereal crops are alternated with fallow, without irrigation on these lands while 1.5 million other hectares are irrigated. Morocco therefore suffers of water shortage for its agriculture. Yet 4 billion m³ per year are wasted in irrigation systems, and discharge of sewage without treatment is not only an ecological threat, but also a loss of potential freshwater. It may be noted that some elements do not plead for the treatment of wastewater for irrigation:

- absence of irrigable land downstream of the spills in several centers, including coastal cities
- high cost of canalization to remote sites
- still satisfactory availability of conventional water at lower cost

By 1994, most of wastewater treatment plants were out of service or were not connected to network for inadequacy to the needs, design issues, or unavailability of operation financing. In 2005, only 32 plants were still operated. Of these facilities, 9 are of activated sludge type. Others are lagooning, infiltration-percolation and trickling filters types. Rural areas not connected to drinking water network are more directly concerned by risks of ground water pollution as drinking water is in general untreated, straight from wells that are often close to potential source of pollution (septic pit, livestock sheds, etc.).

Efforts made to remedy to the present situation are praiseworthy but should be reinforced with long term planning; choice of adequate processes and technologies adapted to the context. Studies should as well be encouraged as very little has been done.

1.7 Objectives of the study

The concern about environment pollution and water resources preservation has increased worldwide. Literature is rich with performance assessment of wastewater treatment processes as well as possible modifications to improve results and adapt to specific conditions. The most limiting factor is probably financial (operation & management and capital cost). Optimal choice requires a careful study of wastewater treatment plant project to ensure sufficient sustainability of the project and low global cost of wastewater treatment. Decentralized treatment is an interesting solution where possible, extended processes are suitable for small collectivities cheaper and require very little maintenance, but in expanding agglomerations advanced treatment

processes should be considered. Aeration control systems are reliable tools for good and stable performance and an effective way of optimizing energy consumption.

This research proposes to test the novel AOSD system and fixed ON/OFF time intermittent aeration during a 120 min constant cycle under controlled conditions on complete mix single tank activated sludge process. Choice of such system was driven by its simplicity and easy convertibility of most activated sludge systems into this configuration.

This study was undertaken with the following objectives:

1. To evaluate wastewater treatment performance and sludge quality of a lab-scale single compartment complete mix activated sludge tank using the novel AOSD control system and fix ON/OFF sequences of time in a 120min aeration-anoxia cycle.
2. To assess AOSD system as an environmental friendly system allowing energy saving and greenhouse gases emission reduction.
3. To investigate possible improvements of the AOSD system by determining its assets and limits and eventual additional control parameters.

Chapter 2: Materials and preliminary experiments

2.1 Introduction

Lab-scale single compartment reactors were built to study effect of AOSD and fixed On/Off aeration time systems on standardized domestic wastewater. Preliminary experiments on continuous aeration, diffuser type, aeration flow rate, sludge concentration and AOSD parameters settings were then conducted in order to determine optimal conditions of operation. A test on extrusion system was also run to envisage possibility of discarding stirring in order to save more energy.

The intermittently aerated single reactor used in this study is known as Alternating Aerobic Anaerobic (AAA) complete mix activated sludge system. Main advantages of AAA systems over conventional activated sludge are TN removal and energy saving. Both are directly affected by aeration time. Therefore choice of diffusers, air flow rate, aeration time and cycle duration were important steps. AOSD system's main novelty consists in relying presently solely on DO and temperature measurements provided by a single probe to calculate optimal aeration time. Those two parameters are reliably measured by new apparatuses compared with ORP probes for example which tend to drift and require frequent readjustments. The AOSD system's algorithm determines optimal aeration time in 2 hours aerobic/anoxic cycle based on DO levels collected from the previous cycle. Initial cycle (at start of the system) is set to 60/60 aeration/anoxia. Modifiable parameters were tested during those preliminary experiments to optimize AOSD functioning under selected conditions.

This chapter is dedicated to materials description and main operation parameters that were selected for the experiments as well as the preliminary tests results.

2.2 Experimental device

The AAA system was selected as previously mentioned for its simplicity and possibility to easily adapt existing BNR (Biological Nutrients Removal) processes, especially oxic ditches and SBR, to AAA system. It fits better small collectivity wastewater treatment plants. Modifications to the basic process were suggested by researchers such as dividing tank into 2 compartments (Insel et al., 2006). Despite its simplicity and good performance, it is not a much widespread system. The most resembling system to it is SBR with the influent flow type as major difference.

Laboratory scale experimental device (**Figure 2-1, Picture 2-1**) was designed to replicate functionalities of secondary treatment in an activated sludge wastewater treatment plant using single compartmented tank reactor and settling tank.

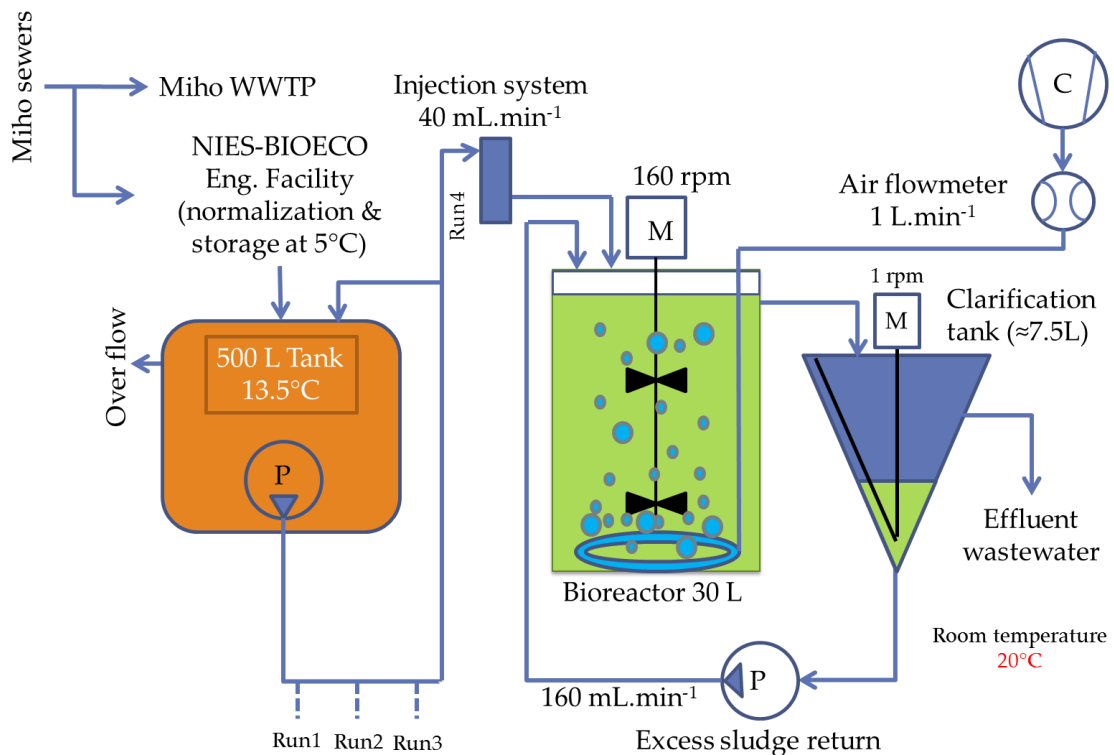
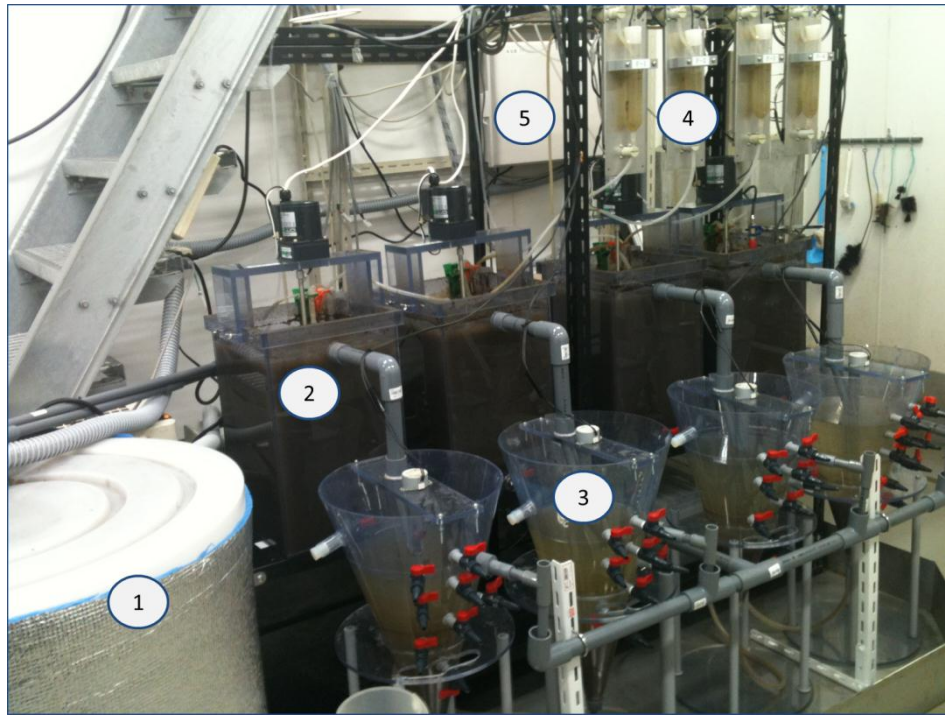


Figure 2-1 Experimental device and main operation parameters



Picture 2-1 Experimental device

Legend: ① Influent 500 L intermediary storage tank. ② Reactor ③ Clarification tank ④ Injection system composed of a glass holder, an upstream Normally Closed electrovalve (controlled by level and timer) and a downstream Normally Open electrovalve (controlled by timer) ⑤ Electrical panel containing timers and speed variators.

It consisted of four Runs, each composed of a 28x28 cm² section reactor with approximately 30 L working capacity as well as a conic decantation tank with working volume set to around 7.5 L by selecting the appropriate effluent outlet. Each reactor was equipped with a continually operated two fans stirrer operated at around 160 rpm by a speed variator. A chain slowly rotating (1 rpm) scraped walls of settling tank and helped dispersed sludge floc. There was no trap to prevent scum from washout. An attempt was made by installing a T tube connector to make water below surface level flow out the tank free from scum. But it was quickly abandoned as it was blocking chain and eventually caused motor to break down. Air was blown by a 40W air compressor. Flow rate was controlled by in-line flow meter. Hygrometry becoming very high around summer period, condensed water was accumulating in aeration tubes and reaching flow meters. Dew traps were therefore installed at compressors' outlets (**Picture 2-2**).



Picture 2-2 Condensed water trap

High hygrometry in summer period caused condensation deposits on tubes and pipes carrying influent due to its lower temperature but also inside air tubes and air flow meters under effect of air compression by blowers. Bottles were successfully used to trap water drops and prevent their reaching flow meters.

Condensation on influent tubes and pipes also caused its share of incidents that were remediated to by simple actions (**Annex A**).

2.3 AOSD system

One of the most important features of the AAA process is the control flexibility that copes with unavoidable fluctuations of influent's quantity/quality load. Control systems are therefore the core of the process performance (Chachuat et al., 2005; Kim et al., 2000; Zhao et al., 1995). Most of the existing control systems require at least two main control parameters making the system relatively complex and often requiring maintenance, high technical operation skills and frequent adjustments.

During the development of the AOSD system, some considerations were taken into account. Importance was given to simplicity of the system, basing the functioning

principle on DO and temperature measurement, designing an environment friendly tool for easy and optimal control of nitrification/denitrification processes, adaptability to a variety of wastewater treatment plant configurations and last but not least to openness to possibilities of improvement and upgrade to full automation.

The AOSD system is composed in its hardware part of a Programmable Logic Controller (OMRON sysmac CP1L) that receives readings of DO and temperature from a HACH LDO probe and its sc100 model controller (**Picture 2-3**).

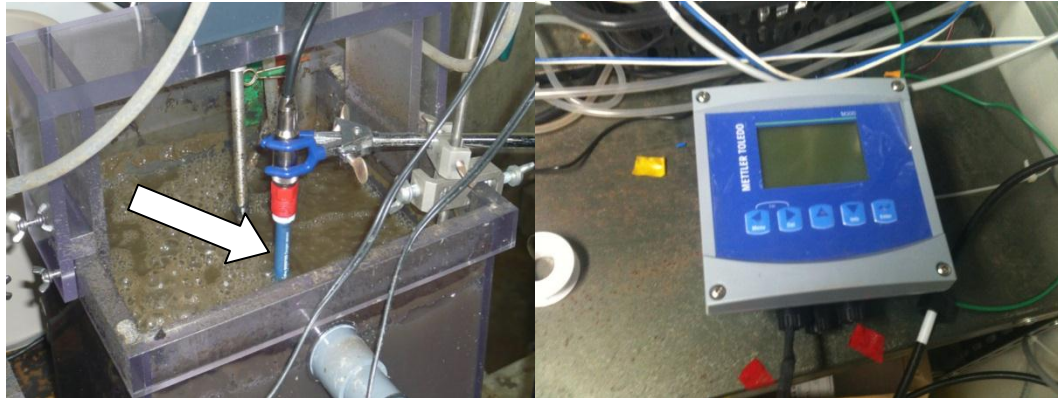


Picture 2-3 AOSD hardware components

The DO probe (a) is immersed in aeration tank (collar and angle iron pieces were used for fixation to tank's edge) and connected to the probe controller (b). DO and temperature values are transmitted to the PLC (c) by 4-20mA output signal.

The PLC was also used to control air compressor's functioning duration of the fixed On/Off Runs.

Prior to installation of a luminescent DO probe, part of preliminary tests were run using a Mettler-Toledo M300 transmitter connected to a polarographic InPro6050 DO sensor (**Picture 2-4**).



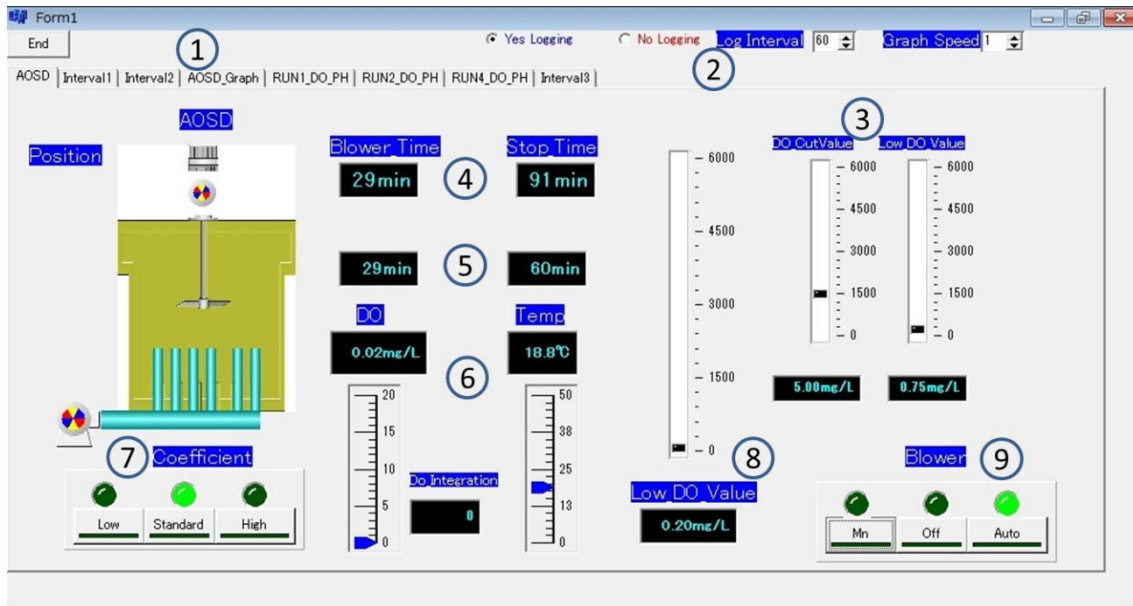
Picture 2-4 Polarographic DO probe and its controller

Picture on the left shows the polarographic DO probe installed in the reactor. Picture on the right shows the probe controller used for interpretation of signal from the probe, displays control menu and measurements, and provides 4-20mA output signal

This type of DO meter was abandoned for its inadequacy to wastewater treatment conditions. Indeed, the sensitive membrane, fouled by sludge and worms, was easily torn during the very frequent cleanings it required under those conditions, leading to high inaccuracy in DO measurement and thus to inadequate aeration time calculated by ASOD algorithm. Control and maintenance (replacement of the membrane, refilling the probe with solution and recalibration) were therefore often necessary, time consuming and results were biased.

On the other hand, except for the occasional preventive cleaning of sensor's cap to remove slime deposits and keep measurements accurate, the LDO probe required virtually no maintenance under those conditions and caused no downtime in almost 3 years of utilization.

The PLC was connected via Ethernet cable to a laptop used as control interface and for data storage. The graphic interface permits to easily modify some parameters related to AOSD system response, to stop blower or set it to manual or automatic mode and to set interval time of data logging into a Microsoft Office Access database (**Picture 2-5**).

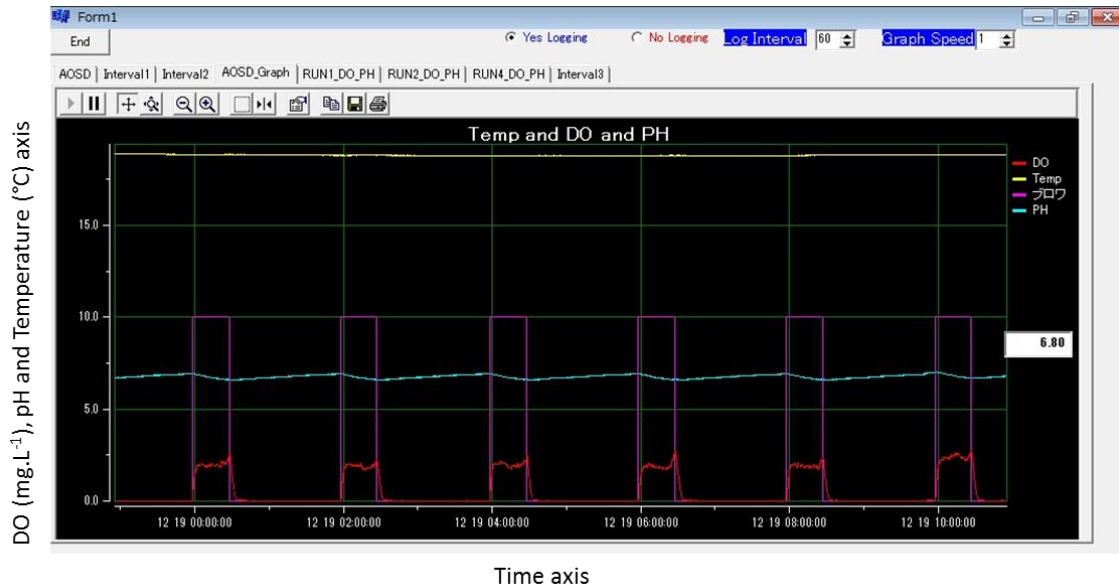


Picture 2-5 Control panel for AOSD system parameters setting

Legend: ① The tab menu allows choosing information to be displayed (parameters or graph for each Run). ② Options for data logging and graph display are available. ③ AOSD system can be forced to stop the blower if DO reaches a maximum defined value ($5.00 \text{ mg}\cdot\text{L}^{-1}$ in the picture) during aeration calculated time; the minimum threshold ($0.75 \text{ mg}\cdot\text{L}^{-1}$ in this case) is used to restart blower if aeration time is not over. ④ Display of calculated aerobic and anoxic phases in a 120min intermittent aeration cycle. ⑤ Remaining time of each phase. ⑥ Digital and graphic display of DO and temperature instant readings, DO integration represents cumulative DO readings during aeration time. ⑦Preset values to control nitrification/denitrification ratio performance. ⑧ Threshold setting for denitrification. ⑨ Blower operation mode (Manually turn ON/OFF, or leave control to PLC)

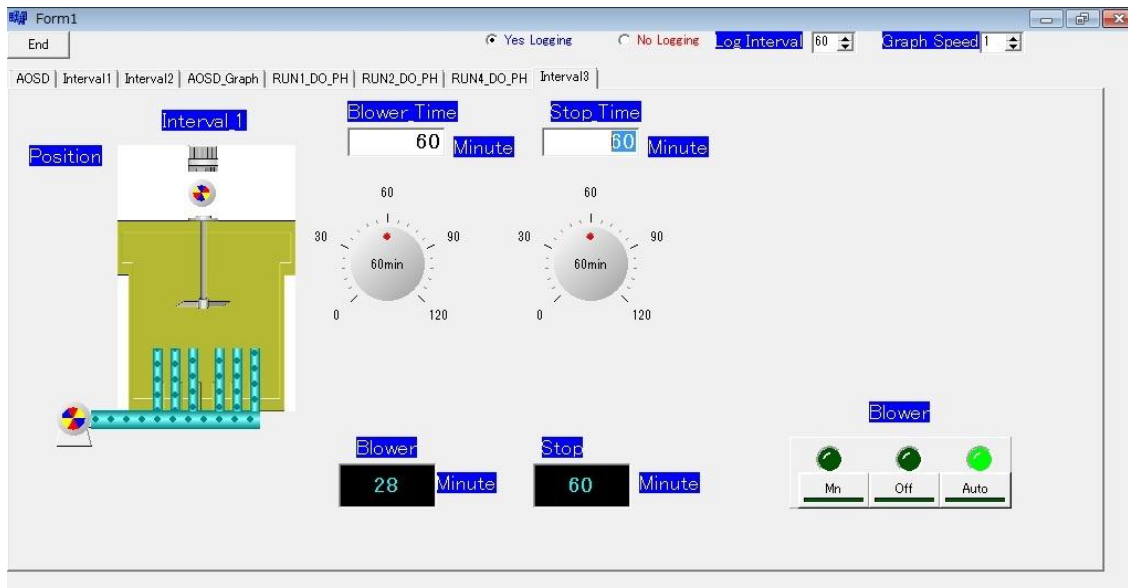
Monitoring of instant situation and history visualization are made easier by a graphic plot of DO, temperature and blower operation (**Picture 2-6**). In the AOSD interface software, menu tabs facilitate modification of fixed ON/OFF time systems parameters and put their respective blowers to automatic or manual mode (**Picture 2-7**). Improvements and features were gradually added to the software according to

the needs. Attention was not accorded to user-friendliness as its main purpose was solely to serve this study.



Picture 2-6 Graphical display of AOSD data

Data of DO (red line), pH (cyan line) and temperature (yellow line) are plotted (Y axis) versus time (X axis). Blower's operation is also represented (magenta line). Tools to modify scale of axis, scroll time axis, zoom in/out part of the graph, save and print are available to facilitate visualization of data.



Picture 2-7 Control panel of fixed 60/60 aeration system

Control panel for fixed On/Off time allows easy modification of aeration and anoxia phases durations as well as switching between automatic and manual modes.

When AOSD program is initiated, a default cycle of 60/60 is initialized. Optimal aeration time is then metaheuristically calculated based on data collected from ended cycle (DO, temperature, OUR) using nitrification & denitrification integrated models and considering coefficients and parameters settings. During aeration period, it is possible to control DO range by simply setting thresholds. This has the double interest of keeping DO at desired range and to save energy. However, this option was not used for long term evaluation periods reported in Chapters 3 & 4. Collected data from each cycle are used to calculate time for the next in an endless loop unless system is stopped or reinitialized (**Figure 2-2**).

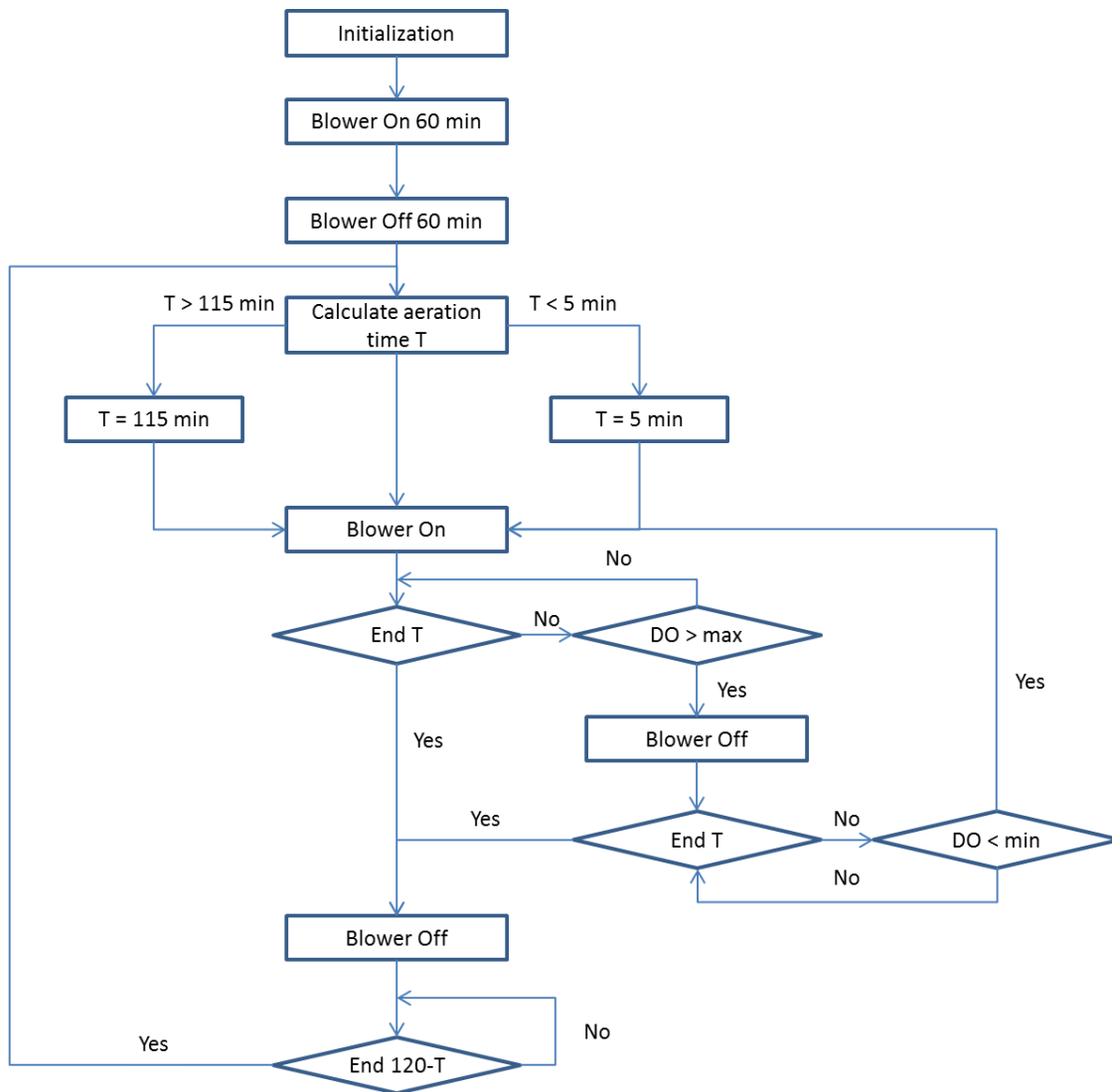


Figure 2-2 Simplified algorithm of AOSD system

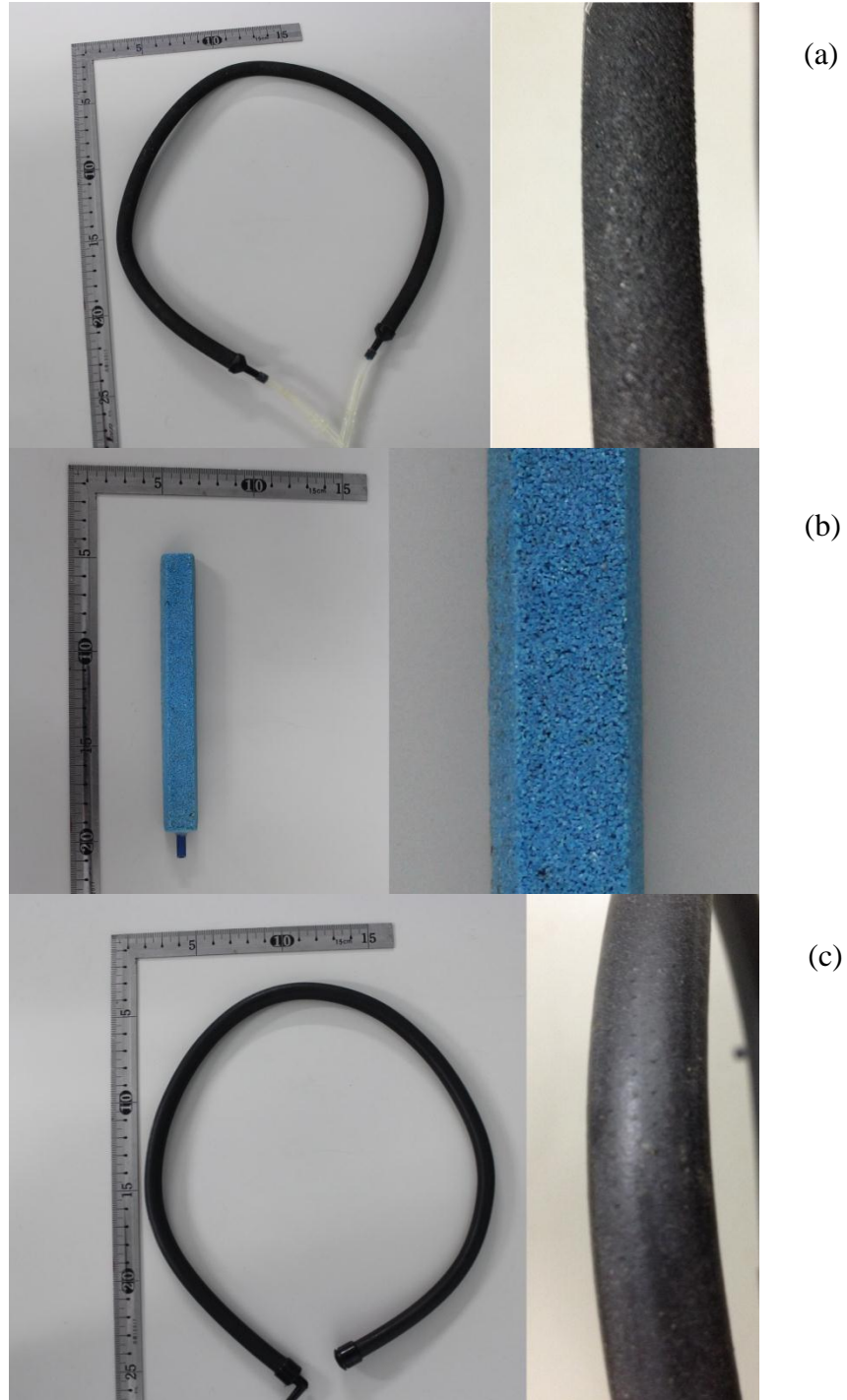
A minimum aeration time of 5min per cycle is justified by the necessity to provide oxygen for endogenous oxidation to maintain sludge activity in case influent feeding is stopped for maintenance reasons for example. Yilmaz et al. (2007) reported that biomass of a lab-scale SBR underwent a 6week period of starvation with 15min aeration per 6h cycle and recovered full performance after 4days resuscitation period during which influent load was gradually increased. In the same logic, a minimum anaerobic period of 5min was kept in case of very low DO to maintain denitrifying organisms. In this study though, no experiment was conducted to confirm the pertinence of maintaining minimum aeration and anaerobic periods or whether 5min was sufficient in both situations. The extreme values of aeration time were nevertheless observed respectively during unexpected long periods of technical failure in the experiment device or the annual maintenance in Bio-eco Engineering Laboratory (where the intermediary storage tank capacity is insufficient), and during utilization of polarographic DO probe.

The AOSD coefficients and parameters were set in the reported main experiment periods of the next chapters to favor denitrification and energy saving by minimizing aeration time. The system was therefore bound to adjust aeration time to around 30% of total cycle time under normal conditions of operation. Experience showed though the ability of AOSD, with same parameter settings, to reduce aeration time to 5 min in case of influent feeding stopped, and increase it up to 35 min in case of influent strength higher than normal.

2.4 Diffusers

Diffuser is a major component of the aeration system. Size of bubbles is determined by diffuser's pores size and air pressure (itself conditioned by blower's characteristics as well as linear and singular pressure losses) but also by depth of the tank. It is meant to operate under conditions of chemical and biological corrosion. Choice of appropriate diffuser and its maintenance are therefore important.

Three types of aquarium diffusers were tested (**Picture 2-8**). The (a) type diffuser was first used. It allowed satisfactory oxygen transfer by producing small enough bubbles especially at high air flow rates.



Picture 2-8 Types of diffusers

After a period of time air flow rate was decreasing due to slime and growth of filaments (big numbers of worms were visible too) that fouled diffuser's pores as shown in **Picture 2-9**. Fouling is a common issue with fine bubble diffusers (USEPA, 1999b). Frequent air flow rate controls and diffusers cleaning, by soaking in solution of H_2O_2 for 24 hours, were therefore required. Position of the diffuser at the bottom of tank, in contact with settled slimy filamentous sludge, the material it is made of and its rough texture probably favored fixation of sludge on it and thus the fouling.



Picture 2-9 Diffuser fouled by slime, filaments and worms.

The rod shaped stone (b) was tested to see if its material would help reduce the fouling problem but it produced too coarse bubbles resulting in poor oxygen transfer and thus low performance of treatment. It was therefore replaced by another flexible diffuser with smooth surface (type (c)). Last type gave more satisfaction with acceptable quality of bubbles and material not allowing fixation and growth of microorganisms on it like with the (a) type. This kind of diffuser was 60 cm long with pores made by needles of 0.5 mm in diameter. Recommended operation air flow rate was a minimum of $2.0 L \cdot min^{-1}$. In order to improve bubble size, length can be modified by simply cutting the diffuser (as it is composed of a ductile metallic frame covered with a soft

moss-like material) and close the cut end with the easily dismantled plastic extremity. This was not done though in order to keep the diffuser in a larger diameter circular shape allowing a better spatial distribution of bubbles.

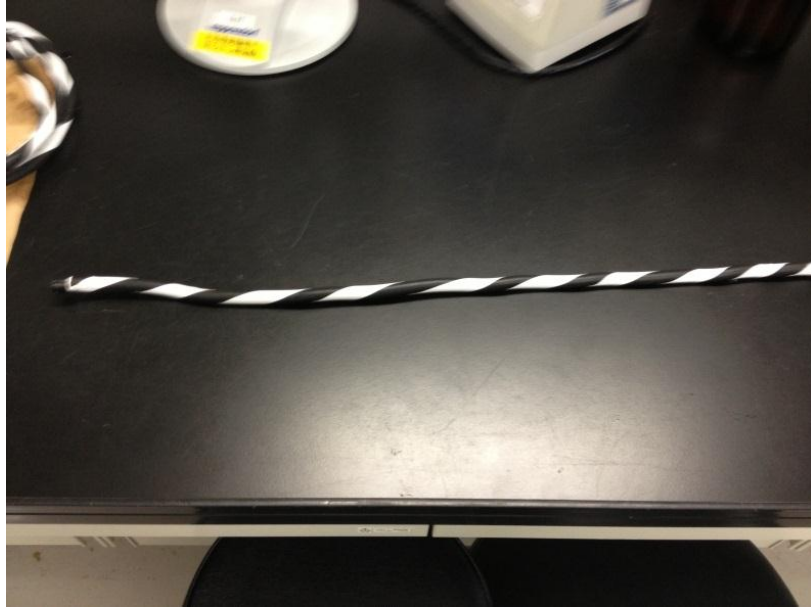
The test shown in **Picture 2-10** was run to get a gross idea of the difference in bubble size and spatial distribution between the three types of diffusers operated at flow rate of $1.0 \text{ L}\cdot\text{min}^{-1}$.



Picture 2-10 Test of diffusers operated at $1.0 \text{ L}\cdot\text{min}^{-1}$ air flow rate

It revealed clearly that the stone diffuser produced big and thus quickly rising bubbles causing therefore insufficient O_2 dissolution. For the two others it was more difficult to judge which was more efficient as type (a) produced bigger and less bubbles at its middle part while this occurred at end part type (c) diffuser.

An attempt was made to improve(c) type diffuser bubbling quality at low Q_a while keeping its 60cm original length by trying to reduce its exchange surface using PTFE tape as shown in **Picture 2-11**.



Picture 2-11 Type (c) diffuser partially covered with PTFE tape

Tightness of the tape on the diffuser and pitch of the thread proved unlikely to be perfectly reproduced for all Runs. Bending the diffuser also altered tape's tightness and PTFE adhered less on diffuser's surface once wet. It was though tried for a short period in the four Runs but results were not conclusive.

2.5 Wastewater characteristics

While some researchers choose to use synthetic wastewater, opportunity was granted here to conduct experiment with real domestic sewage and benefit from all necessary laboratory equipment and consumable at Bio-eco Engineering Laboratory of the National Institute of Environmental Sciences-Japan (NIES).

Domestic wastewater -from Miho agricultural village wastewater treatment plant in Inashiki, Ibaraki Prefecture - Japan (Lat. $+36^{\circ} 0' 18.06''$, Long. $+140^{\circ} 21' 26.39''$)

was used for experiment. A pipe derived directly from sewers collector of the plant brings wastewater to the NIES- Bio-eco Engineering Laboratory situated at approximately 700m from municipal plant on Kasumigaura lakefront. Sewage is analyzed and adjusted to standards of Japanese decentralized wastewater treatment system -known as Johkasou- (TN: 45 mg•L⁻¹; TP: 5 mg•L⁻¹; BOD: 200 mg•L⁻¹; SS: 150 mg•L⁻¹) and then stored in tanks at 5°C until its utilization. Standardization of water is achieved by addition of (NH₂)₂CO to adjust TN, CH₃OH for BOD, KH₂PO₄ for TP and toilet paper for SS. An intermediary storage tank was used for this experiment. It served prevention of substrate shortage but also helped raise water's temperature to 13.4~15.4°C to lessen thermic shock effect on the reactors which were operated at 20°C room temperature.

Inoculum of sludge used for experiment was also brought from Miho wastewater treatment plant. Sufficient volume was taken directly from plant's aerator stabilized at around 4 000 mg•L⁻¹ MLSS.

Samples of raw water were systematically taken from up stream of injection system (inlet of reactors) along with treated water's. The fluctuations of SS; BOD and COD in time and compared to standardized influent are mainly due to equipment and microbial activity. Partial decomposition of organic matter couldn't be avoided as influent was stored for an average of 24 hours at temperatures ranging from 13.5 to 15.5°C. Initial design of experimental device was improved by adding an immersed pump in storage tank for the sole purpose of mixing resulting in increased homogeneity of influent and decrease in frequency of tank walls cleaning from solid deposits. Position of wastewater supply PVC pipe was also changed to shorten length of the small diameter silicon tubes used to feed each Run's injection system allowing to limit pressure drop and quantity of deposits in the tubes (**Picture 2-12**). Gain in pressure also reduced time necessary to fill influent glass holders, decreasing thus deposit of solids and maintenance frequency of the injection system. Feeding and

mixing pumps in storage tank affected though wastewater composition by screening part of suspended solids, dissolving oxygen and dissipating heat in wastewater.



Picture 2-12 Deposits removed from influent tube during maintenance

Silicon tubes connecting main feeding PVC pipe to glass cylinders of volumetric injection system were made shorter. This improvement diminished pressure loss and contact surface resulting in less solids deposit in the inner wall of silicon tubes.

Improvements of experimental device reduced cleaning and maintenance operations from 2-3 times a week to once a week and also decreased occurrences of some technical failures related to maintenance.

Averages of influent quality analyses are reported in **Table 2-1**.

Table 2-1 Characteristics of influent wastewater

	SS (mg•L ⁻¹)	BOD₅ (mg•L ⁻¹)	COD_{Mn} (mg•L ⁻¹)	TN (mg•L ⁻¹)	TP (mg•L ⁻¹)
Mean	145.2	131.9	98.0	43.2	4.75
Max	172.0	210.4	134.5	51.2	5.78
Min	124.0	88.8	73.5	35.0	3.87
S.D.	13.2	29.2	12.4	3.3	0.51

Assuming that there are inevitable variations in wastewater quality during normalization process and storage due to analysis, volumes of wastewater, mixing, etc., considering that SS, TN and TP are in average close to standard values (respectively 150, 45 and 5 mg•L⁻¹), considering that BOD₅ measurement is statistically unreliable and assuming that pollutants losses from the system are negligible due to their transformation mainly into SS that ends up in the reactors, influent quality standard values were adopted in calculi instead of mean values reported in the table above.

2.6 Preliminary experiments

Determination and control of optimal cycle length and aerated time fraction were investigated by many researchers using various systems and reactor configurations under different operation parameters to treat wastewaters (Battistoni et al., 2003; Chachuat et al., 2005; Doan and Lohi, 2009; Dotro et al., 2011; Gresch et al., 2011; Hanhan et al., 2011; Hasar et al., 2002; Kim et al., 2000; Lim et al., 2007; Paul et al., 1998; Tocchi et al., 2012).

For this study, in order to check response of the AOSD system, fine tune its parameters and determine main operation conditions to carry on long term follow ups, a series of preliminary experiments was conducted. Commonly used values for most operation parameters of activated sludge process were considered in our decisions such as standards for HRT, MLSS and SRT. Among the most important questions raised at start of experiments regarded air flow rate, aeration ON/OFF time ratio and total length of the cycle. Some hardware modifications were also undertaken during this period of preliminary tests. Average results of effluent analysis and corresponding parameter changes are summarized in **Table 2-2**.

Table 2-2 Main preliminary tests and results obtained in each Run

Run1	Run2	Run3	Run4
Room temperature=14°C, HRT=12 hours, MLSS=2,000 mg•L ⁻¹ , Qa=2.0 L•min ⁻¹			
Continuous aeration	30/30	30/60	AOSD
BOD = 36 mg•L ⁻¹ COD = 42 mg•L ⁻¹ TN = 42.5 mg•L ⁻¹ TP = 4.3 mg•L ⁻¹	BOD = 28 mg•L ⁻¹ COD = 27 mg•L ⁻¹ TN = 29.4 mg•L ⁻¹ TP = 3.4 mg•L ⁻¹	BOD = 13 mg•L ⁻¹ COD = 15 mg•L ⁻¹ TN = 8.7 mg•L ⁻¹ TP = 2.7 mg•L ⁻¹	BOD = 28 mg•L ⁻¹ COD = 23 mg•L ⁻¹ TN = 26 mg•L ⁻¹ TP = 2.7 mg•L ⁻¹
	30/90		
	BOD = 21 mg•L ⁻¹ COD = 39 mg•L ⁻¹ TN = 29.4 mg•L ⁻¹ TP = 3.4 mg•L ⁻¹		
Qa 1.5 L•min ⁻¹			
BOD = 21 mg•L ⁻¹ COD = 39 mg•L ⁻¹ TN = 39.6 mg•L ⁻¹ TP = 4.1 mg•L ⁻¹ pH = 5.5	BOD = 16 mg•L ⁻¹ COD = 13 mg•L ⁻¹ TN = 10.9 mg•L ⁻¹ TP = 2.6 mg•L ⁻¹ pH = 6.9	BOD = 12 mg•L ⁻¹ COD = 12 mg•L ⁻¹ TN = 15.8 mg•L ⁻¹ TP = 2.7 mg•L ⁻¹ pH = 6.9	BOD = 12 mg•L ⁻¹ COD = 11 mg•L ⁻¹ TN = 13.8 mg•L ⁻¹ TP = 2.6 mg•L ⁻¹ pH = 6.9
Room temperature 25°C, Qa 1.0 L•min ⁻¹			
BOD = 15 mg•L ⁻¹ COD = 34 mg•L ⁻¹ TN = 41.7 mg•L ⁻¹ TP = 4.2 mg•L ⁻¹ pH = 5.4	BOD = 42 mg•L ⁻¹ COD = 44 mg•L ⁻¹ TN = 38.5 mg•L ⁻¹ TP = 4.0 mg•L ⁻¹ pH = 7.3	BOD = 16 mg•L ⁻¹ COD = 11 mg•L ⁻¹ TN = 16.7 mg•L ⁻¹ TP = 2.7 mg•L ⁻¹ pH = 6.7	BOD = 14 mg•L ⁻¹ COD = 12 mg•L ⁻¹ TN = 20.1 mg•L ⁻¹ TP = 2.1 mg•L ⁻¹ pH = 7
Mixing replaced by extrusion flow system, Qi= 33 mL•min ⁻¹			
BOD = 7 mg•L ⁻¹ COD = 17 mg•L ⁻¹ TN = 33.4 mg•L ⁻¹ TP = 3.3 mg•L ⁻¹ pH = 6.0	BOD = 10 mg•L ⁻¹ COD = 17 mg•L ⁻¹ TN = 34.3 mg•L ⁻¹ TP = 2.6 mg•L ⁻¹ pH = 7.1	BOD = 11 mg•L ⁻¹ COD = 19 mg•L ⁻¹ TN = 28.1 mg•L ⁻¹ TP = 2.1 mg•L ⁻¹ pH = 7.0	BOD = 11 mg•L ⁻¹ COD = 17 mg•L ⁻¹ TN = 31.6 mg•L ⁻¹ TP = 1.4 mg•L ⁻¹ pH = 6.9
Back to mixing system, Qi= 40 mL•min ⁻¹ , Room temperature 20°C			
BOD = 10 mg•L ⁻¹ COD = 31 mg•L ⁻¹ TN = 42.3 mg•L ⁻¹ TP = 4.2 mg•L ⁻¹ pH = 4.1	BOD = 14 mg•L ⁻¹ COD = 12 mg•L ⁻¹ TN = 7.1 mg•L ⁻¹ TP = 2.7 mg•L ⁻¹ pH = 6.6	BOD = 13 mg•L ⁻¹ COD = 11 mg•L ⁻¹ TN = 9.3 mg•L ⁻¹ TP = 1.9 mg•L ⁻¹ pH = 6.6	LDO probe installed BOD = 9 mg•L ⁻¹ COD = 12 mg•L ⁻¹ TN = 8.8 mg•L ⁻¹ TP = 2.9 mg•L ⁻¹ pH = 6.5

Table 2-2 Main preliminary tests and results obtained in each Run (continue)

Run1	Run2	Run3	Run4	
MLSS=3,000 mg•L ⁻¹				
Qa 0.5 L•min ⁻¹ BOD = 13 mg•L ⁻¹ COD = 29 mg•L ⁻¹ TN = 41.5 mg•L ⁻¹ TP = 4.0 mg•L ⁻¹ pH =4.3	BOD = 31 mg•L ⁻¹ COD = 24 mg•L ⁻¹ TN = 14.6 mg•L ⁻¹ TP = 2.8 mg•L ⁻¹ pH =6.7	BOD = 5 mg•L ⁻¹ COD = 9 mg•L ⁻¹ TN = 11.7 mg•L ⁻¹ TP = 2.7 mg•L ⁻¹ pH =6.5	BOD = 22 mg•L ⁻¹ COD = 13 mg•L ⁻¹ TN = 16 mg•L ⁻¹ TP = 1.9 mg•L ⁻¹ pH =6.9	
Qa 0.25 L•min ⁻¹			AOSD coef. Std	
BOD = 17 mg•L ⁻¹ COD = 15 mg•L ⁻¹ TN = 34.1 mg•L ⁻¹ TP = 3.0 mg•L ⁻¹ pH =6.45	Media introduced		BOD = 20 mg•L ⁻¹ COD = 16 mg•L ⁻¹ TN = 11.5 mg•L ⁻¹ TP = 2.8 mg•L ⁻¹ pH =6.6	
	BOD = 26 mg•L ⁻¹ COD = 14 mg•L ⁻¹ TN = 20.0 mg•L ⁻¹ TP = 1.5 mg•L ⁻¹ pH = 6.6	Permutation of AOSD & 30/60 systems		
		Cycle 100min		
		BOD = 24 mg•L ⁻¹ COD = 23 mg•L ⁻¹ TN = 19.6 mg•L ⁻¹ TP = 2.9 mg•L ⁻¹ pH =6.8	BOD = 9 mg•L ⁻¹ COD = 9.8 mg•L ⁻¹ TN = 8.0 mg•L ⁻¹ TP = 2.5 mg•L ⁻¹ pH = 6.4	
		BOD = 16 mg•L ⁻¹ COD = 25 mg•L ⁻¹ TN = 28.6 mg•L ⁻¹ TP = 2.3 mg•L ⁻¹ pH = 6.6		
Stone diffuser (b) installed				
30/90 Qa 1.0 L•min ⁻¹	Fan added to stir media zone	Cycle 120min AOSD coef. High	40/80	
BOD = 26 mg•L ⁻¹ COD = 12 mg•L ⁻¹ TN = 16.4 mg•L ⁻¹ TP = 2.6 mg•L ⁻¹ pH = 7.0	BOD = 18 mg•L ⁻¹ COD = 12 mg•L ⁻¹ TN = 7.3 mg•L ⁻¹ TP = 2.9 mg•L ⁻¹ pH = 6.9	BOD = 7 mg•L ⁻¹ COD = 10 mg•L ⁻¹ TN = 17.3 mg•L ⁻¹ TP = 2.7 mg•L ⁻¹ pH = 6.7	BOD = 23 mg•L ⁻¹ COD = 12 mg•L ⁻¹ TN = 16.1 mg•L ⁻¹ TP = 2.9 mg•L ⁻¹ pH = 6.9	
Needle perforated flexible diffuser (c) installed				
BOD = 13 mg•L ⁻¹ COD = 12 mg•L ⁻¹ TN = 8.1 mg•L ⁻¹ TP = 3.2 mg•L ⁻¹ pH =7.0	BOD = 10 mg•L ⁻¹ COD = 12 mg•L ⁻¹ TN = 6.9 mg•L ⁻¹ TP = 3.2 mg•L ⁻¹ pH =6.9	Qa 0.5 L•min ⁻¹ BOD = 6 mg•L ⁻¹ COD = 10 mg•L ⁻¹ TN = 16.2 mg•L ⁻¹ TP = 3.2 mg•L ⁻¹ pH =6.7	BOD = 11 mg•L ⁻¹ COD = 12 mg•L ⁻¹ TN = 10.3 mg•L ⁻¹ TP = 3.1 mg•L ⁻¹ pH = 7.0	

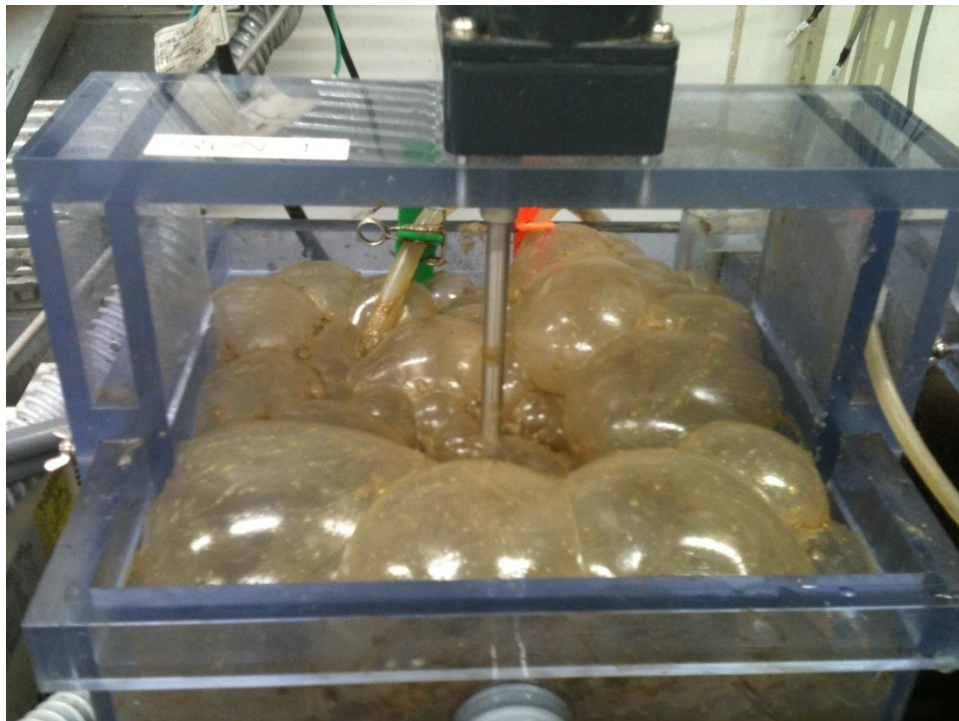
In order to obtain HRT of 12 hours, and considering the volume of reactors (30 L), influent flow rate was calculated to be around $40 \text{ mL}\cdot\text{min}^{-1}$. Due to the low flow rate, and for feasibility reasons as well as maintenance concerns, injection system was set to deliver 120 mL every 3 min. Influent flow to the reactor was though considered as continuous. Return sludge peristaltic pump was set to a flow rate of $160 \text{ mL}\cdot\text{min}^{-1}$ (four times Q_i).

The main purpose of the preliminary tests was to help determine optimal AOSD parameters, pertinent fixed ON/OFF aeration to compare with and improve experimental device, but it also helped predict some of the results detailed in the next chapters. The above results are however to be considered with caution as (1) continuity of the tests in each Run affected sludge quality (history of sludge is an important factor as treatment performance is influenced by initial characteristics of sludge), (2) some of the modifications were made under forced circumstances or for periods as short as 3-4 weeks.

Continuous aeration was assigned to Run1 in order to spotlight main differences with intermittent aeration. The most striking results obtained with continuous aeration were the very low pH reached (as low as pH 4.1) and the very poor TN removal (below 10%). Nitrification was occurring despite extreme pH values for bacterial activity and no chance was given for anaerobic denitrification to occur. Infestation by worms (**Picture 2-13**) combined with some extreme conditions (low pH and high DO), and high effluent turbidity made it very difficult to keep MLSS at the set minimum. When Q_a was decreased to $0.25 \text{ L}\cdot\text{min}^{-1}$, conditions improved a little but effluent quality in general and TN removal specifically remained very poor. Other than high turbidity of effluent, Run1 also showed episodes of foaming (**Picture 2-14**).



Picture 2-13 Balled up worms from Run1 under continuous aeration conditions



Picture 2-14 Phenomenon of foaming in Run1 (continuous aeration)

Regarding cycle duration, it ranged from 30min to 8 hours in most reviewed literature. In this study, considering constancy of influent load (in quantity and quality) and of temperature, considering the relatively low maintained MLSS, cycles of 90 and 120 min resulted in average to good effluent quality. But performance seemed highly affected by aeration/anoxia ratio and air flow rate. In the AOSD system, the ON/OFF ratio time is controlled by a parameter named here nitrification/denitrification coefficient (AOSD coef. in **Table 2-2**). The words Low, Standard and High respectively represent ratios theoretically adjusting system's response to influent low, standard or high load. Short aeration time and poor quality air bubbles caused bulking and sludge washout on the long term (especially with 30/90 system as shown in **Picture 2-15**). Long aeration time in a cycle (AOSD at coef. "High" and 30/60) resulted in low BOD and very good nitrification but to the detriment of denitrification. Measurements of pH were indicators of treatment performance as pH higher than 7 meant insufficient aeration for nitrification and increased risk of bulking, while pH lower than 6.8 was a sign of over aeration, pin floc and accumulation of $\text{NO}_3\text{-N}$.



Picture 2-15 Bulking in decantation tank after 4 months of 30/90 operation

Air flow rate is a very important parameter in terms of energy consumption and treatment performance. Theoretical volume of O₂ required to remove pollutants is the product of air flow rate into aeration time. It defines therefore power of the blower. But Q_a also affects transfer rate of O₂ as bubble size and distribution, are determined by air pressure and thus by Q_a. Detailed information on diffusers characteristics was unfortunately unavailable and operating at low Q_i undoubtedly decreased quality of bubbles but other factors such as dimensions of the reactor, stirrer, spatial distribution of bubbles and fouling issues concurred to opt for the needle perforated flexible diffuser. Optimal Q_a was first estimated using a simulation software to be around 1.15 L•min⁻¹. Experiment showed indeed that higher air flow rates yielded poorer TN removal.

As for temperature, it was initially set to Johkasou standard but energy saving policy after the Touhoku earthquake in March 2011 compelled increase of room temperature in summer. It was then decided to keep temperature at 20°C for the rest of experimentation period, among other reasons, to collect data in prospect of full scale testing in warmer countries.

The idea behind replacing stirring by extrusion flow system (**Figure 2-3, Picture 2-16**) that forces influent upward in the mixed liquor was to save more energy as mixing is beside aeration the main energy consuming operation. Influent flow rate was modified to 100 mL•min⁻¹ to keep the same HRT as mixed liquor volume was decreased by installation of the panel.

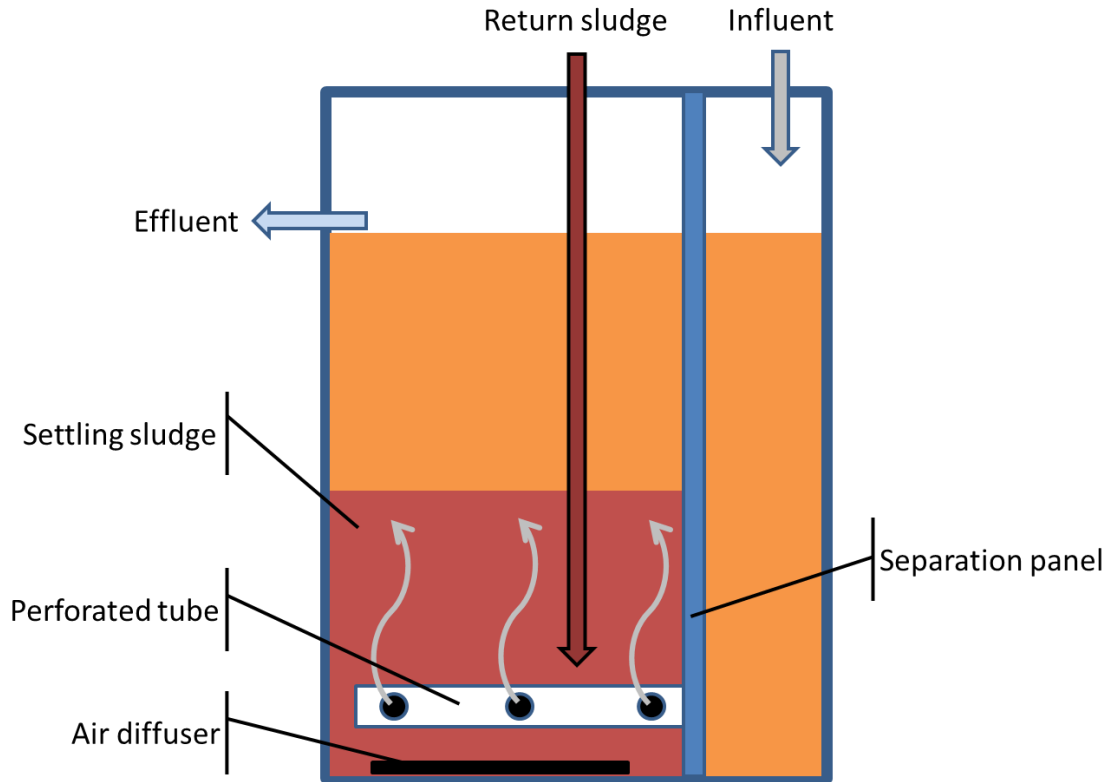
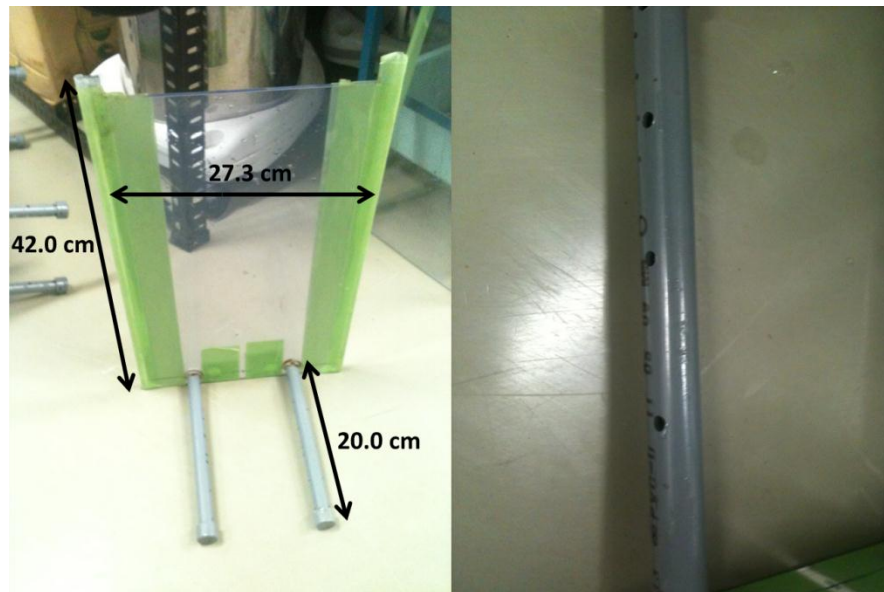


Figure 2-3 Extrusion Flow system

Separation panel with perforated tubes in replacement of mixing. During aeration Off time, sludge settles and influent is forced upward through the sludge layer. Tube of return sludge was extended to the bottom of reactor.



Picture 2-16 Extrusion flow system's panel

Tube $\text{Ø}3/4''$, hole $\text{Ø} 5\text{mm}$. the panels were placed at 5cm from the back wall of tanks reducing mixed liquor compartment to around 25L capacity; influent flow rate was modified accordingly to keep HRT of 12 hours.

Effluent quality was similar in all runs in terms of BOD and TN removals. If performance of Run1 was not much affected, the other Runs were not sufficiently mixed by bubbles during aeration leading to poor nitrification and low influent flow rate was insufficient to create important sludge settled during anoxic phase. Effect of settling on denitrification could not be assessed as the low levels of nitrates are most probably due to bad nitrification in the first place.

2.7 Conclusion

Choice of diffusers and air flow rate are critical. No information was given on the matter in reviewed researches though. Size of the experimental device restricted aeration to low flow rates and diffusers' selection to those used in small aquariums. Bubble size distribution was not studied but quality was obviously mediocre thus limiting O₂ transfer.

Continuous aeration (standard system in Japan) was not adapted to the reactor's configuration as denitrification was completely inhibited and only extremely low air flow rates, impossible with the used equipment, might have slightly improved treatment performance. However, it showed that nitrification could still occur at very low pH and many types of microorganisms survived, even well proliferated (worm infestations) under those extreme conditions.

Extrusion system in replacement of mixing yielded very poor results under all aeration systems. In order to save more energy with AOSD system was therefore suggested. It consisted of operating mixing in alternation with aeration.

Low competitiveness, slow activity of both nitrifying and denitrifying microorganisms and specific conditions they require make choice of minimum duration for each phase of aeration/anoxia cycle important. On the other hand too long phases may create disequilibrium in the nitrification/denitrification processes particularly with short HRT and high influent load variations. Hence the importance

of selecting optimal cycle duration. Under conditions of this study 120min seemed appropriate for total cycle length. Aeration/anoxia time ratio was then to be determined considering energy saving and denitrification process's slower rate. The tests led to selection of optimal parameters for AOSD and to choice of both longer (40min) and shorter (30min) fixed aeration periods to compare with on long term operation evaluation.

Chapter 3: Effect on effluent and sludge quality

3.1 Introduction

Once materials and operation parameters were selected considering targeted objective of improving TN removal and results of preliminary tests, it was decided to study long term effect of aeration control systems on effluent quality and sludge characteristics.

Kim et al. (2000) reported an average TN removal of 70 to 90% and other advantages to the AAA system such as significant energy saving, enhanced O₂ transfer, stability of pH, and slightly improved sludge reduction.

Main quality parameters -BOD, COD, nutrients and SS removals as well as pH and sludge characteristics- were measured to evaluate ability of AOSD system to determine optimal aeration time and its effect on effluent quality and sludge stability compared to fixed On/Off aeration time systems.

Despite efforts of maintenance to keep experimental device in flawless operation, incidents occasionally occurred but odd results were kept as part of sludge evolution process and because no actions were taken except fixing or changing equipment that caused the problem. The reported periods in this chapter and the next were selected for being the longest with very few upsets and no major equipment failure.

3.2 Materials and methods

3.2.1 Methods of sampling and analysis

Samples from mixed liquor, influent (see analysis results in previous chapter) and effluent were collected to run wastewater treatment quality parameters tests and analysis.

- **Effluent**

Effluent water was weekly collected for 24 hours. After careful mixing to homogenize collect tanks content, samples of 1 L volume were taken.

In order to measure SS, volumes varying from 50 to 200 mL depending on sample's turbidity were measured using a graduated cylinder and filtrated using glass fiber filters and vacuum vessel. Volumes of 1 mL were taken from SS filtrate with Eppendorff variable volume micropipette and diluted 10-fold with deionized water for nutrients analysis. For TN-TP determination, volumes of 1 mL were taken from initial samples, diluted 10-fold with deionized water, to which 5 mL of reagent solution - made of 0.9 mg NaOH and 4.0 mg $K_2S_2O_8$ per 100 mL of deionized water stirred until complete dissolution of granules- was added and the mixture autoclaved for 70 min. Pretreated samples were then filtrated and processed by a Bran-Luebber Traacs 2000 Analyzer. As for COD_{Mn} , it was processed by COD analyzer Hiranuma COD-1500 while BOD_5 was determined by the standard method (APHA, 1995).

Samples of influent were treated using the same procedure as effluent samples.

- **Mixed liquor**

Samples of mixed liquor were taken three times a week in average for MLSS measurement. A 50 mL bottle was filled from each run. Measurements of MLSS were achieved by taking 5mL from the vigorously mixed bottle and filtration using vacuum vessel and glass fiber filters that were then dried in oven set to 110°C.

Settling test was performed using 1 L of mixed liquor from each Run. After mixing, cylinders were left for sludge to settle. Sludge volume was measured at 30 min and at 180 min. Once a week, a more detailed test was run by measuring sludge volume every 5 min during the first 30 min and every 30 min after that until 180 min.

While DO was continuously monitored in Run3, measurements in other runs were made on regular basis using a HACH HQ-30d portable LDO meter. Only one apparatus was available and its internal memory is limited to 512 entries. Measurements were therefore made once a week for each reactor sequentially. Probe was immersed in the reactor to 20 cm from mixed liquor surface (same position in all tanks). Data log file was saved in USB drive before probe was moved to the next tank and data logging reinitiated. The apparatus was set to store one reading per minute allowing to plot a rather detailed DO profile of four consecutive cycles in Microsoft Excel file after data log text file was imported using a VBA macro recorded and modified (Annex B). On the other hand pH was measured almost daily in each reactor using a TOA DDK handheld pH meter.

3.2.2 Experimental setting

In order to assess long term capacity of AOSD system to calculate adequate aeration time for good TN removal at low energy cost, it was compared to fix ON/OFF time aeration systems according to the last setting of preliminary tests, namely: fixed aeration time of 30 minutes in Runs 1&2, plastic media (**Picture 3-1**) added to Run2 in a volume ratio of 20%, Run4 set to be aerated during 40 minutes and AOSD assigned to Run3 (**Figure 3-1**).



Picture 3-1 Media used in Run2

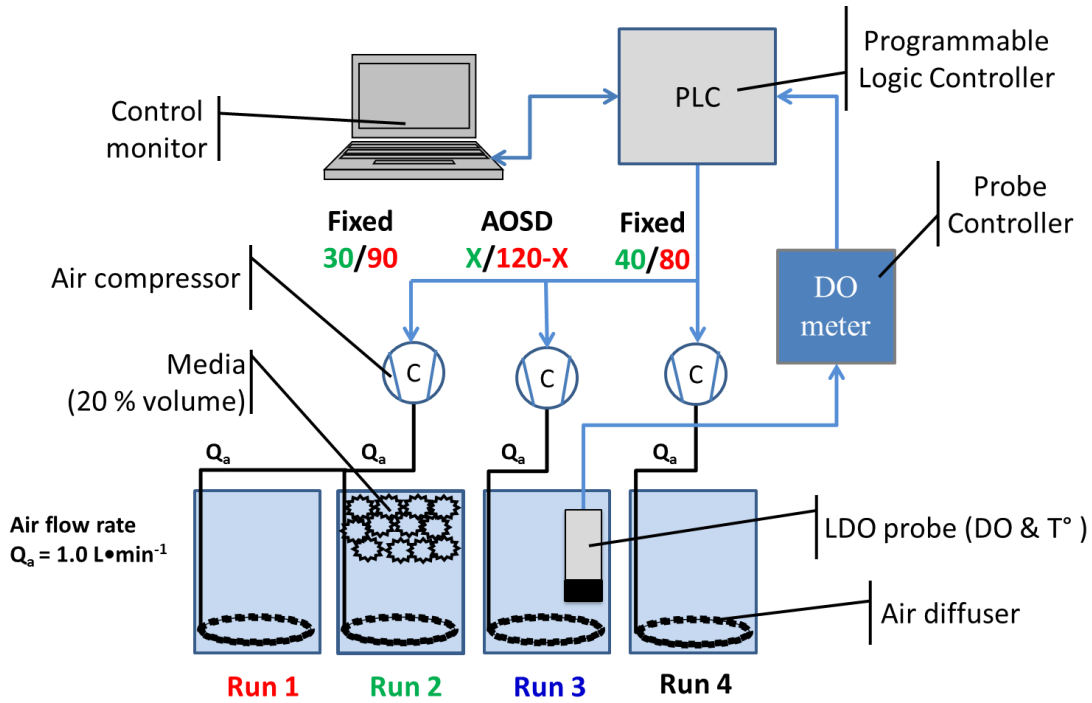


Figure 3-1 First long term experiment setting

Main operation parameters were controlled: ambient temperature of 20°C, HRT of 12 hours, Q_i of 40 mL·min⁻¹ and Q_a of 1.0 L·min⁻¹. As for MLSS, it was kept within a range of 3,000 to 3,800 mg·L⁻¹ by regular excess sludge withdrawals.

3.3 Results and discussion

Aeration time in Run3 determined by AOSD system ranged between 29 and 33 min with an average of 32 min during reported period. Main standard parameters of effluent wastewater were measured in the four Runs. Average results of a 9 weeks period without upsets are reported in this chapter. An estimation of potential energy saving by AOSD was also made.

3.3.1 Dissolved Oxygen and pH in reactors

Measurements of DO were not simultaneously made in all Runs. Results shown in **Figure 3-2** should therefore be considered with some reservations. Combined effects of raw wastewater composition variations, upset episodes (due to material failure, clogging, etc.) as well as MLSS concentration changes could not be evaluated.

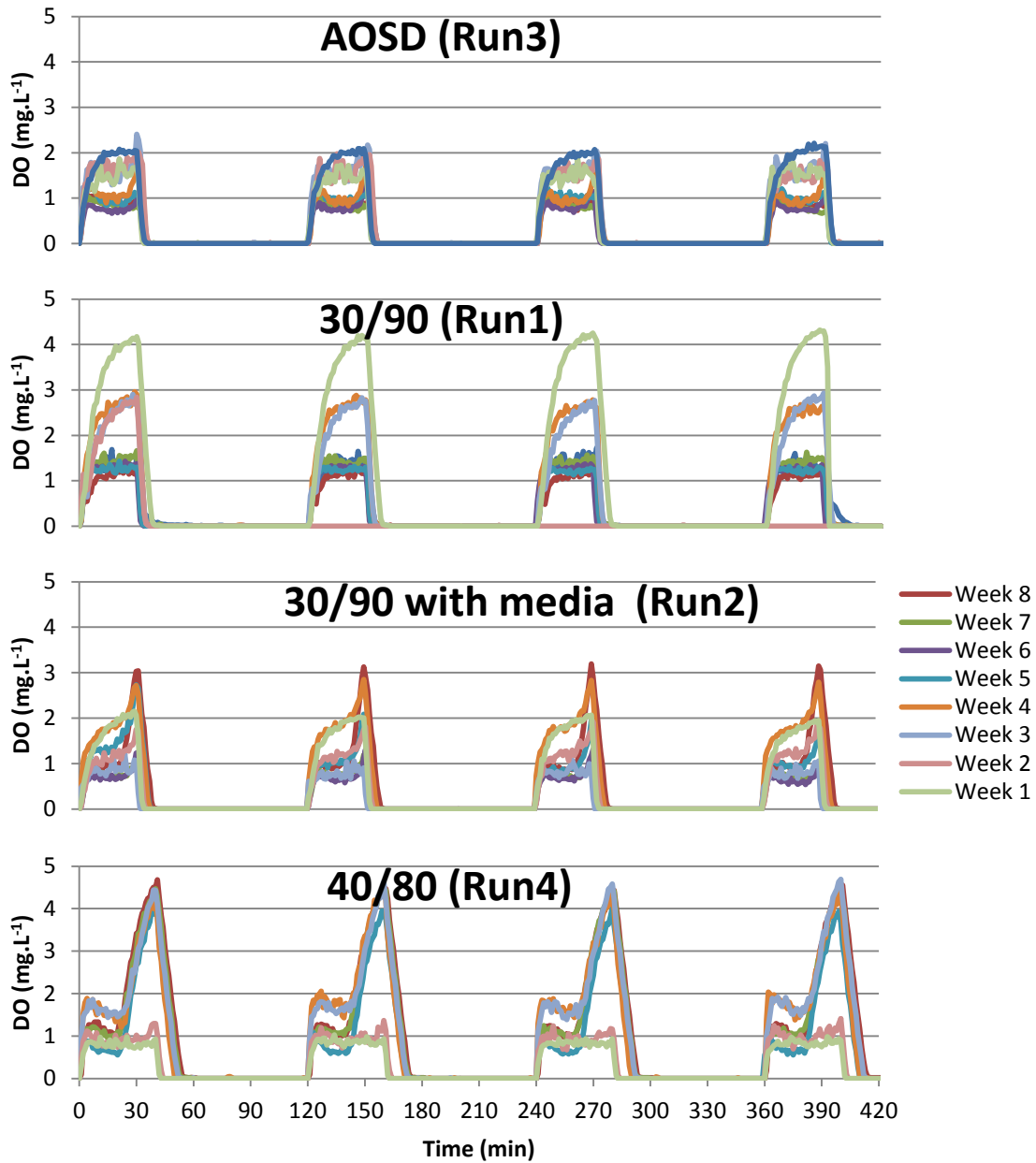


Figure 3-2 Dissolved Oxygen patterns

Profile of DO is an indicator of combined effects of influent variations, biotic strains intense selection and equipment. Increase rate of DO just after start of blower is related to equipment characteristics (latency for pressure stabilization and bubble size) and to other O₂ transfer efficiency factors (sludge characteristics, depth of tank, etc.). The various aerobic microorganisms existing in the reactor are then activated (at rates depending on their respective ecological niches) leading to DO consumption, which explains the plateau in DO profile where microbial activity rate corresponds to DO transfer rate. Depletion of NH₄-N and organic compounds reduces DO consumption and thus to peak of DO limited in these plots by blower stopping (otherwise, it would have reached saturation which is function of temperature and mixed liquor characteristics). Peaks indicate therefore over aeration and possibility to save energy.

A general decrease trend in DO plateau value was observed with Run1 which had the highest variations. From over $2.9 \text{ mg}\cdot\text{L}^{-1}$, it dropped to $1.2 \text{ mg}\cdot\text{L}^{-1}$ in the second half of experiment period. Patterns in Run3 showed that the average values during oxic periods decreased from $1.8 \text{ mg}\cdot\text{L}^{-1}$ in beginning to $1.2 \text{ mg}\cdot\text{L}^{-1}$ at the end of reported period. Run4 featured typical profile of over aeration with a bending point at around the 20th minute of aeration and DO quickly rose to $4.5 \text{ mg}\cdot\text{L}^{-1}$ after a plateau usually roaming around $1 \text{ mg}\cdot\text{L}^{-1}$ but occasionally stabilizing around $1.7 \text{ mg}\cdot\text{L}^{-1}$. Run2 DO profiles were intermediary between those of Runs 3 & 4. Values were plateauing at $1.8 \text{ mg}\cdot\text{L}^{-1}$ but decreased to $1.0 \text{ mg}\cdot\text{L}^{-1}$ and started showing bending point in second half of experiment period, probably due to O₂ transfer improvement, under effect of media, in combination with biota acclimatization and selection. Long term intermittent aeration and/or hypoxia lead to overgrowth of specific types of filamentous bacteria (Dotro et al., 2011) as well as to slime overproduction. Both affect air bubbles retention, floc oxygenation and nutrients absorption and thus growth of desirable organisms (Richard, 2003). Longer aeration time in Run4 and enhanced oxygen dissolution in Run2 limited these two phenomena. The DO peaks in patterns of Runs 2 & 4 also indicate a decrease in the oxygen uptake rate (OUR), which is represented by the DO slope of anoxic period, resulting in shorter anaerobic period. Optimal oxygen volume, considering dissolution coefficient (affected by factors such as temperature, reactor depth and bubble size), should be delivered at sufficient concentration. Authors diverge on optimal remaining DO ranging between $1 \text{ mg}\cdot\text{L}^{-1}$ and $2 \text{ mg}\cdot\text{L}^{-1}$ or higher than $2 \text{ mg}\cdot\text{L}^{-1}$ beside correct duration (Dotro et al., 2011; Hanhan et al., 2011; WEF and ASCE, 2006). But others point that these values, and DO profile in general, are highly influenced by F/M ratio and SRT (Richard, 2003; Wang et al., 2009). AOSD managed to keep DO within a narrower range showing a higher stability of biota activity.

From these results, it was deduced that, depending on aeration system, intense selection of microorganisms and different levels of slime production lead through time

to variations in DO consumption patterns. Seasonal effect of non-indigenous biota (brought to the reactor by influent) might also be of importance.

Although optimum pH for nitrification is between 7.8 and 8.0, and lower values may affect nitrifying bacteria's growth rate, the process is not completely inhibited as thought before but it requires from organisms to acclimatize (Lin et al., 2009; Painter and Loveless, 1983; Shammass and Wang, 2009). The pH may also disturb ecology of receiving waters, cause release of toxic compounds (Metcalf & Eddy, 2003; Smith, 2005) or affect toxicity levels (especially with industrial wastewater) (Richard, 2003). As for this experiment, average values were 6.95, 6.87, 6.92 and 6.78 respectively for Runs 1 to 4 (**Figure 3-3**).

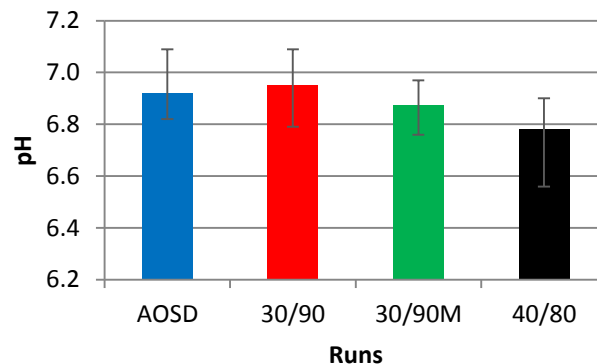


Figure 3-3 Average and variation amplitude of pH

Although difference between Runs is not important, it appears that average pH is lower at longer aeration time but also at better oxygen dissolution conditions most probably sign of higher $\text{NH}_4\text{-N}$ oxidation. Variations mostly depended on moment of measurement (slightly lower pH during oxic phase than anoxic). Average values were nevertheless close to neutral indicating correct conditions and complying with effluent standards.

3.3.2 Effluent quality

Main analyses results for BOD_5 , COD_{Mn} , TN and TP are presented in **Figure 3-4** and **Table 3-1**. Nutrients analyses are reported in **Table 3-2**.

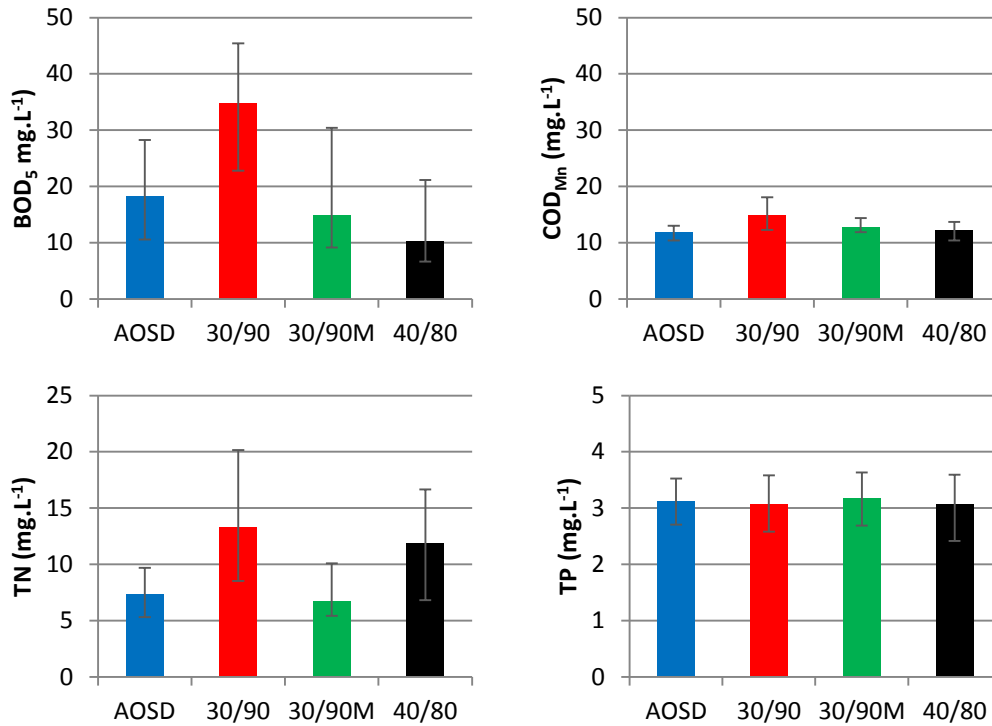


Figure 3-4 Main effluent characteristics for a straight period of 9 weeks

Table 3-1 Statistical analysis of effluent characteristics

Parameter	Descriptive statistics	AOSD	30/90	30/90M	40/80	Anova test (Fcrit=2.90)
BOD (mg·L⁻¹)	Mean	18.33	34.71	14.77	10.25	F=22.3 Difference is significant between the Runs
	Max	28.26	45.41	30.42	21.15	
	Min	10.58	22.82	9.18	6.68	
	S.D.	6.43	8.21	7.10	4.90	
COD (mg·L⁻¹)	Mean	11.91	14.89	12.81	12.11	F=9.4 Difference is significant between the Runs
	Max	13.00	18.10	14.40	13.70	
	Min	10.40	12.30	11.90	10.40	
	S.D.	0.87	2.00	0.79	1.31	
TN (mg·L⁻¹)	Mean	7.30	13.31	6.73	11.87	F=15.2 Difference is significant between the Runs
	Max	9.71	20.15	10.10	16.67	
	Min	5.33	8.54	5.43	6.81	
	S.D.	1.43	3.75	1.35	2.76	
TP (mg·L⁻¹)	Mean	3.11	3.06	3.18	3.07	F=0.17 Runs achieved statistically similar TP removal
	Max	3.52	3.58	3.63	3.59	
	Min	2.71	2.58	2.69	2.42	
	S.D.	0.32	0.34	0.42	0.47	

Table 3-2 Nutrients analysis averages

Run	NH ₄ -N (mg•L ⁻¹)	NO ₂ -N (mg•L ⁻¹)	NO ₃ -N (mg•L ⁻¹)	PO ₄ -P (mg•L ⁻¹)
AOSD	3.07	0.26	2.19	2.77
30/90	8.84	0.38	1.23	2.60
30/90M	1.78	0.29	2.37	2.79
40/80	1.19	0.34	8.66	2.71

- **BOD₅ and COD_{Mn} removal**

Except for the slightly higher value in Run1 (15 mg•L⁻¹), average effluent COD_{Mn} removal performance in other runs (12.81, 11.91 and 12.11 in Runs 2 to 4 respectively) did not yield significant conclusions. On the other hand, Runs 1 to 4 respectively averaged 35 mg•L⁻¹, 15 mg•L⁻¹, 18 mg•L⁻¹ and 10 mg•L⁻¹ in effluent BOD₅. Although BOD at those values is considered statistically unreliable, its removal performance seems positively correlated to aeration time. Run4 with higher aeration time achieved lower BOD. Aeration time in Run3 was closely similar to Runs 1 & 2, but there was a difference in performance. The poorer COD and BOD removals in Run1 suggest insufficient aeration. This was confirmed by nutrients analysis that returned for this Run an average of 8.84 mg•L⁻¹ NH₄-Nitrogen concentration in effluent. The low nitrification was at the origin of high NBOD and thus higher total BOD. Media in Run2 seem to have acted as a physical lagging factor to bubble rise and improved at the same time mixing, which enhanced oxygen dissolution. Stirring speed and position of stirrer's fans didn't allow sludge fixation on media to constitute a significant biofilm bed, and there were no consequences on MLSS concentration. Average concentration of NH₄-N in effluent from Run2 (1.78 mg•L⁻¹) showed good nitrification hence the higher BOD removal performance. As for AOSD system, it managed to compensate poor oxygen dissolution by adjusting aeration time to meet influent composition variations and microorganisms' needs. NH₄-N averaged 3.07 mg•L⁻¹ in effluent of Run3. Excepting the two first weeks, 40/80 was the only Run to

meet BOD removal fixed objective of $10 \text{ mg}\cdot\text{L}^{-1}$. This, along with nitrification performance ($1.19 \text{ mg}\cdot\text{L}^{-1}$ remaining $\text{NH}_4\text{-N}$ in average), was obtained at the cost of longer aeration time meaning more energy consumption.

- **Nitrogen removal**

Nitrogen is commonly removed in two processes. The first one, nitrification, requires oxygen to turn $\text{NH}_4\text{-N}$ into $\text{NO}_2\text{-N}$ (by the AOB type such as *Nitrosomonas sp.*) and then into $\text{NO}_3\text{-N}$ (by NOB type such as *Nitrobacter sp.*). Denitrification, transforming $\text{NO}_3\text{-N}$ to inert N_2 gas (also via $\text{NO}_2\text{-N}$), occurs under conditions of DO saturation lower than $0.2 \text{ mg}\cdot\text{L}^{-1}$ (WEF and ASCE, 2006). Runs 2 & 3 carried out higher and statistically similar average TN removal with respectively $6.7 \text{ mg}\cdot\text{L}^{-1}$ and $7.3 \text{ mg}\cdot\text{L}^{-1}$ remaining in effluent ($F=0.76$, $F_{\text{crit}}=4.49$). The values for these two Runs were below effluent standards during all reported period. Lower average of $\text{NO}_3\text{-N}$ ($1.23 \text{ mg}\cdot\text{L}^{-1}$) obtained in Run1 was not only due to sufficiently long anaerobic period for denitrification, but also to smaller amount of $\text{NO}_3\text{-N}$ produced during nitrification leading to an average effluent TN of around $13.3 \text{ mg}\cdot\text{L}^{-1}$. Nitrification process was limited by decreasing DO levels. Despite the high nitrification rate in Run4, effluent TN didn't meet standards either, with an average of $11.9 \text{ mg}\cdot\text{L}^{-1}$. Anaerobic period didn't last long enough for further denitrification, which was confirmed by the $8.7 \text{ mg}\cdot\text{L}^{-1}$ average $\text{NO}_3\text{-N}$ in treated water.

As oxic-anoxic cycle is fixed at 120 min, longer oxic periods allow more ammonia and BOD removals, but shorter time is then available for the denitrification process that not only reduces TN but also emits oxygen that is reclaimed for other reactions. Both heterotrophic anaerobic denitrifying and autotrophic aerobic nitrifying bacteria are very sensitive slow growing and feebly competitive microorganisms (Grady et al., 2011). This implies that sufficient aeration time and DO level should be provided for nitrogenous compounds to be oxidized followed by a long enough period of anoxia with sufficient easily degradable organic matter for denitrification. Therefore, aeration phase extension, in a fixed cycle time, is limited if advanced TN removal is sought.

This denotes the importance of optimizing aeration time and of nitrification/denitrification ratio coefficient in AOSD settings as well as influence of other factors such as SRT, DO, F/M ratio and C:N:P equilibrium.

- **Phosphorus removal**

Runs 1 to 4 achieved very similar average TP removal rates with effluent TP roaming around $3.1 \text{ mg}\cdot\text{L}^{-1}$. These results do not comply with standards of treated water quality although they are higher than the 30% TP removal characteristic of conventional activated sludge process (Smith, 2005). This could be explained by the presence but very slow growth of Phosphorus Accumulating Organisms' (PAOs) that require very specific conditions (Guerrero et al., 2011; Hu et al., 2002; Sasaki et al., 1996). Advanced phosphorus removal process such as chemicals or a different reactor configuration favorable to PAOs is thus necessary.

- **Suspended solids**

Results of SS measurement are reported in **Figure 3-5**. Lower effluent SS was obtained in Run3 controlled by AOSD with an average of $7.5 \text{ mg}\cdot\text{L}^{-1}$. Second better performance was realized in Run4 with $8.2 \text{ mg}\cdot\text{L}^{-1}$. Run2 achieved $9.7 \text{ mg}\cdot\text{L}^{-1}$ in average. Run1 had episodes of sludge washout that increased its average performance ($13.1 \text{ mg}\cdot\text{L}^{-1}$).

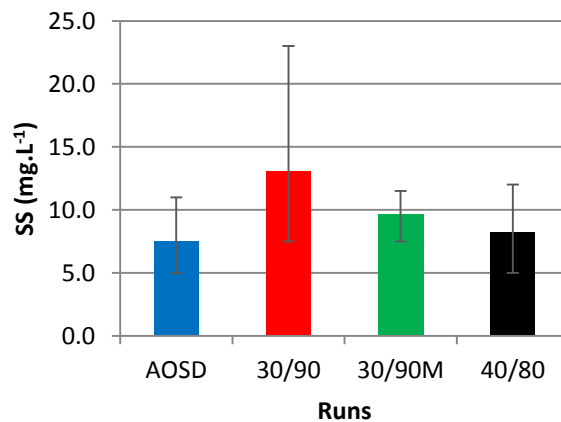


Figure 3-5 Average and amplitude of effluent SS

Note that no scum trap was installed in the settling tanks and the, otherwise regularly performed, manual scum removal was not made during the 24 hours effluent collect for sampling to avoid any perturbation.

3.3.3 Activated sludge properties

- **Sludge volume index**

Run4 featured the lowest average SVI (249). Runs 1 to 3 scored 283, 280 and 276 respectively. Although average values of SVI are high enough to consider sludge as bulking, there were no settling issues in clarification tanks during the reported period. Example of 3 hours SV graph (**Figure 3-6**) from middle of experiment period actually shows how trends are similar during the 30 first minutes of settling test. Settling rate increased in Run4 faster than in Runs 2&3 while it remained almost constant in Run1.

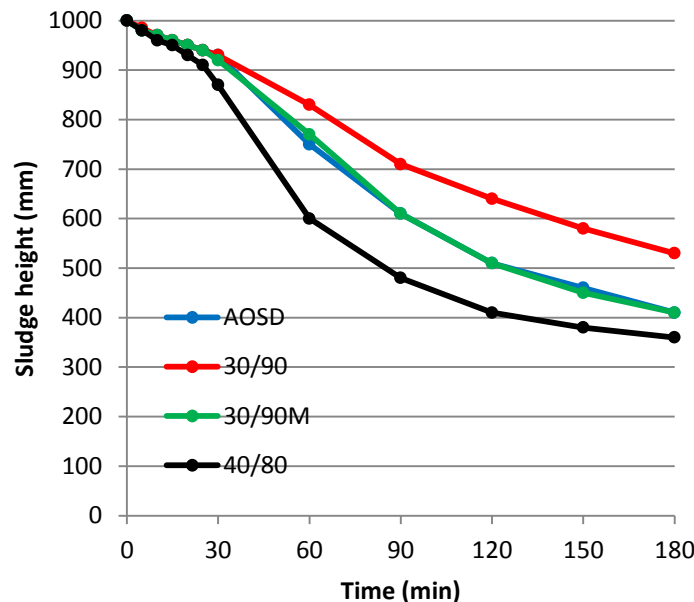


Figure 3-6 Settleability test

Note that settling test was performed according to standard method (APHA, 1995) except that cylinders were not equipped with stirring. This may have resulted in biased SVI values. It is also important to remark that, in the long term, sludge inevitably tended to become fluffy and poorly settling with 30/90 (Run1). This was

believed to be mostly related to the previously mentioned seasonally occurring long periods of low DO saturation.

- **Sludge conversion ratio**

Average sludge conversion ratios for Runs 1 to 4 were respectively of 0.61, 0.57, 0.59 and 0.85. Results in Run1 could be explained by the fact that hypoxia restrains microorganisms to endogenous respiration rather than growth (Liu and Tay, 2001). Wang et al. (2009), on the other hand stated that increasing aeration causes higher endogenous mass loss pointing though that SRT is an important factor influencing specific substances use rates. Low SCR with 60/60 but also difficulties to keep MLSS at $3000 \text{ mg}\cdot\text{L}^{-1}$ with continuous aeration in preliminary experiments confirm that sludge reduction is also increased in conditions of over aeration. The 40/80 cycle resulted in higher SCR. It might be explained by comfortable conditions of proliferation for microorganisms. Lower aeration time provided by AOSD, compared to 40/80, resulted in SCR very comparable to those obtained in conditions of 30/90 (hypoxia) and 60/60 (excess of aeration). Jung et al. (2006) reported also that shorter cycles of intermittent aeration systems tend to achieve higher sludge reduction, which comforts the choice of 2 hours cycle as a good mean to reduce excess sludge disposal costs.

- **Activated sludge biota characteristics**

Protozoa and micro-metazoa, having specific habitat conditions and sensitivity to medium changes, can help determine the ecosystem's condition and evolution. Regular biota enumeration is therefore a relatively quick and useful tool of wastewater treatment plant diagnosis (Canler et al., 2011). The populations of main microorganisms were periodically (every fortnight) estimated in this experiment. A sample inventory of identified genera and their respective count range in a period of two months is shown in **Table 3-3**, pictures and main characteristics of some of which are presented in **Annex C**. The *Arcella sp.* protozoon for example is a bio-indicator of low influent strength or sufficient aeration time and good nitrification performance. Its

presence in higher average numbers with AOSD and 40/80 is confirmed by nitrification rates achieved. Presence of important peritrich species total population, in reactor controlled by AOSD, is consistent with enhanced sludge reduction and low turbidity. Identification and enumeration are difficult. Interpretation of results is also tricky due to interactions between species and combined effects of environment variations as well as influent biotic load.

Table 3-3 Biota enumeration in the experiment Runs

Phylum	Genus	Estimated population (N•mL ⁻¹)			
		AOSD	30/90	30/90M	40/80
Rotifers	<i>Philodina</i>	60-120	20-100	0-120	40-80
	<i>Cephalodella</i>	60-240	0-60	0-40	20-240
Polychaetes	<i>Aeolosoma</i>	0-10			
	<i>Nais</i>	0-6		0-20	0-6
Tardigrades	<i>Macrobiotus</i>		0-20		
Nematodes	<i>Nematoda</i>	40-120	120-240	20-120	20-40
Peritrich	<i>Epistylis</i>	1500-2640	800-5400	840-6360	120-1960
	<i>Carchesium</i>	480-3920	240-1200	300-1680	540-2880
	<i>Vorticella</i>	1920-9600	1920-10680	1560-10320	720-1560
	<i>Opercularia</i>	480-1000	120-180	80-360	140-240
Suctorian	<i>Tokophrya</i>	0-240		0-120	
Hypotrich	<i>Aspidisca</i>	720-3960	240-1080	120-600	1080-2040
	<i>Euplotes</i>	20-80	60-120	0-20	40-360
Amoeba	<i>Arcella (S)</i>	1320-6000	240-1080	120-2160	4920-10320
	<i>Arcella (L)</i>	0-20			0-20
	<i>Euglypha</i>		0-120		
Swimming ciliates	<i>Colpidium</i>	760-1032	1080-4080	360-4800	4560-16800
	<i>Spirostomum</i>	480-1080	480-960	120-720	360-720
	<i>Trachelophyllum</i>	120-240		0-120	0-120
Flagellates	<i>Bodo</i>	0-120	0-120		0-120
	<i>Peranema</i>	0-120			

The AOSD system, by adjusting aeration time to the needs, allowed growth of a large variety of desired microorganisms and in high average density of populations,

which indicates optimal conditions for diversity and sustainability of food chain implying stability of the ecosystem.

3.3.4 Energy saving assessment

In this part, considering that power of electric devices (air blowers, stirrers and pumps) used in the experiment was oversized and that many factors should be taken in account in real scale conditions related to the plant design (energy losses due to resistivity and transmission, penalties on contract power overrun and $\cos\phi$, possibilities to adjust functioning to low tariff time periods, etc.), evaluation was made only in terms of air blowers' operation time and in terms of pollutants removal efficiency (**Table 3-4**). The latter was calculated by dividing total average daily removal to total daily volume of air delivered in each Run.

Table 3-4 Pollutants removed in proportion of aeration volume

Run	Units of pollutant removed per unit of air volume delivered			
	BOD (mg•L ⁻¹)	COD (mg•L ⁻¹)	TN (mg•L ⁻¹)	TP (mg•L ⁻¹)
AOSD	28.39	21.58	5.89	0.30
30/90	27.55	22.52	5.28	0.32
30/90M	30.87	22.86	6.38	0.30
40/80	23.72	17.24	4.14	0.24

AOSD saved an average of 20% aeration operation energy compared to 40/80 system, and utilized each liter of air with higher efficiency. Attempt to save 25% aeration time in Run1 showed signs of hypoxia. Performance of 30 min aeration could be improved though in this experiment by effect of media added in reactor of Run2. These results prove the ability of AOSD to compensate for O₂ transfer deficiency (observed between 30/90 and 30/90M) by increasing aeration time just enough to obtain good effluent quality (similar to 30/90M, better than the respectively under and

over aerated Runs 1 & 4). It also prevents energy waste by benefitting from O₂ reclaim during denitrification process and avoiding over aeration.

3.4 Conclusion

All aeration control settings tested during this experiment had good pH conditions and complied with effluent discharge range that usually varies between 6.5 and 8.5. Patterns of DO are affected by influent load (although volume and strength are considered constant, fluctuations were inevitable) but also by microorganisms' selection (due to DO level itself and to aeration time) and their populations (effect of aeration, food and pH on growth rates and acclimatization as well as biotic composition of influent). No significant difference between AOSD, 30/90M and 40/80, and little variation during reported period was observed with effluent COD_{Mn}. In Run1 (30/90 system), level was higher as a sign of insufficient aeration. Effect of media and the average two supplementary minutes added by AOSD system were sufficient to cope for hypoxia to obtain similar results to those of 40/80. These results imply that BOD₅ values particularly high in 30/90 Run are mostly NBOD, a conclusion correlated by NH₄-N concentrations in each Run. Conditions provided by AOSD and 30/90M were propitious for better TN removal and target was achieved. In 40/80, despite longer aeration time, hardly better nitrification was achieved and higher TN was due to accumulation of NO₃-N caused by poor denitrification. On the other hand, insufficient O₂ transfer in 30/90 system limited nitrification. Aeration time seems to be a determinant factor of sludge settleability. It also has an important effect on biota composition. Diversity of protozoa and metazoan and dense populations of desired ones in Run controlled by AOSD indicate healthy stable sludge.

In this chapter, results show that optimum aeration time in ratio to total cycle duration is important for TN removal as it provides conditions for aerobes and anaerobes to coexist without impairing their activity rate. Required volume of aeration being a function of oxygen transfer efficiency, flow rate and time, it is crucial to

improve O₂ dissolution but as it is difficult to control and adapt to wastewater quality and flow rate fluctuations, AOSD system's flexibility can palliate to those variations and keep biota in stable conditions of DO and pH. However, judicious choice of AOSD settings, hydraulic parameters and targeted performance is necessary to ensure successful treatment. Fine tuning of operation parameters and improvement of O₂ transfer could highly improve effluent quality and help save more energy.

Chapter 4: Effect on pH and gases emissions in correlation with cyclic variations of nutrients' concentration

4.1 Introduction

Experiment undertaken in previous chapter showed that changing aeration time by few minutes influenced DO profiles and effluent quality. As DO and pH are important factors in the TN removal process, their bending points are used along with ORP's as control parameters in many systems. Variations of these two factors during an aeration-anoxia cycle and in the long term could be of interest in shading more light on phenomena occurring in each Run. Including pH to control parameters of AOSD might also be an asset. Another experiment setting was therefore conducted after acquisition of more LDO probes and pH probes for simultaneous and continuous monitoring of the two parameters in all Runs. Factors of pH variation were investigated and led to necessity of analyzing organic acids and possible involvement of CO₂ in the phenomenon. This in turn brought Greenhouse gases emissions, an important parameter of pollution and a factor beside energy saving, in assessing AOSD system as an environment friendly method. Their production was therefore also studied in this experimental setting. Emission of GHG being closely related to nutrients concentrations (Kimochi et al., 1998), their cyclic pattern was investigated concomitantly with GHG's for a better understanding of aeration time control system effect on the nitrification-denitrification process and GHG emissions in each Run.

4.2 Materials and methods

4.2.1 Experimental setting

After removing media from Run2, its aeration conditions were changed from 30/90 to 60/60. Addition of a blower was therefore necessary. Both DO and pH were

continuously monitored by installing DO meters and pH meters in all Runs. A supplementary PLC was necessary to process DO and pH data (**Figure 4-1**). AOSD software was also modified. It included logging and display of DO and pH for all Runs, as well as an improvement related to aeration time calculation according to observations from results obtained in the first experiment setting.

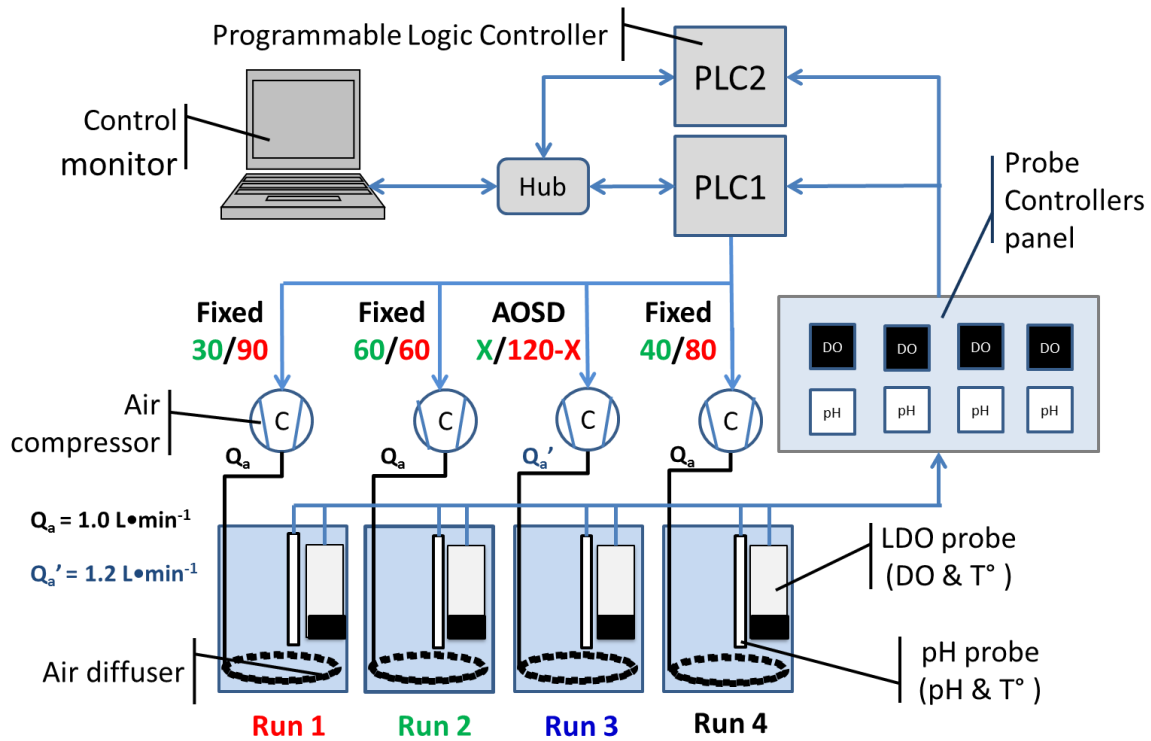


Figure 4-1 Second Experiment setting

After a period of setting this experiment, abnormally high BOD and TN were observed in Run3 controlled by AOSD despite higher aeration time especially compared to former results and to 30/90 performance. A control of the diffuser lead to finding a slash caused by stirrer's fan to be at the origin of low O_2 transfer and thus the lower effluent quality (**Picture 4-1**). Those results were therefore discarded. Meanwhile, both 30/90 and 40/80's DO plateau level dropped to less than $1.0 \text{ mg}\cdot\text{L}^{-1}$. This sudden variation in DO pattern had already been observed at season's weather change. It was, along with the optimal air flow rate value of $1.15 \text{ L}\cdot\text{min}^{-1}$ calculated using simulation software made by AOSD system's developers, the main reasons for

air flow rate increase to $1.2 \text{ L}\cdot\text{min}^{-1}$ in Run3. It was an opportunity to study effect of air flow rate on DO level and calculated aeration time as well as benefit from its control.



Picture 4-1 Aeration defect in Run3 controlled by AOSD

During one of the weekly cleanings, while scrubbing walls of the tank, position of diffuser was probably moved close to stirrer causing a slash in the diffuser. Left picture shows that all the air was exhausting from that slash. Picture on the right shows how easily torn the material of diffuser is as the small initial slash was aggravated in the process of removing diffuser to change it.

Data of DO and pH in each run were stored in the Microsoft Access file of AOSD. Scatters shown below were plotted after selection of date interval using Access query, importing data to Excel file and processing them using macros (automation of sorting, determination of minimum, maximum and average of each cycle, etc.)

4.2.2 Sampling and analysis of greenhouse gases and nutrients

To measure dissolved GHGs in each Run, a syringe was used to take 30 mL of mixed liquor from reactor to which a 30 mL volume of N_2 gas was added. The syringe was shaken for around 1min and the gas phase injected in both ECD and FID Shimadzu gas chromatographs. First one was used for N_2O analysis while second measured CH_4 and CO_2 . The two machines were calibrated prior to starting sampling.

Sampling was made every 10 min during aeration time and every 20min for the rest of the 2 hours cycle.

Emitted gases sampling was done using a small plastic box at the bottom of which a hole was made to fix a tube connection. Box was then put upside down on the surface of mixed liquor in the reactor and fixed to its wall. A small compressor was used to fill aluminum bags with gas stripping from reactor into the box. Gas samples were then taken for Gas Chromatography analysis. Samples for emitted gases were taken every 10 min during aeration time. Gas emissions being very feeble during anoxic period, tube was clipped to accumulate gas in the box and just before start of new cycle's aeration time, box was pushed down in mixed liquor to pressurize gas it contained and collect it in aluminum bag with the help of compressor for the last sample that actually represents cumulated emitted gases of anoxic phase.

Concomitantly with sampling of dissolved and emitted gas, every 10 min a volume of 2 mL was taken from reactor and filtered using chromatography syringes and filters. A filtrate volume of 1 mL was then diluted 10-fold with deionized water and submitted for nutrients analysis.

Weekly effluent quality analyses were performed, as described in previous chapter, to which TOC and COD_{Cr} analyses were added to confirm that high BOD was mostly due to NBOD. To measure COD_{Cr}, low COD (0-150 mg•L⁻¹) HACH test kits HACH digester and HACH DR 2800 spectrophotometer were used. As for TOC, a SHIMADZU Organic Carbon Analyzer was used.

4.3 Results and discussion

4.3.1 Monitoring of dissolved oxygen and pH

Dissolved oxygen and pH were continuously monitored in the four runs. This setting could help observe variations in DO level and pH, and eventually correlate them to effluent performance or to upsets that may cause important changes in biota.

Unfortunately the volume of data and time necessary to process them require development of appropriate tools to facilitate access to details as well as general trends and make more accurate analysis. However a period of 3 days was randomly picked and represented in **Figure 4-2**.

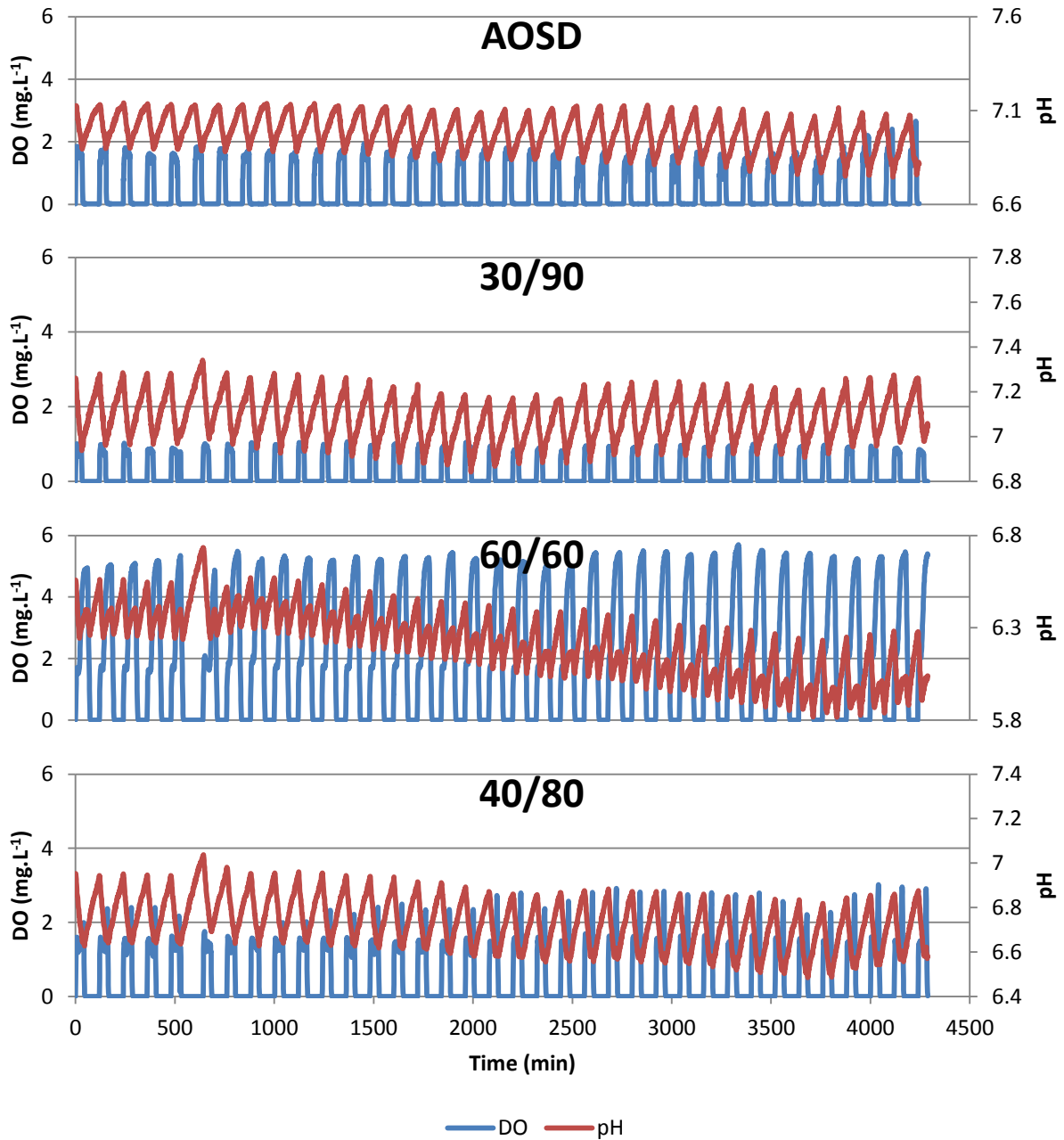


Figure 4-2 Evolution of DO and pH in time for a period of 3 days.

Both parameters varied with time. These fluctuations may be explained by MLSS variations (due to sludge production and periodical excess sludge withdrawal), influent strength (which despite standardization and continuous mixing in storage tank is prone to slight changes) and ratios of readily oxidizable organic compounds, as well as microorganisms' populations fluctuations in reactor due not only to intensive selection but also to contamination from influent.

During this period, DO plateau of AOSD was at around $1.8 \text{ mg}\cdot\text{L}^{-1}$ and around $1.0 \text{ mg}\cdot\text{L}^{-1}$ for 30/90. In 40/80 plateau was at similar level with AOSD but DO profile featured a peak increasing up to $3.5 \text{ mg}\cdot\text{L}^{-1}$ showing over aeration. The DO plateau of 60/60 was very short also around $1.8 \text{ mg}\cdot\text{L}^{-1}$ but DO quickly increased to almost $6 \text{ mg}\cdot\text{L}^{-1}$. Aeration time clearly affects DO profile but degree of air flow rate effect could not be properly evaluated under this experiment setup. Little change in the trend could be observed in such short period especially for plateau level. It seems though that AOSD with aeration time closer to the 30/90 system managed though to keep higher DO plateau.

Responses of pH are comparable to those of DO. Longer aeration caused higher variation in pH during an aeration-anoxia cycle and decrease of average value. AOSD showed though very stable pH and little variations.

Visualization of pH variations on longer period would be useful in understanding the DO-pH-biota interactions. Maxima, minima and means of pH for each cycle during 10 days including the period above cited are represented in scatters of **Figure 4-3**. Although variations between two consecutive cycles or even days may be very small in the absolute; a general view of scatters allows us to better see the trends; periods of stability and time necessary to recover from upsets. Important variations in a short time occurring simultaneously in all Runs or in a particular one may indicate technical problems and help in diagnostic for quick remediation.

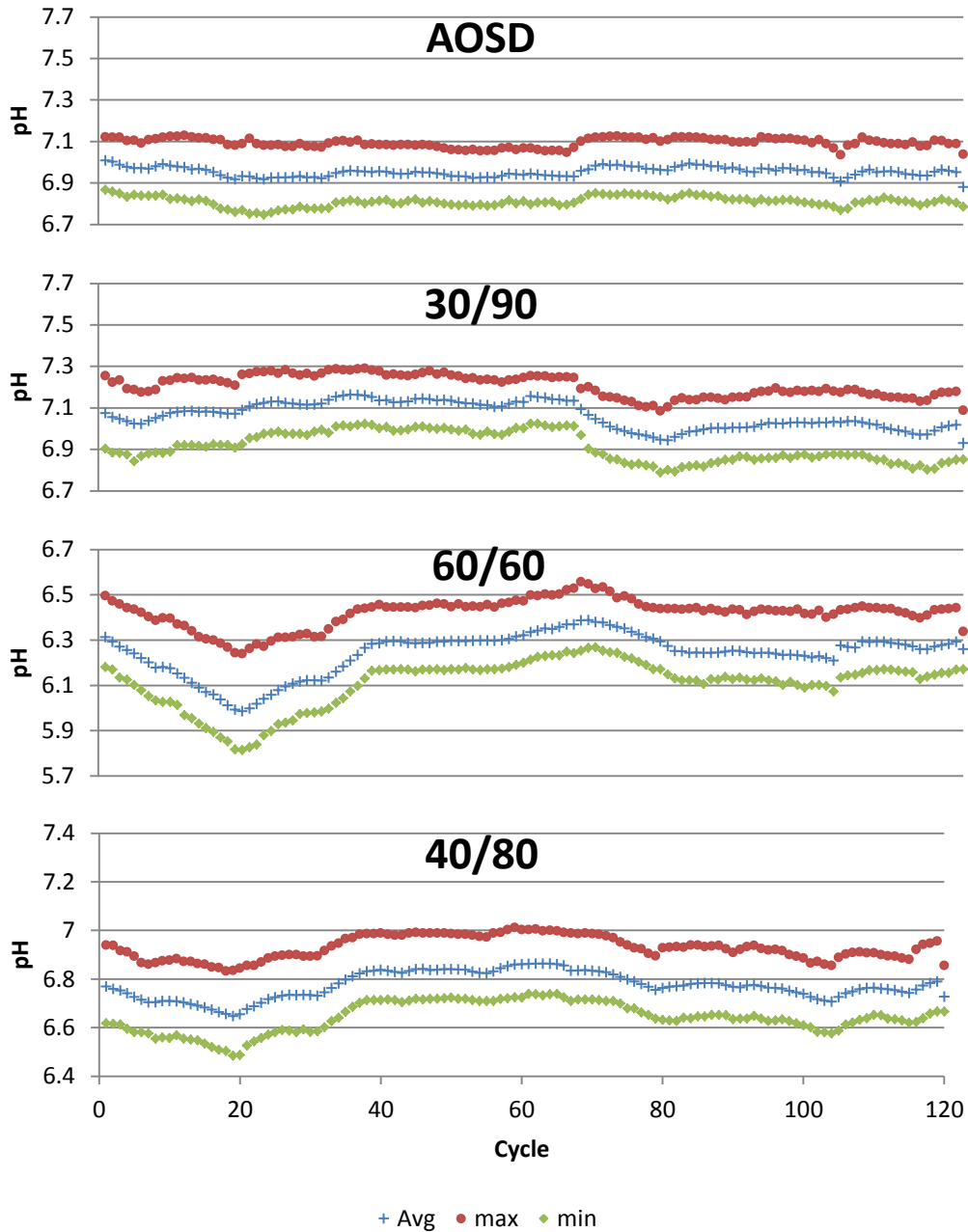


Figure 4-3 Cyclic variations of pH during a period of 10days

For example; a decrease in pH can be observed in Runs 1, 2 & 4 around cycle 66 indicating an event that affected all runs. With AOSD system, on the contrary, scatter showed at the same moment a slight increase of pH. This odd behavior could be explained by checking aeration time of those specific cycles. In response to the upset (origin could not be exactly determined, but most probably attributed to a change in

influent quality), aeration time decreased from 32 min to 28 min and stabilized at 30 min for few cycles.

This experiment setting as well as previous results clearly correlated longer aeration time to lower average pH (**Figure 4-4**). It also showed that longer aeration time caused higher range of pH fluctuation within a cycle. In this regard, AOSD provided more stability while 60/60 system had the higher fluctuations.

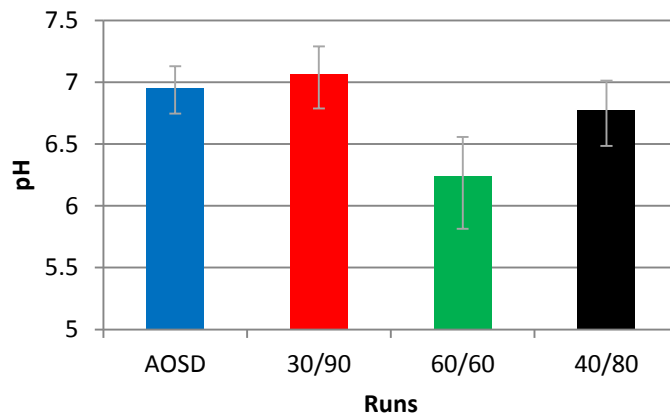


Figure 4-4 Average pH and amplitude

One of the most important observations was the response of AOSD and its ability to cope with sudden variations by adjusting aeration time to keep stability in the reactor. In an attempt to better understand these pH variations a closer look at a single aeration-anoxia cycle.

Bending points of pH have been used along with ORP and DO to control aeration in intermittent systems. Nevertheless no document to our best knowledge explained in detail reasons of pH variations. Represented in **Figure 4-5**, is an example of DO profiles with pH variations in each Run.

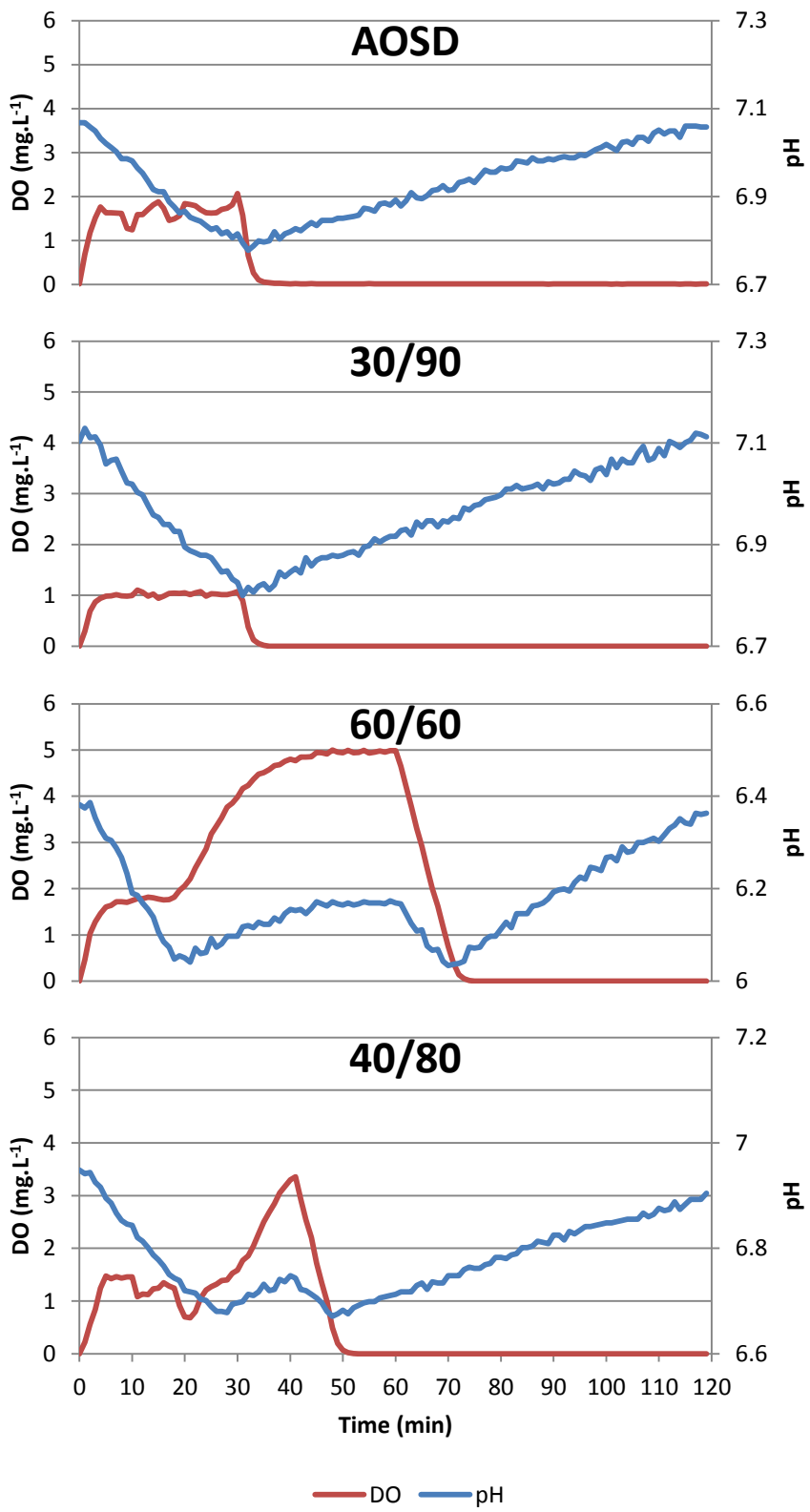


Figure 4-5 Cyclic pattern of DO and pH

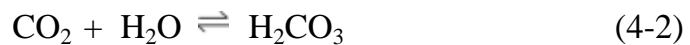
As soon as aeration is stopped, steep decrease of DO and slow increase of pH occur in Runs 1&3. Runs 2 & 4 feature elbows in both DO and pH before end of aeration time. These are caused by depletion of ammonia (Won and Ra, 2011) as a result of nitrification which releases protons according to the following reaction (Lin et al., 2009):



Presence of simultaneous increase in DO and pH indicates over aeration. The small sketch of peak featured in AOSD pattern informs that aeration was cut at the exact moment nitrification ended.

The DO profile of 40/80 system gives a good example of situations in which control systems relying on DO bending points may fail in detecting end of nitrification point, requiring therefore another control parameter (ORP, pH or both).

It was observed that a small peak of pH concomitant with DO peak systematically appeared in the over aerated runs. (Won and Ra, 2011) emitted the hypothesis of CO₂ being stripped in the air Dissolved CO₂ is in chemical equilibrium in water and produces carbonic acid:



In this research, both dissolved CO₂ and organic acids concentrations were considered in order to explain the phenomenon. Results of Gas Chromatography for VFA did not yield any consistent correlation. On the other hand, measurement of DCO₂ perfectly fit with DO and pH patterns (**Figure 4-6**).

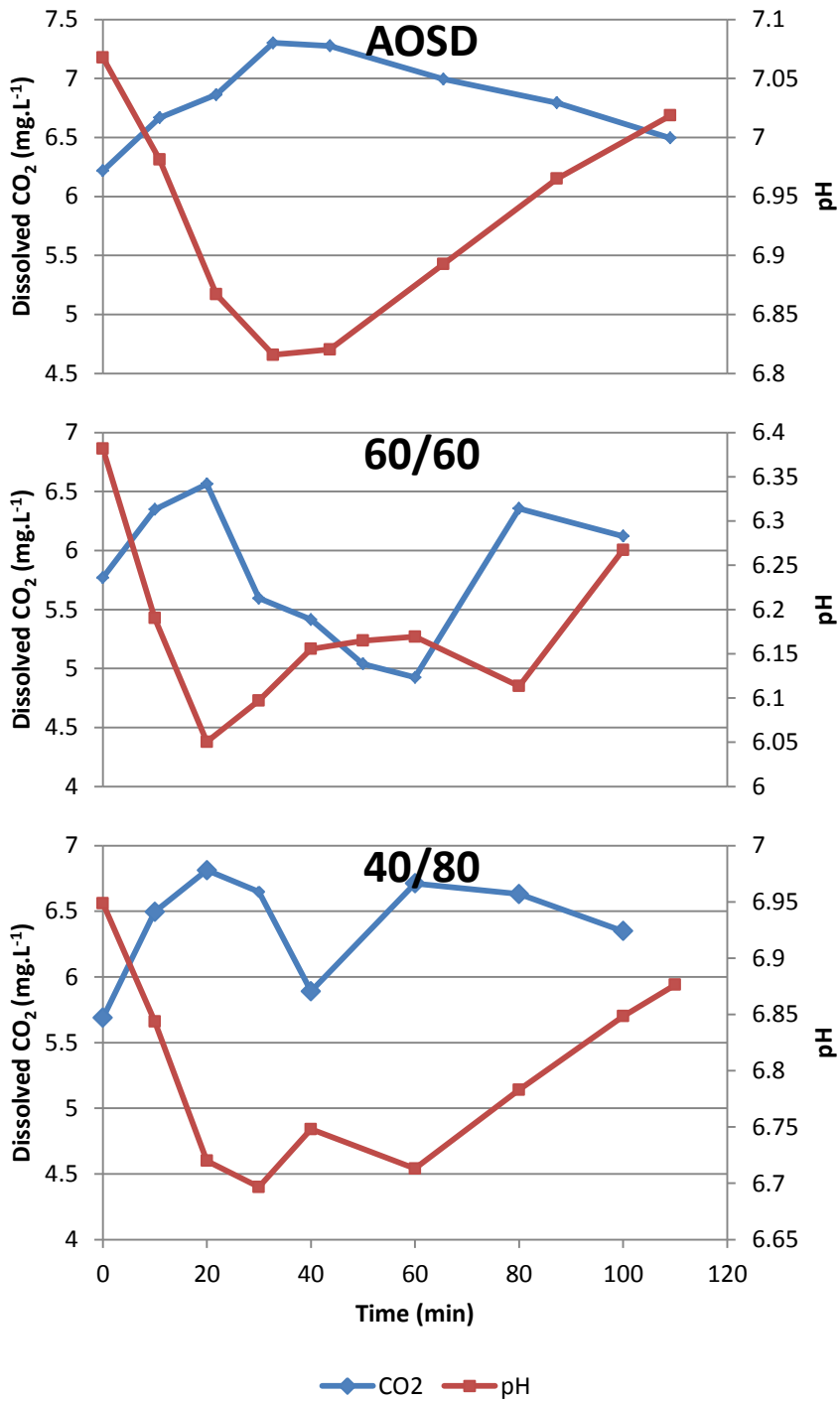


Figure 4-6 Dissolved CO₂ in plot versus pH

Dissolved CO₂ increase during aeration time is due to organic matter oxidation. Rate is higher with over aerated runs 60/60 and 40/80 as conditions are more propitious for aerobic microorganisms growth (higher DO levels and shorter anaerobic time). After

NH₄-N depletion, DO partial pressure increased stripping CO₂ into the atmosphere. With AOSD system, as DO did not exceed 2 mg•L⁻¹ and aeration stopped at optimal time, DCO₂ reached higher levels. During first period of anoxic phase (presence of DO but aeration stopped), DCO₂ increased again under effect of CO₂ production and DO consumption. This period being very short in Run3, no DCO₂ increase was detected. Anaerobic phase was characterized by DCO₂ decrease at seemingly similar rate in all runs.

In an aeration/anoxia cycle, pH decreases during aeration under combined effect of nitrification and DCO₂ buildup. Continuing aeration after nitrification ended, leads to DCO₂ stripping at higher rate than it is produced, causing regain in pH. Decrease of DO restores DCO₂ and thus, affects pH until start of anaerobic phase where pH increases again due to alkalinity from influent and DCO₂ lessening. Including pH changes in AOSD system control parameters could yield higher performance if best ranges of DO levels, aeration time and pH conditions are accurately determined.

4.3.2 Greenhouse gases production

Involvement of CO₂ in pH variations naturally raises the concern about not only CO₂ but also other GHGs production and emission as a part of AOSD system performance assessment.

Dissolved and emitted gases were analyzed by gas chromatography to determine concentrations of N₂O, CH₄ and CO₂. Examples of results obtained are reported in **Figure 4-7**, **Figure 4-8** and **Figure 4-9**, respectively.

- ***Nitrous oxide (N₂O)***

The over aerated Run2 registered higher dissolved and emitted N₂O. Both appeared with beginning of aeration phase. Dissolved N₂O peaked around 5 ppm while emission exceeded dissolved from the 15th min of aeration to reach as high as 8 ppm. Quick buildup of N₂O could be explained by high DO that causes unbalanced activity of AOB and NOB. Accumulation of NO₂-N favors a process called nitrifier-

denitrification by AOB themselves believed to be responsible of most emitted N_2O by activated sludge (Mello et al., 2013). In high concentrations NO_2-N inhibits both types of bacteria (AOB and NOB) jeopardizing nitrification.

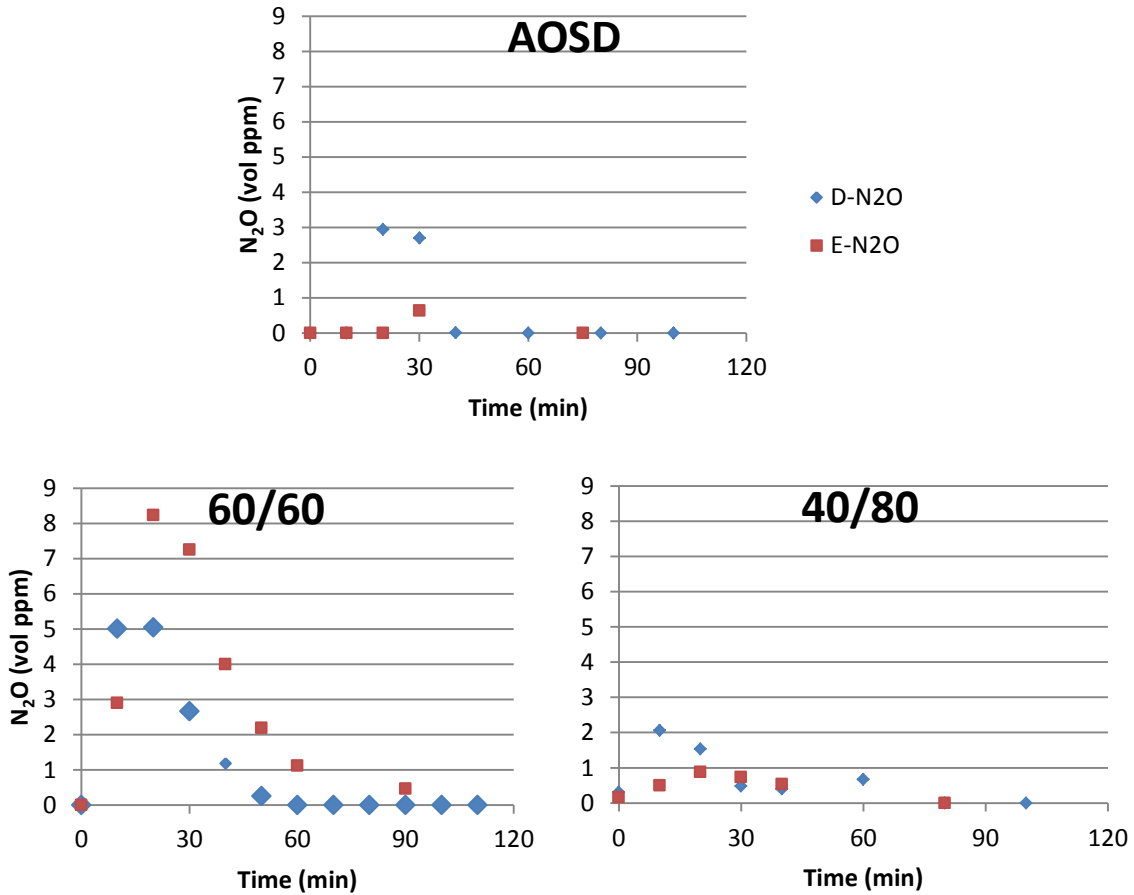


Figure 4-7 Dissolved and emitted N_2O

Production of N_2O is caused either by, low DO during nitrification, increase in NO_2-N concentration during nitrification or denitrification, or by a disequilibrium in COD/N ratio during denitrification (Kampschreur et al., 2009).

In Run controlled by 60/60 system, both dissolved and emitted N_2O peaked during aeration time at respectively 5 and 8 ppm but were hardly detected during anoxic period. Over aeration, and consequently short anaerobic phase (which starts only around 80th minute of the cycle), may cause deficit in easily degradable organic source

for denitrifying bacteria, leading to $\text{NO}_2\text{-N}$ accumulation around end of cycle and thus production of N_2O . This explanation is supported by experiments conducted by Park et al. (2000).

With AOSD and 40/80, both dissolved and emitted N_2O levels were very low especially considering that air of the room (used by blowers to aerate the reactors) already contains N_2O produced by this experiment or by other experiments of the shared space (a concentration of 0.6ppm was measured in a sample of air taken near blowers). Emission of N_2O from Runs controlled by AOSD and 40/80 may therefore be considered negligible. Precise measurement would require purified air to discard possible noise.

From these results, it was concluded that higher DO levels increase N_2O buildup and longer aeration times lead to bigger emission volumes. The AOSD system can help control N_2O emission a gas with over 300 times more impact per unit mass in global warming potential than CO_2 .

- ***Methane***

Methane is a byproduct of fermentation with very low solubility in water (with a Bunsen's absorption coefficient of 0.0331mL/mL at 20°C). Results reported in **Figure 4-8** show indeed that CH_4 is easily stripped in atmosphere during aeration. Dissolved CH_4 drastically dropped from 230 ppm to less than 10 ppm. First point in 40/80 actually measured with very small delay shows how fast CH_4 decreased and its effect on accuracy. Increase of dissolved CH_4 started as soon as aeration was stopped in Runs controlled by AOSD and 40/80 at comparable rate leading to higher concentration with AOSD as anaerobic phase is longer. In 60/60 Run on the other hand, CH_4 buildup started after 80 min due to slow depletion of DO. Note that sample of ambient air contained around 10 ppm of CH_4 .

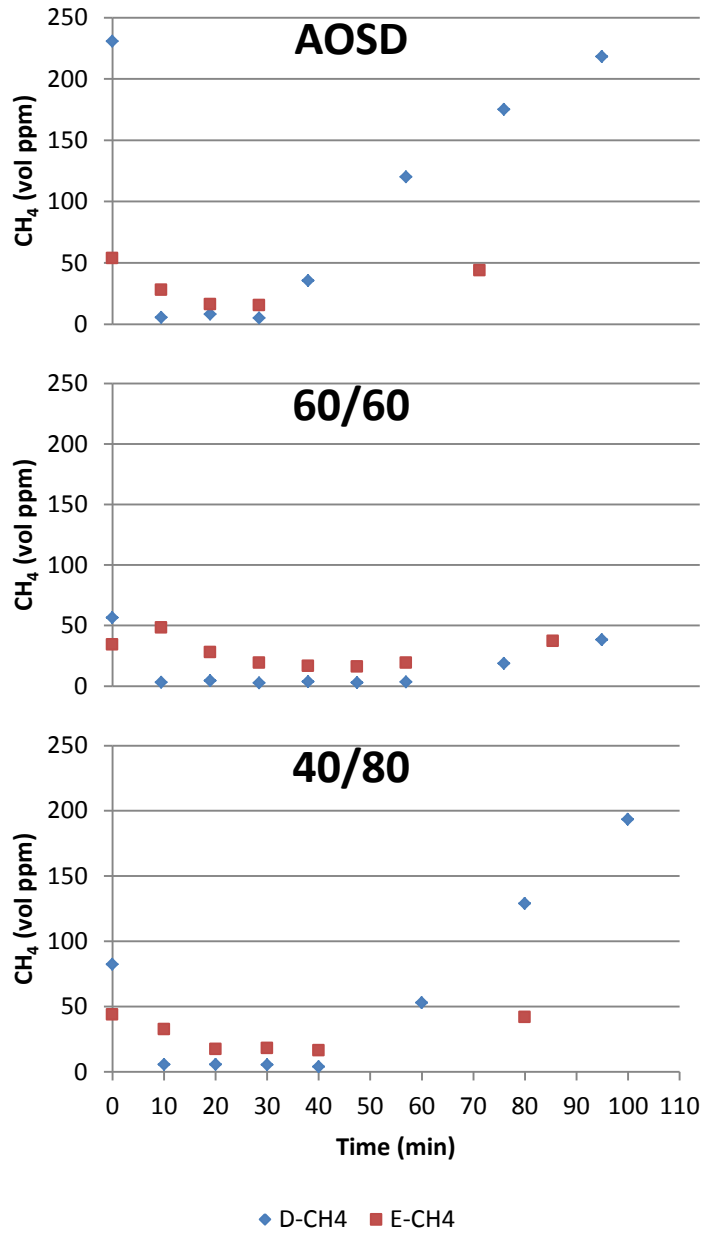


Figure 4-8 Dissolved and emitted CH₄

Increase of dissolved CH₄ in 40/80 and AOSD runs during anoxia is a sign of adequate conditions for anaerobic microorganisms' activity. Despite higher production in those runs compared to 60/60, emission is lower with shorter aeration time systems.

- **Carbon dioxide**

According to **Figure 4-9**, dissolved CO₂ is higher in Run3 controlled by AOSD compared to 60/60 and 40/80 systems with concentration ranging between 6.2 and 7.3 mgC.L⁻¹. Nevertheless, emission rate is higher with 60/60 and lower with AOSD. Moreover, considering aeration time, it appears that total volume of emitted CO₂ is in 60/60 more than 4 times that of AOSD. Sample of ambient air contained 580 ppm of CO₂.

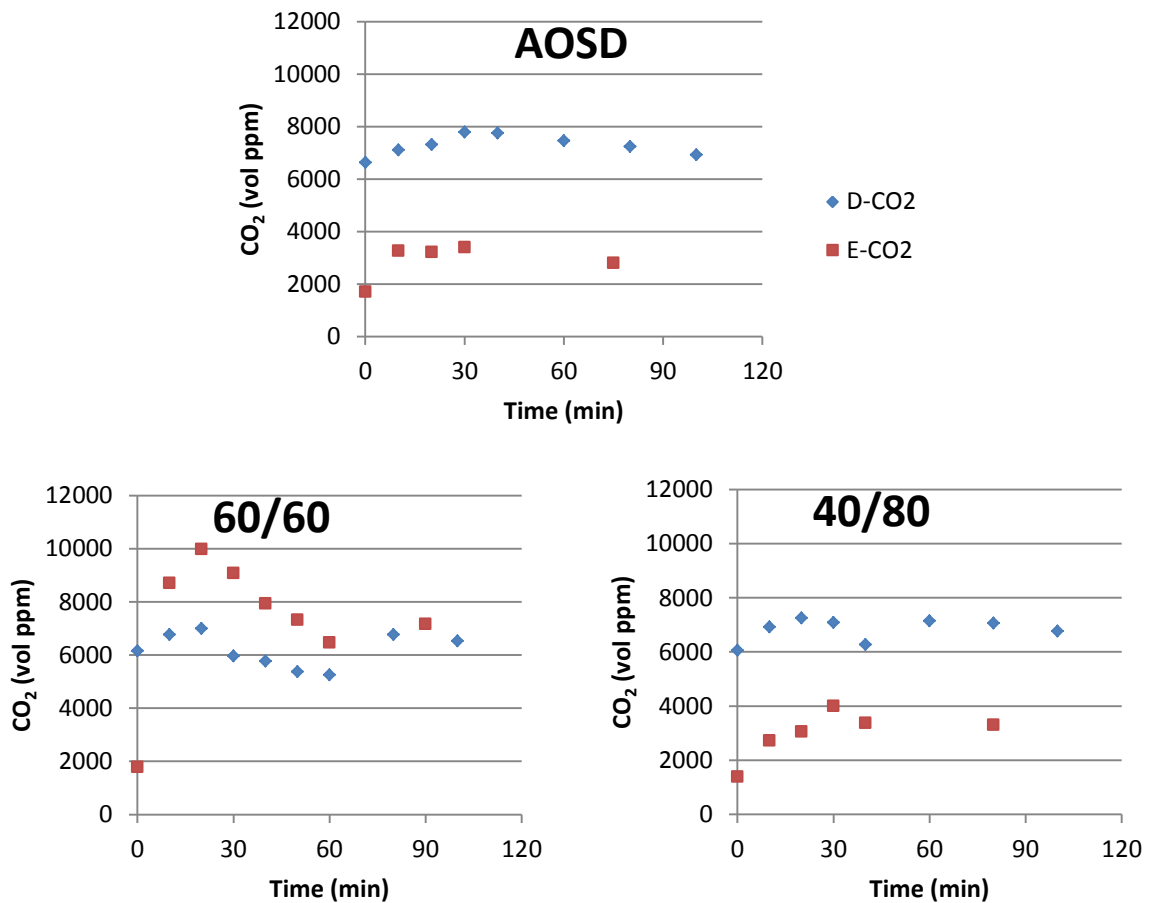


Figure 4-9 Dissolved and emitted CO₂

Estimation of GHG emissions from the three Runs is reported in **Table 4-1**. These roughly calculated values serve only the purpose of comparing between the Runs as blown air's contribution and emissions during anoxic phase were neglected. Emitted volume of each GHG for each Run was first calculated by estimated integration of

area delimited by their respective plots of concentration versus time during aeration period multiplied by air flow rate. Emitted mass was then calculated using molar mass of each molecule and the ideal gases law. Considering the 100 year Global Warming Potential (GWP_{100}) of 310-fold and 21-fold, respectively for N_2O and CH_4 compared with CO_2 (IPCC, 2007), total CO_2 mass-equivalent emitted by each run was calculated.

Table 4-1 Estimated GHG emissions during one cycle aeration period

GHG	AOSD	60/60	40/80
N_2O (μg)	14.01	470.18	50.18
CH_4 (mg)	0.69	1.10	0.71
CO_2 (mg)	235.34	922.34	253.83
Total CO_2 equivalent (mg)	254.25	1091.27	284.28

The table clearly shows that length of aeration time is a determinant factor of GHG emissions. Under the studied conditions, 60/60 system induced production of very high amounts of N_2O and CO_2 compared to the other Runs. The optimal balance in microbial activity provided by AOSD is an asset for reduction of GHG emissions by AAA activated sludge processes.

4.3.3 Cyclic evolution of nutrients in reactors

In order to better picture DO level and aeration time effects on treatment performance, samples were taken for nutrients analyses simultaneously with those of dissolved and emitted gases (**Figure 4-10**). From trends and concentrations it appears clearly that same reactions are occurring at very different rates between Runs.

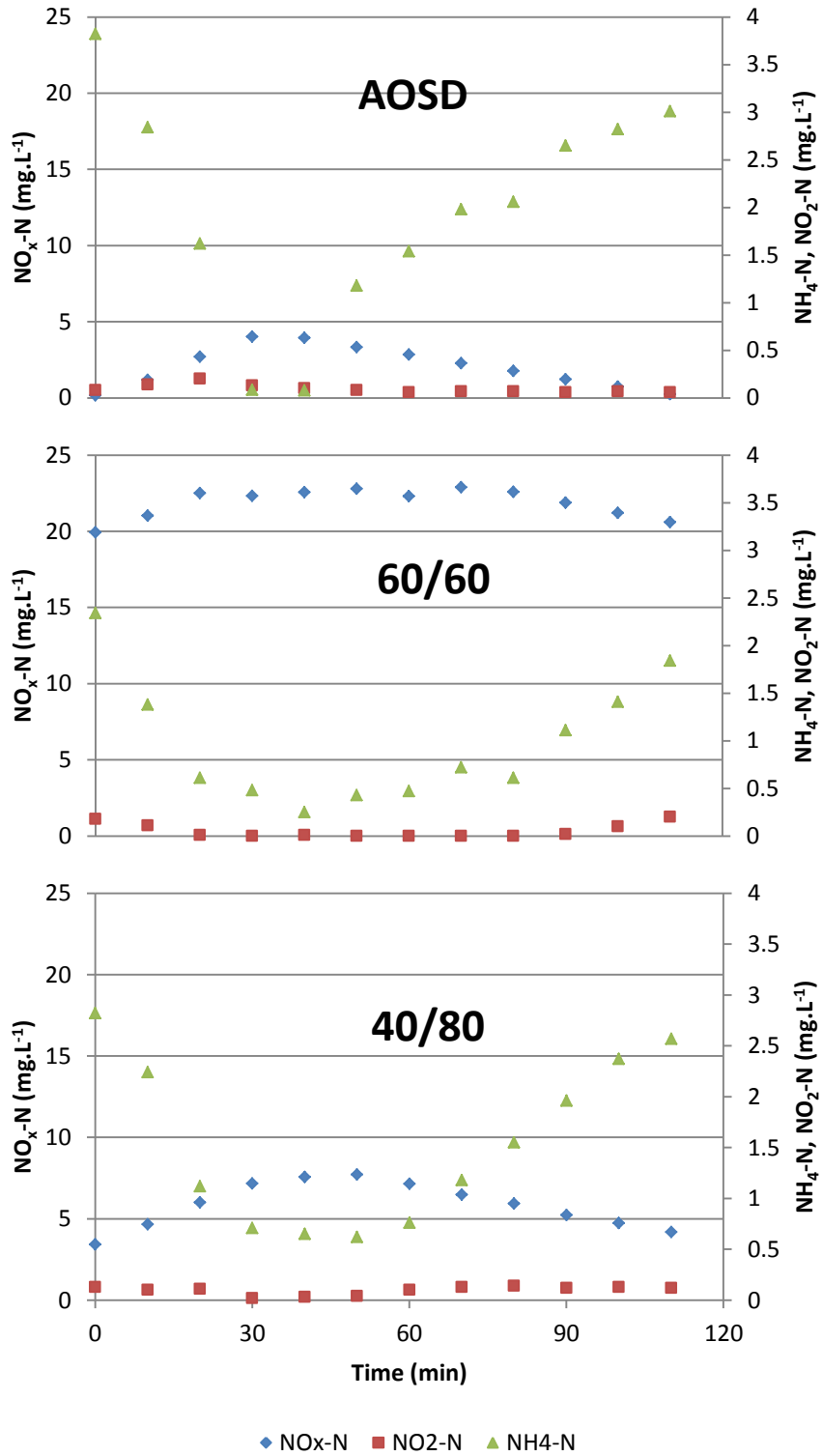


Figure 4-10 Concentrations of Nitrogen nutrients during one cycle

- **Nitrogens**

Although 40/80 featured a DO bending point usually considered as indicator of $\text{NH}_4\text{-N}$ complete depletion, it appeared that $\text{NH}_4\text{-N}$ oxidation reached a threshold of around $0.6 \text{ mg}\cdot\text{L}^{-1}$ ammonia remaining concentration, and stabilized at that level for almost 30 min. This suggests that AOB activity greatly decreased, until it met influent load rate, after 30th min of aeration, and explains the DO bending point. Considering $\text{NO}_2\text{-N}$ levels, undetectable level of N_2O , absence of limitations in DO or CO_2 , no clear justification could be given to this phenomenon except perhaps that major strains responsible for nitrification in that reactor have lower activity beyond $2 \text{ mg}\cdot\text{L}^{-1}$ DO. Concentrations of $\text{NH}_4\text{-N}$ did not start to increase at steady rate until anaerobia completely took place. As for $\text{NO}_2\text{-N}$, it seems to increase only during denitrification period and aeration phase actually helps decrease its concentration as NOB are activated. High levels of $\text{NO}_x\text{-N}$ (mostly $\text{NO}_3\text{-N}$) denote the slow rate of denitrification which, combined to around 70 min anaerobic period, leads to less Nitrogen removal in the form of N_2 gas. Nitrates ranged therefore between 3.4 and $7.7 \text{ mg}\cdot\text{L}^{-1}$.

Same description goes with 60/60 system in terms of trend. Difference resides in shorter anaerobic phase (45 min) hindering accumulation of $\text{NH}_4\text{-N}$ but also further denitrification. Major disparity was with $\text{NO}_x\text{-N}$ as scatter trend shows lower denitrification rate in addition to shorter decrease period. This lead to a fourfold concentration of $\text{NO}_x\text{-N}$ compared to 40/80.

AOSD system provided what could be considered as very good conditions for nitrogen removal. Indeed, in Run3, nitrification occurred until almost complete depletion of $\text{NH}_4\text{-N}$ (less than $0.1 \text{ mg}\cdot\text{L}^{-1}$) exactly around aeration stop time. Oxidation of $\text{NH}_4\text{-N}$ occurred at higher rate compared to other Runs as it dropped from $3.8 \text{ mg}\cdot\text{L}^{-1}$ to traces within 35 min. Little accumulation of $\text{NO}_2\text{-N}$ (and little production of N_2O), during aeration phase only, suggests though a slight disparity between AOB and NOB. This can be explained by the relatively recent replacement of

defective diffuser, after a period of bad O₂ transfer that most probably affected biota considering the poor performance during that period, and the modification of air flow rate setting from 1.0 to 1.2L.min⁻¹.

Lower maximum level of NO_x-N compared to that of 40/80 system suggests though another explanation which is occurrence of simultaneous nitrification-denitrification (SND) at least at higher rate with AOSD than with 40/80. Indeed faster rate of NH₄-N decay but lower accumulation of NO₃-N as well as the slight buildup of NO₂-N during aeration hint that part of NO₂-N is directly reduced to stripping N₂. This not yet well understood physical or biological “shunt” in nitrogen removal is considered to spare 25% of O₂ consumed for nitrification (2nd part of nitrification process) during aeration time (Henze, 2002; Zhang and Qi, 2007).

Observation of the scatters and comparison of AOSD with 40/80 and 60/60 results indicate that it would be very difficult to obtain a better nitrogen removal performance. The only possible way to obtain lower TN might be to reduce cycle duration by 10 to 20 min to limit accumulation of NH₄-N or modify shape of reactor (increase distance between influent tube and reactor’s outlet which was only around 25cm) to ensure that part of influent is not directly flowed into settling tank by stirrer’s speed .

- ***Orthophosphates***

Levels of PO₄-P were almost constant during all cycle (**Figure 4-11**). Tiny variations show a trend of very slow increase during anaerobic phase. And oddly, lower concentrations were found in less aerated Runs.

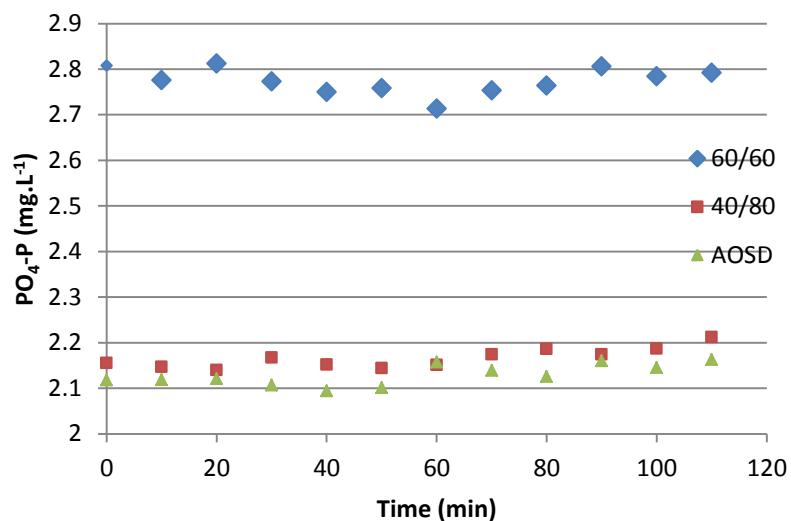


Figure 4-11 Concentrations of orthophosphates during one cycle

4.3.4 Effluent quality and energy saving

Average analysis results of 24 hours samples for a period of 2 months of all four Runs are reported in **Table 4-2**.

Table 4-2 Pollutants removal performance in % of influent

Run	COD _{Cr} (mg•L ⁻¹)	COD _{Mn} (mg•L ⁻¹)	BOD ₅ (mg•L ⁻¹)	TOC (mg•L ⁻¹)	TN (mg•L ⁻¹)	TP (mg•L ⁻¹)	NH ₄ -N (mg•L ⁻¹)
AOSD	91.1	87.5	93.5	88.8	87.1	46.0	95.9
30/90	85.8	83.0	88.8	86.9	83.1	41.4	93.9
60/60	89.2	86.1	95.2	87.6	46.1	36.3	97.0
40/80	91.0	93.6	95.5	87.2	76.7	42.6	96.7

Organic matter removal was very comparable, except for 30/90 which showed lower performance. Discrepancies between Runs in terms of organic matter parameters are mostly due to precision of methods and to types of organic matter they allow to quantify, as COD_{Mn} measurement does not include polymers such as cellulose and TOC represents non purgeable organic carbon (NPOC) while BOD₅ here included NBOD. Removal of NH₄-N was also within close range except for 30/90

again. On the other hand, TN removal was significantly higher with AOSD due to the very good denitrification conditions it provided, while 40/80 and 30/90 achieved similar performance. Run2 operated at 60/60 achieved poor performance (46%) as this aeration/anoxia ratio caused accumulation of $\text{NO}_x\text{-N}$ (21 $\text{mg}\cdot\text{L}^{-1}$ in average). Regarding TP there was no statistically significant difference.

Efficiency of air utilization is reported in **Table 4-3**. As AOSD averaged 31.5 min aeration time at a $1.2 \text{ L}\cdot\text{min}^{-1}$ flow rate, volume blown per cycle was 37.8 L. Air flow rate being kept at $1.0 \text{ L}\cdot\text{min}^{-1}$ in Runs 1, 2&4, aeration volumes were respectively 30, 60 and 40 L per cycle.

Table 4-3 Pollutants removed in proportion of aeration volume

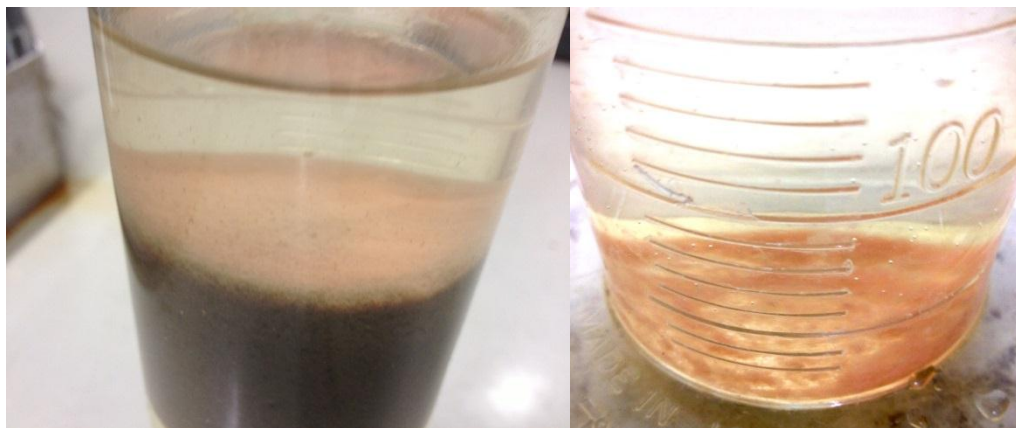
Run	Units of pollutant removed per unit of air volume delivered						
	COD_{cr} ($\text{mg}\cdot\text{L}^{-1}$)	COD_{Mn} ($\text{mg}\cdot\text{L}^{-1}$)	BOD ($\text{mg}\cdot\text{L}^{-1}$)	TOC ($\text{mg}\cdot\text{L}^{-1}$)	TN ($\text{mg}\cdot\text{L}^{-1}$)	TP ($\text{mg}\cdot\text{L}^{-1}$)	$\text{NH}_4\text{-N}$ ($\text{mg}\cdot\text{L}^{-1}$)
AOSD	40.9	11.4	24.7	6.0	5.0	0.31	3.6
30/90	47.7	13.6	29.6	7.3	5.6	0.35	4.0
60/60	25.5	7.0	15.9	3.6	1.7	0.16	2.4
40/80	38.7	11.2	23.9	5.6	4.1	0.29	3.5

These results assert, like in previous experiment setting, that higher efficiency is obtained with shorter aeration time. These results combined with nutrients analysis prove that nitrification can be achieved within around 30 minutes of aeration time and that higher $\text{NH}_4\text{-N}$ levels in shortly aerated tanks is mainly due to long anoxic phase and probably also to design of tanks. The ratio aeration duration to total cycle is therefore a major parameter. Although higher air flow rate relegated AOSD to second position in terms of air utilization efficiency, it is important to consider the final effluent's quality and the many other parameters not included in the above comparison (such as SS, SCR, dewatering cost, unit cost of each pollutant's removal, long term performance stability, bulking risk, etc.). Real assessment of AOSD system's cost

effectiveness should include environmental cost, operation cost, investment, maintenance, frequency of upsets and remediation costs as well as targeted results. Vanrolleghem et al. (1996) reported that standards are being defined in regard to wastewater treatment technologies advancement rather than to environmental impact. Nevertheless, difference between mandatory and what may be qualified as “luxury” additional pollutant removal is thinning.

4.3.5 Biota composition

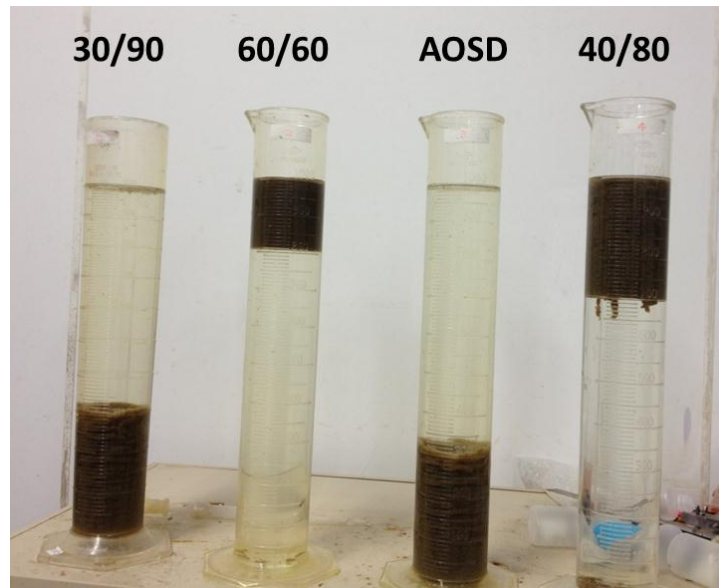
Evaluation of main biota species populations in Runs 1 to 4 was consistent with treatment performance observed. The dominance of floc feeder type *Aelosoma hemprichi* in Run2 was visible to the naked eye during settling test where a red layer appeared on top of sludge and another one was left at the bottom of cylinder after sludge started rising (Picture 4-2). Free swimming type protozoa *Colpidium caudatum* was the more strikingly present in Run1, and filter feeder protozoa *Carchesium* sp in Run4. On the other hand, Run3 showed a balanced population of *Colpidium caudatum*, *Carchesium* sp., *Epistylis* sp., nitrifying indicator protozoa *Arcella* sp. and filter feeder rotaria *Philodina* sp.



Picture 4-2 Sludge from 60/60 Run swarming with *Aelosoma* sp.

Picture on the left shows a red layer of *Aelosoma* sp forming on the settling sludge surface. After 3 hours, rising sludge leaves another layer of worms at the bottom of cylinder as seen in right picture

Phenomenon of rising sludge in the settling test is explained by trapped bubbles of N_2 produced during the denitrification process. It frequently occurred with Run2 (60/60) around end of the 3 hours settling test and occasionally in Run4 (40/80) but after 4 to 5 hours of settling (**Picture 4-3**). This was not an issue though as it was not happening in the clarification tank due to short sojourn of sludge there. Occasional foaming was also observed in Run1 operated by 30/90 system (**Picture 4-4**).



Picture 4-3 Rising sludge phenomenon in Runs 2&4 during settling test



Picture 4-4 Foaming under 30/90 conditions

4.4 Conclusion

Considering the globally constant conditions of experiment, monitoring DO and pH on a daily basis can be very helpful in spotting problems and incidents (power shortage, material dysfunction, change in influent load, etc.). On the long term, it shows a trend towards an equilibrium state in the interaction between DO, pH and biota.

AOSD system provided conditions for good stable pH of effluent, sufficient DO level (generally recommended to range from 1.0 to 2.0 mg•L⁻¹) to avoid competition between heterotrophic and nitrifying organisms. Control by pH can be of interest only in case of systems using bending points to determine aeration stop moment. Including it to AOSD might though improve accuracy and sensitivity to effluent load variations.

Over aeration leads to dissolved CO₂ stripping, which causes in turn increase in pH. This phenomenon could be used not only to determine NH₄-N depletion but also to control CO₂ emission levels

Monitoring variations of pH within a cycle coupled with DO could prove to be an interesting mean of controlling aeration time. As in some cases DO bending point can be difficult to detect, pH elbow is more likely to be spotted. This control will also help prevent high CO₂ emission. Weekly trends could also inform on sludge evolution and trigger preventive remediation.

Optimization of aeration duration by AOSD enables energy saving, helps reduce GHG emissions and improves TN removal by increasing reactions rate through efficient strains selection, favoring SND process which requires 25% less O₂ and securing enough time for the slow denitrification process which in return releases O₂ reclaimed for other reactions improving efficiency of the system.

Similar average aeration time calculated by AOSD to that of previous experiment setting seems to indicate that it is not much affected by air flow rate. The latter had

though an effect on global effluent quality improvement and should be further investigated to consider including PID control of air flow rate in AOSD system.

Chapter 5: Overall Conclusions

Wastewater treatment consists of removing pollutants it carries before discharging effluent in receiving waters or reclaim. . Since antiquity, growth of cities and increase of wastewater production favored cholera and other diseases epidemics. Before industrialization, wastewater was probably just seen as a threat to health and a source of discomfort because of the bad smells. Higher pollution from industries and awareness of environment degradation lead to development of more and more effective sewage treatment processes. Biological processes were privileged for their efficiency in treating wastewaters at lower costs. Activated sludge is the most used type. Historically, configuration of the bioreactor evolved to enhance BOD, TN and then TP removals. Researchers and engineers strived to improve design and performance of wastewater treatment plants. Recently, research turned more to operation & management issues and costs. Aeration of bioreactors followed the same pathway: after development of reliable mechanisms in terms of bubble size and homogeneity for good oxygen transfer, concern became more about fouling, maintenance and energy consumption problems. If advance treatment has pushed further the limits of effluent quality, legislation and discharge standards have also become more exacting.

Intermittent aeration has already drawn interest as a solution for energy saving by reducing aeration time, and for nitrogen removal by alternating aeration-anoxia cycles and favoring SND process. Fulfilling delivery of theoretical oxygen volume necessary for wastewater depollution depends on aeration time and air flow rate, which are easily controllable, as well as on the oxygen transfer efficiency. The latter parameter's value is affected by aeration system characteristics, shape of reactor, homogeneity of air diffusion and mixing. But it also depends on wastewater characteristics (surfactant,

slime...). A variety of AAA activated sludge process configurations exist and many systems of aeration control are also proposed.

This study throws into relief the effects of aeration time and DO level on pollutants removal and on GHG emissions, by testing a novel control system named AOSD and different fixed ON/OFF aeration sequences, during a 120min cycle, in single activated sludge reactor intermittent aeration system.

Under controlled environment temperature, fixed operation parameters (HRT, MLSS, influent return sludge and air flow rates, etc.) and influent quality standardization (by addition of urea for TN, methanol for COD, and toilet paper for SS), the aeration control settings showed in the two main experiments different results. It seems to be due to the effect of oxic period duration, combined with variations in influent load (organic matter and biota).

Obtaining excellent results in all water quality parameters by biological treatment only is very challenging, especially in the conditions of this research. Antagonistic conditions for nitrification and denitrification, good settling and good pollutants removal, sufficient aeration and energy saving...etc., all occurring in one single tank force to set clear target results and to choose optimization rather than maximization. In this research priority was given to TN removal and energy saving.

This study was conducted in three phases: (i) preliminary tests to justify intermittent aeration, select materials as well as main operation conditions and parameters, (ii) long period monitoring using 30/90, 30/90 with media, 40/80 fixed On/Off settings and AOSD to compare their effect on effluent quality, sludge characteristics and energy saving potential, (iii) addition of online pH and DO monitoring, GHG and nutrients analysis during aeration anoxia cycle for a better understanding of long term follow-up with 40/80, 60/60 and AOSD systems.

- **Preliminary tests**

During this period, continuous aeration was unable to remove TN. Nitrification was occurring despite low pH values attained but there was no possibility for denitrification process. This aeration system also caused pin floc, turbidity and too high endogenous respiration to keep MLSS at desired level. Improvement of continuous aeration would probably require lower air flow rate or different configuration of reactor.

Some researchers reported that aeration ratio to total cycle time of 50% yielded satisfactory results. More energy could be saved with shorter oxic phase. A test with 30/30 system yielded poor results compared to 30/60 showing that not only energy could be saved but also that performance could be improved if cycle duration was adequate. Fixed 120min of the aeration/anoxia cycle duration was then decided those results and as compromise between pros and cons of both longer and shorter cycles found in literature.

Choice of diffusers and air flow rate was an important step as they directly affect O₂ transfer quality. Nevertheless, under same conditions of aeration quality, ratio of aeration to anoxia proved to be a determinant factor. Stirring also plays an important role in treatment performance as the extrusion system used to replace it was not efficient.

The 30/90 and 40/80 systems were selected for the 2nd phase of experiment for they framed average aeration time calculated by AOSD.

- **Long term effluent quality monitoring**

In long term follow-up, the 30/90 system resulted in hypoxia conditions. Higher levels NH₄-N indicated poor nitrification which caused BOD₅ and TN to exceed targeted threshold of 10 mg•L⁻¹ for both parameters. Even COD_{Mn} showed insufficient organic matter degradation. On the long term bulking problems gradually increased.

Run containing media (30/90M) on the other hand achieved better results with $BOD_5 < 20 \text{ mg} \cdot \text{L}^{-1}$, $COD_{Mn} < 15 \text{ mg} \cdot \text{L}^{-1}$, and $TN < 10 \text{ mg} \cdot \text{L}^{-1}$. Low TN was ensured by combination of good nitrification and denitrification. Position of stirrer's upper fan and rotation speed created too much turbulence for sludge to significantly fix on media. Good effluent quality was therefore attributed to enhanced O_2 transfer due to bubble rise slowed down by media that constituted physical obstacles.

The 40/80 system achieved an average TN removal close to 75% in both experimental settings which may be considered as stable overall performance yet at the lower performance level of most AAA systems in literature (70-90%). It seemed that denitrification was limited by shorter anaerobic duration and possibly by insufficient –or fierce competition on- readily degradable organic matter. The aeration time and remnant DO during anoxia on the other hand decreased BOD_5 and $NH_4\text{-N}$ to low levels.

The 60/60 system, set in the 3rd phase, favored as expected nitrification over denitrification yielding poor TN removal (40%). Anoxia duration was too short for optimal conditions of denitrifying organisms' activity, and levels of DO were too high for SND to occur. Under these conditions of operation and influent load, results of this system might be comparable to conventional systems designed to perform nitrification and BOD removal.

AOSD system satisfactorily achieved the target of TN removal with a stable 84% in both experiment settings at lowest aeration time possible considering conditions and equipment characteristics. The average 2 min per cycle difference with 30/90 seemed determinant. Increase of air flow rate in 3rd experiment phase may though have influenced results too as there was an improvement in BOD removal.

Achieved TP removal in all Runs was higher than the maximum 30% reported for conventional activated sludge process. However, it was insufficient to comply with the $1 \text{ mg} \cdot \text{L}^{-1}$ standard limit. Therefore conducting tests on reactor configurations

favoring EBPR or incorporation of physicochemical phosphorus removal is recommended.

Bulking is a very common problem of wastewater treatment plants. Intermittent aeration in general and shorter aeration time particularly predisposed sludge to bulking. High SVI in all runs confirmed that. Liquid solid separation in clarification tanks was good though. However episodes of bulking especially in the long term with short aeration time or low DO could not be avoided. Longer aeration time of 60/60 caused pinfloc sludge and turbidity in the long term. It also resulted in sludge floating after 3 hours in settleability test. This phenomenon didn't occur in clarification tank though.

Biota diversity was more important with AOSD than with other Runs. Presence of some biological indicators confirmed treatment quality results.

The optimal balance between DO and biota requires control of both aeration time and DO level. Proportions of aeration, anoxic and anaerobic periods should be optimized but DO level during aeration is determinant for competition between heterotrophs and nitrifiers as well as for installation of SND organisms. If under this study's constant load and temperature conditions DO profile fluctuated, it will be challenging in full scale process under real conditions of variable load. Implementing a PID control of air flow rate is thus advisable.

- **Greenhouse gases production in relation with nutrients and pH**

As for GHG emissions, their amount is highly correlated to aeration time as dissolved gases are stripped during aeration time mostly. Only CO₂ may strip during aeration OFF time and yet at lower rate. Peak of CH₄ concentration in emitted gases was similar in the tested AOSD, 40/80 and 60/60 Runs. Total emission was though higher with higher aeration time. Very low emitted N₂O was detected with AOSD at the end of aeration time while 60/60 emitted during all aeration time and peaked at more than 8ppm. Volumes of emitted CO₂ also were far more important in 60/60 Run

with concentrations more than double of those with AOSD and 40/80 (which were relatively similar and roamed around 4,000 ppm) during all aeration time.

Analysis of samples from reactors during one aeration-anoxia cycle yielded interesting results. It showed that NO_2 accumulation occurred during denitrification for longer aeration periods and during nitrification for shorter aeration runs. Beside its high toxicity for fish in receiving waters, its accumulation in reactors may result, depending on its level and on whether it is produced by nitrification or denitrification process, in N_2O production, inhibition of both AOB and NOB, or favoring SND process. Lower peak of $\text{NO}_3\text{-N}$ coupled with good $\text{NH}_4\text{-N}$ removal with AOSD suggested a partial TN removal by SND. It also brought to the conclusion that $\text{NH}_4\text{-N}$ concentration in effluent of run controlled by AOSD was not due to insufficient time or oxygen for nitrification (on the contrary rate was higher than with 40/80 or 60/60 runs), but to accumulation of influent ammonia during the long anaerobic phase. Denitrification is a slower process compared to nitrification but an anaerobic phase of 60 to 70 minutes could suffice.

As for pH, it was concluded that over aeration period increased pH by CO_2 stripping. The pH bending point indicates thus the moment where aeration should be stopped but also CO_2 emission rate increase point.

- **Summary of AOSD assessment and recommendations**

Advantages of AOSD system are summarized as follows:

- 1) Improvement of BOD and nitrogen removal efficiency; Removal of BOD and T-N is over 95% and over 85%, respectively.
- 2) Stability of results in the long term
- 3) Diversification and stabilization of biota
- 4) Prevention of sludge bulking
- 5) Improvement of sludge reduction

- 6) Control of GHGs emissions; CO₂, CH₄ and N₂O emission 7%, 2% and 72%, respectively, less compared with lowest fixed intermittent operation Run emissions.
- 7) Reduction of power consumption; Power consumption could be reduced to 46% of 1:1 aeration/anoxia ratio fixed intermittent systems.
- 8) Very little maintenance of AOSD system is required (cleaning of DO probe)

Many technical problems were encountered during this study. Tanks, tubes, peristaltic pumps, flowmeters and electrovalves required regular cleaning maintenance and checking. Frequent and quick clogging of tubes was mainly due to their small diameter, low functioning pressures and deposits on their walls. Clogging of PVC tube from aerator to clarifier also occasionally occurred due to filamentous and slimy sludge balls accumulating in parts not reached by cleaning brush. One of the other causes of problems was the high relative humidity in summer. Phenomenon of condensation led to accumulation of water droplets inside air flow meters and in aeration tubes. High humidity was also responsible for influent tubes to drop off from glass holder, and provoked shunts in influent injection system's volume control which sometimes kept influent electrovalve closed until it was noticed. Although solutions were found for each situation, regular and frequent maintenance was very important and time consuming. Note however that AOSD system itself required virtually no maintenance. Only probe was regularly cleaned to keep high measurement accuracy.

Data processing was also a constraint in this study. Data collected from AOSD were stored in Access database, portable DO meter's output was a text file, and all other parameters were stored in Excel file. Development of queries, macros and pivot tables were therefore necessary. More elaborate software to display pH and DO figures and charts, with possibility to select periods calculate averages fluctuations, AOSD calculated time for each cycle, keep logs on odd values, etc. would have been a valuable tool.

The AOSD system proved to be a reliable tool of aeration modulation for a good biological equilibrium and more homogenous treatment performance. It also proved to reduce GHG emission. Nevertheless, more tests should be conducted to study its response to:

- variable temperature
- variable influent load conditions (flow rate and strength)
- different configurations of reactor
- full scale plants conditions

Improvements could also be considered such as:

- including pH in the control system
- aeration flow rate PID control for DO stabilization and more adaptability to all situations
- including mixing control in alternation with aeration and optimization of its duration to save more energy

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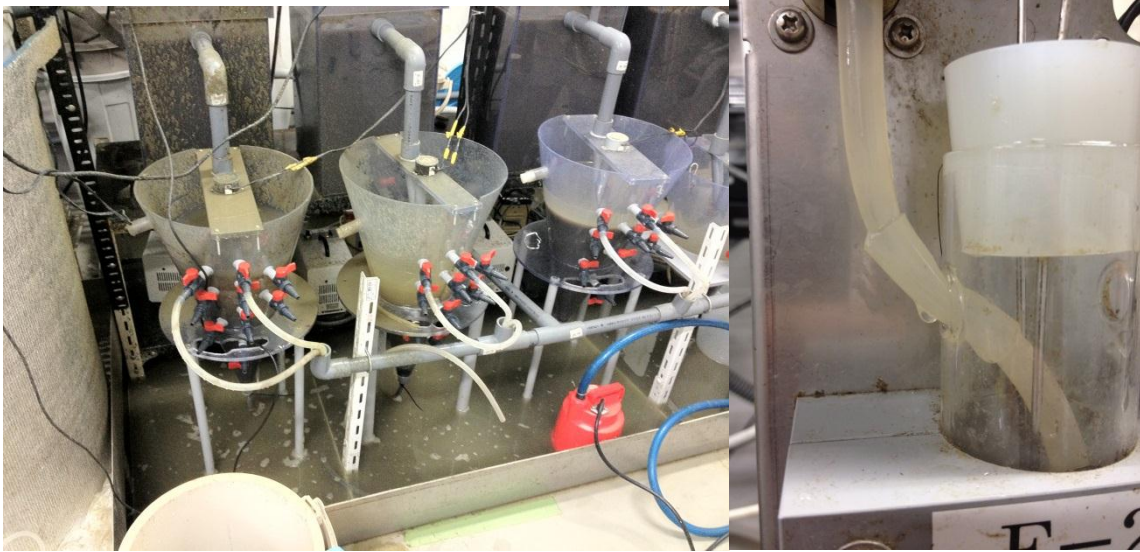
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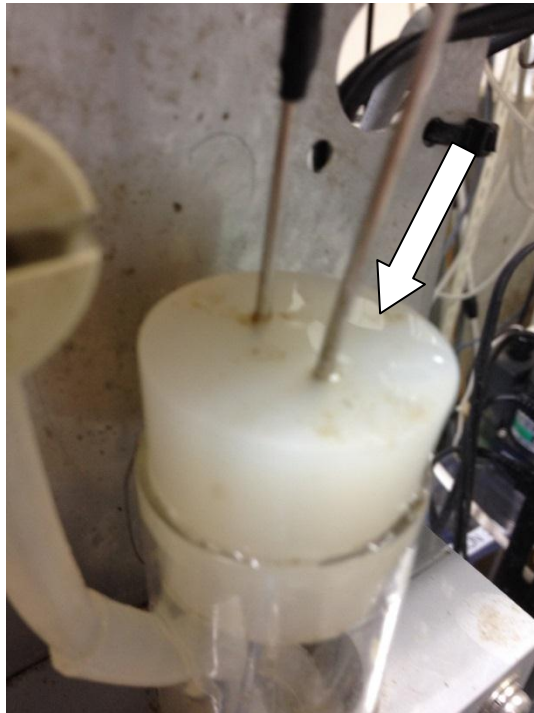
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Annex

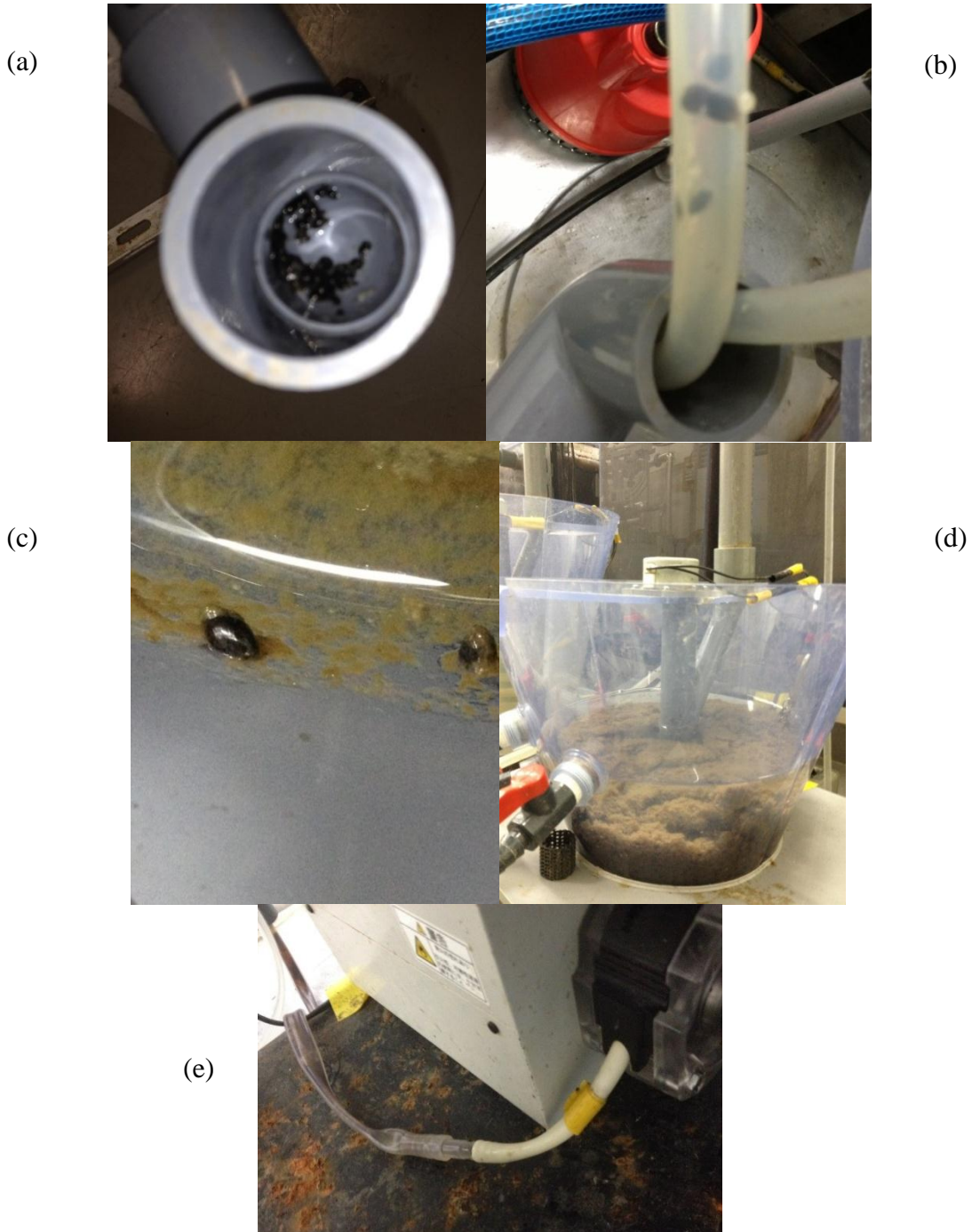
Annex A: Examples of malfunctions and their causes



Picture: Picture on the left shows damage found a morning after influent tube of Run1 had slipped out of the glass holder under effect of dew combined with raw water pressure. A small piece of larger diameter silicon tube was added, as shown in picture on the right, to make influent tube fitter to glass holder's hole size and more rigid.



Picture: Drops from condensation on raw water pipe passing above injection system accumulated on silicon cork were responsible of shunting level control electrodes and thus prevented feeding tank with influent.



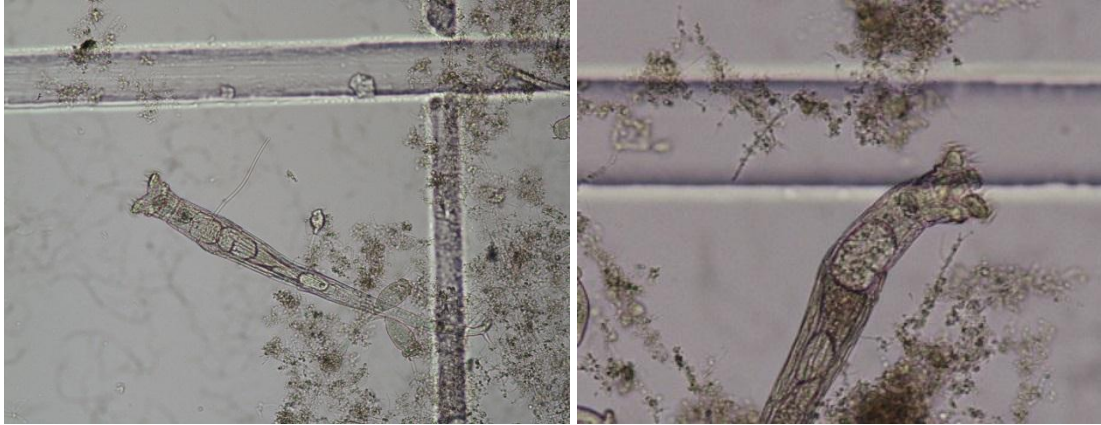
Picture: This small mollusk was at the origin of frequent return sludge tube clogging. Bigger shells obstructed return sludge tubes at junctions, causing accumulation of sludge in clarification tank. This problem was found with Runs 1 & 4. Source of infestation was found to be the effluent discharge sewer. The snails travelled upstream from sewer through PVC tube until its extremities (where discharge tubes of Runs 1&4 are connected) and from there to clarification tank. Snails come up from effluent discharge pipe (a) and follow effluent water upstream into silicon tube connecting pipe to clarification tank (b). They usually gather close to water surface on the wall of the clarification tank (c). when aspirated by return sludge pump they may cause accumulation of sludge in clarification tank (d) as they obstruct plastic tube connector when their size is bigger than connector(s diameter) (e).


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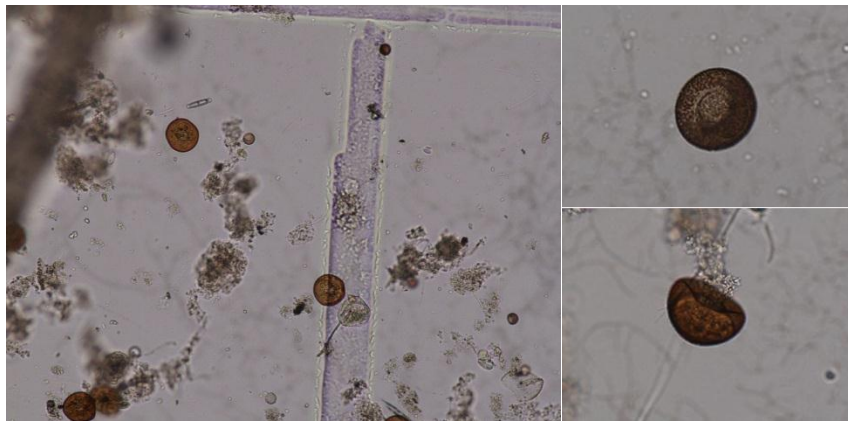
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Range("A:B,E:F,H:I,K:CF").Select
Range("K1").Activate
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ActiveWindow.ScrollColumn = 3
ActiveWindow.ScrollColumn = 2
ActiveWindow.ScrollColumn = 1
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ActiveCell.FormulaR1C1 = "0"
Range("C3").Select
ActiveCell.FormulaR1C1 = "1"
Range("C2:C3").Select
Selection.AutoFill Destination:=Range("C2:C501")
Range("C2:C501").Select
ActiveWorkbook.Worksheets(filename).Sort.SortFields.Clear
ActiveWorkbook.Worksheets(filename).Sort.SortFields.Add Key:=Range("C2"), _
    SortOn:=xlSortOnValues, Order:=xlDescending, DataOption:=xlSortNormal
With ActiveWorkbook.Worksheets(filename).Sort
    .SetRange Range("C2:C501")
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    .MatchCase = False
    .Orientation = xlTopToBottom
    .SortMethod = xlPinYin
    .Apply
End With
ActiveWorkbook.Worksheets(filename).Sort.SortFields.Clear
ActiveWorkbook.Worksheets(filename).Sort.SortFields.Add Key:=Range("C2"), _
    SortOn:=xlSortOnValues, Order:=xlAscending, DataOption:=xlSortNormal
With ActiveWorkbook.Worksheets(filename).Sort
    .SetRange Range("A2:D501")
    .Header = xlNo
    .MatchCase = False
    .Orientation = xlTopToBottom
    .SortMethod = xlPinYin
    .Apply
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Else
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End If
End Sub

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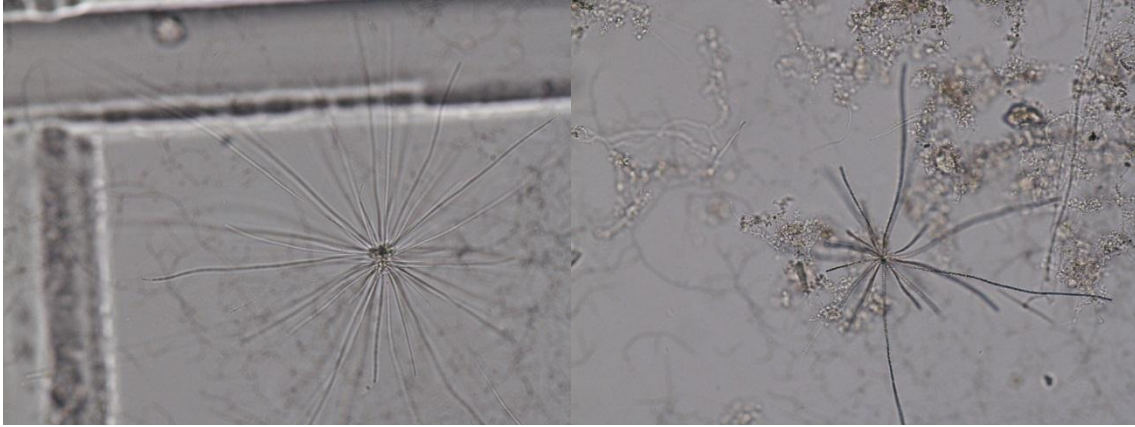

Annex C: Pictures of some organisms identified in sludge



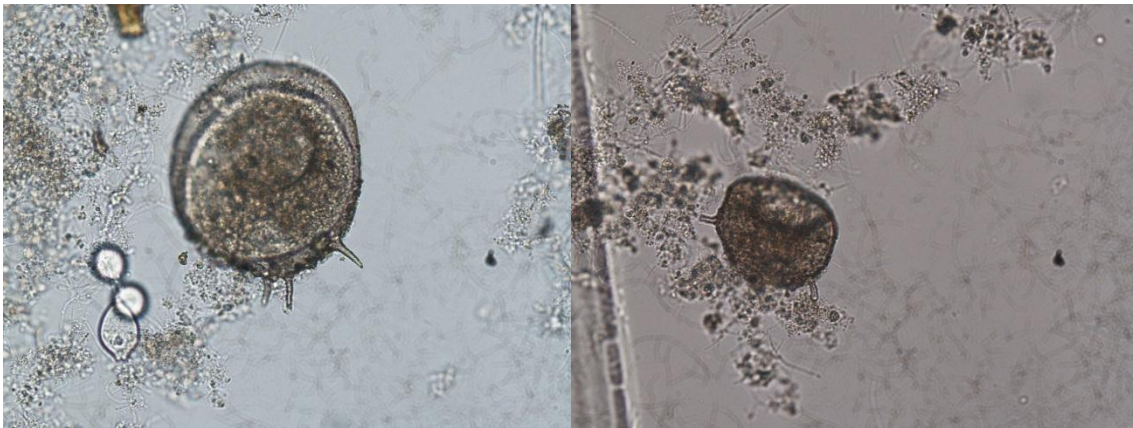
Picture: *Philodina sp* favors sludge reduction and clarification by feeding on bacteria, protozoa and mastigophora. It is an indicator of low BOD and good nitrification conditions. It usually coexists with *Arcella sp* and *Euglypha sp*



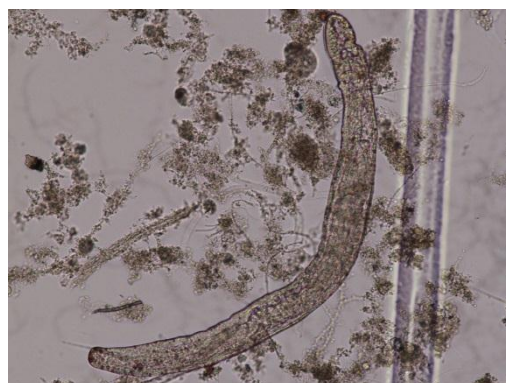
Picture: *Arcella sp* feeds on bacteria algae and mastigophora. It indicates good nitrification conditions and BOD of around $20 \text{ mg}\cdot\text{L}^{-1}$. The picture on the left shows big and small types of *Arcella*.



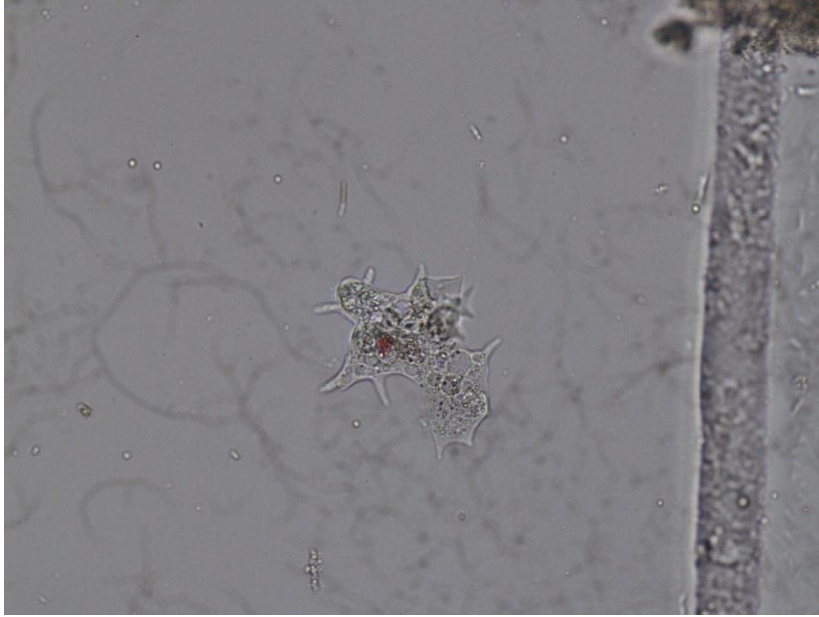
Picture: Anaerobic filamentous bacteria are found in conditions of hypoxia and their proliferation is indicated by putrid smell of H₂S.



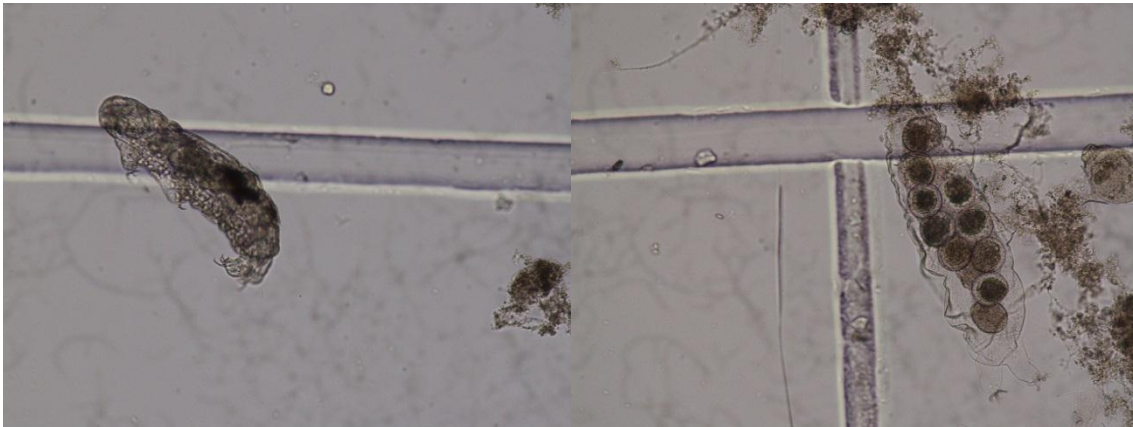
Picture: *Centropyxis* sp is a genus from the order of Arcellinida.



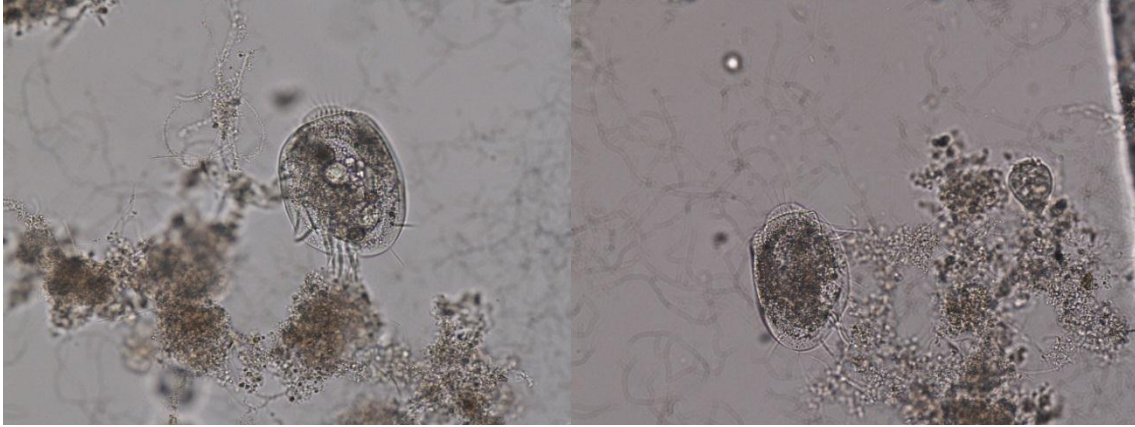
Picture: *Aelosoma* sp in populations of around 1000 N/mL indicates good conditions of nitrification and floc formation, low BOD, and SVI of less than 100. It feeds on bacteria and protozoa.



Picture: *Amoeba sp* feeds on bacteria and protozoa. In big populations it causes floc dispersion and thus bad sludge settling and effluent turbidity. It usually prevails under high DO and low BOD conditions.



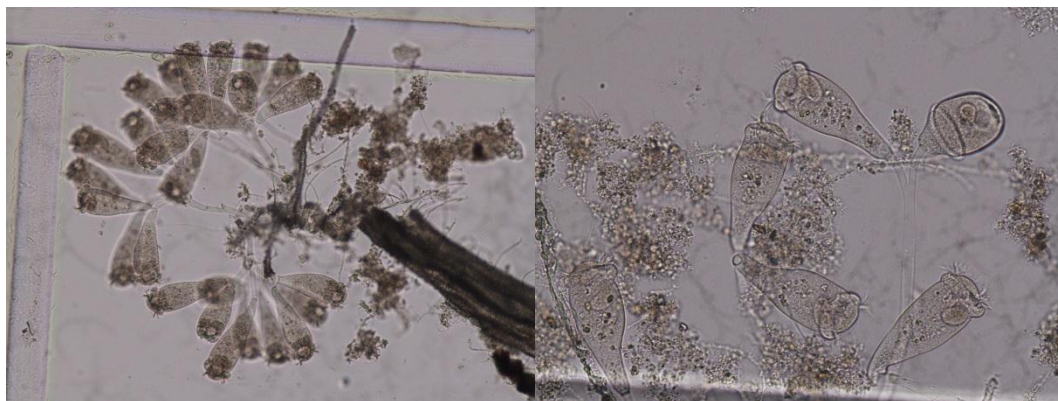
Picture: *Macrobiotus sp*, in populations of around $1000 \text{ N}\cdot\text{mL}^{-1}$, indicates well settling conditions ($\text{SVI} < 100$), low BOD and very good nitrification. It often coexists with *Arcella sp*, *Spirostomum sp* and *Centropixys sp*. Picture on the right shows an individual containing eggs.



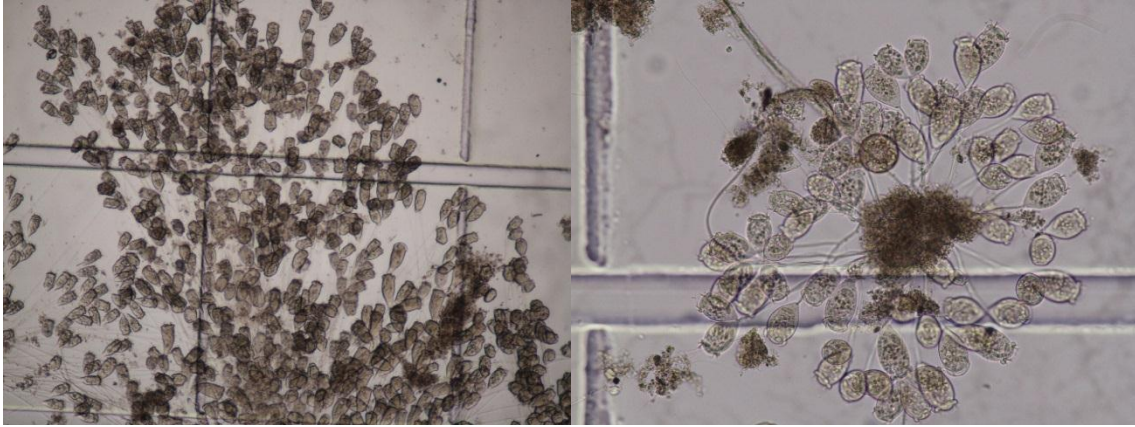
Picture: *Euplotes sp* lives in low BOD condition. It feeds on bacteria, protozoa and ciliates.



Picture: *Lecane sp* usually coexists with *amoeba sp* (BOD of $20 \text{ mg}\cdot\text{L}^{-1}$ and turbid water)



Picture: *Epistylis sp* predates on *Escherichia coli* and *Pseudomonas sp*. It indicates BOD ranging between 10 and $20 \text{ mg}\cdot\text{L}^{-1}$. The picture on the right shows a head budding.



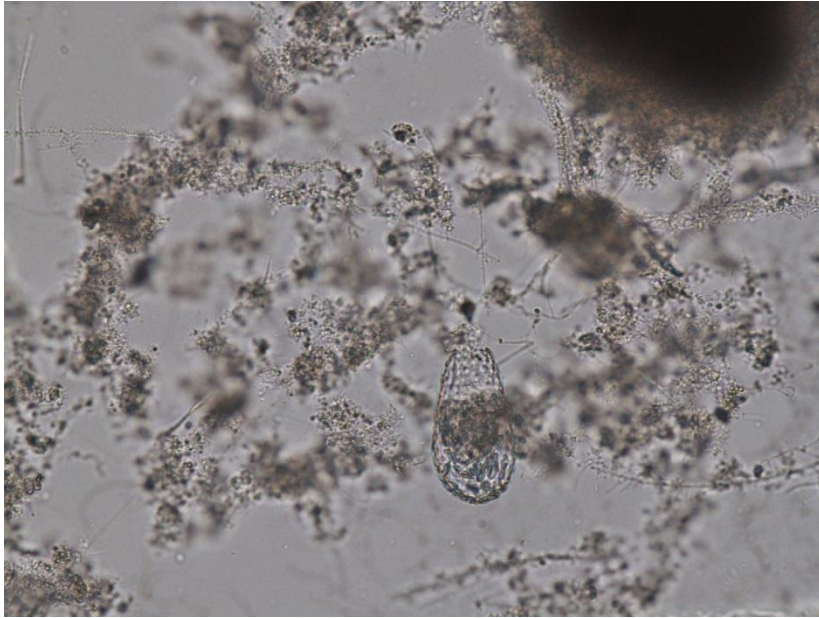
Picture: *Carchesium sp* is a bacteriophage differentiated from *Epistylis sp* by the visible spasmoneme in the peduncle. It favors sludge reduction by feeding on bacteria, protozoa and mastigophora. It indicates when in big populations ($5,000-10,000 \text{ N}\cdot\text{mL}^{-1}$) low BOD and clear effluent.



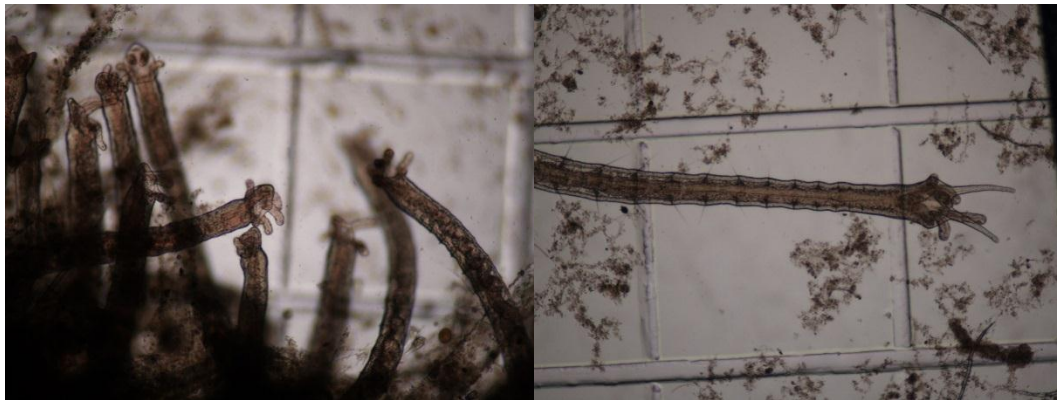
Picture: *Vorticella sp* is a bacteriophage which can be found under low DO conditions at BOD of $15-20 \text{ mg}\cdot\text{L}^{-1}$.



Picture: *Vaginicola sp* has similar functions of *Philodina sp*. It is an indicator of good effluent quality, low BOD, low SVI and good flocking conditions. It is usually found with *Vorticella sp*. It is recognizable by the sheath protecting two heads.



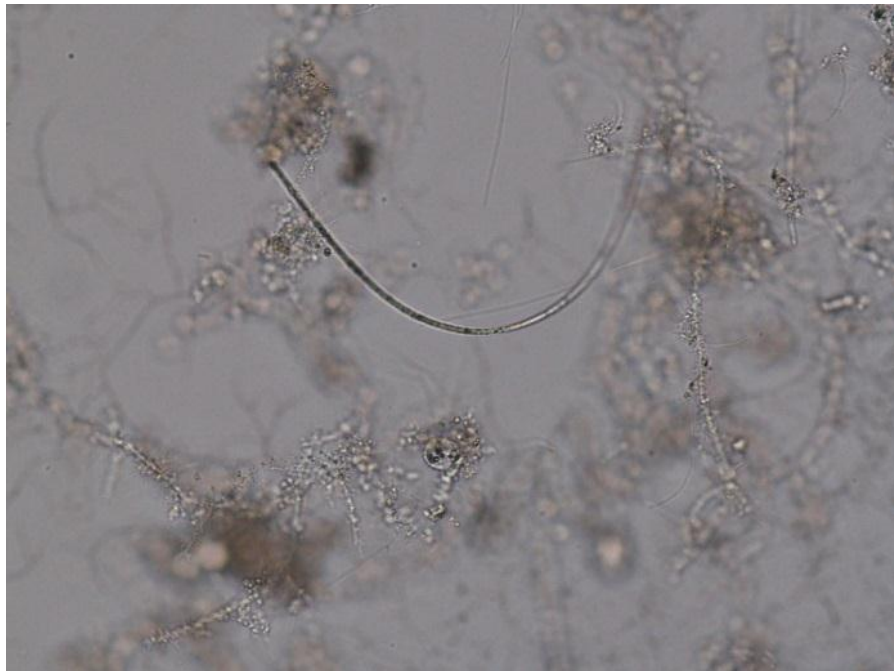
Picture: *Euglypha sp* has an ovoid shape and is covered with shell-plates. It feeds on bacteria and indicates low BOD, long SRT, good nitrification and low turbidity. It is generally found where *Arcella sp* is abundant.



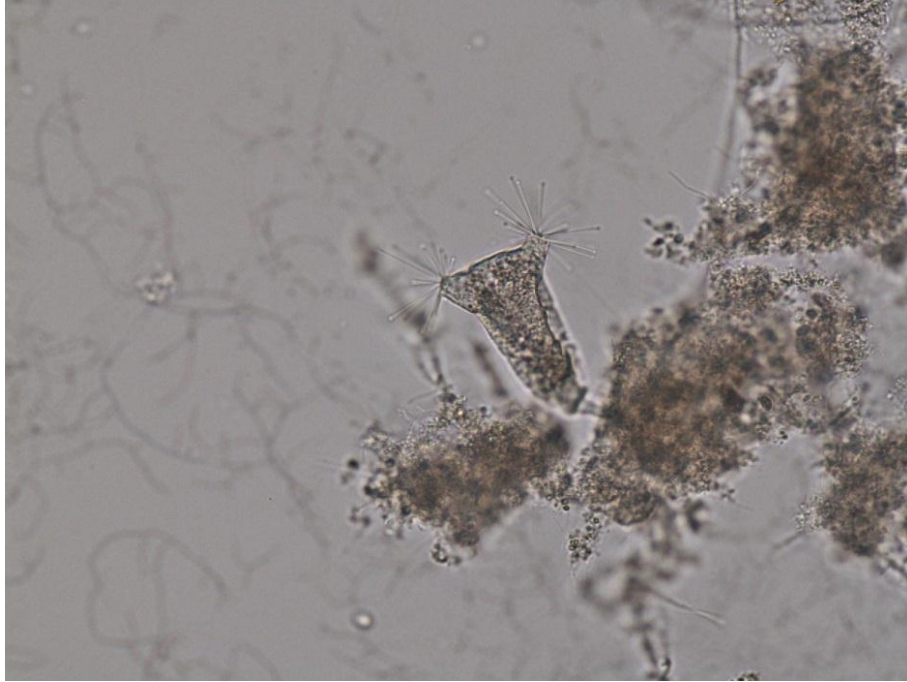
Picture: *Dero sp* is a worm of 10-20mm recognizable by its tentacle-like branchiae. It is usually found in low BOD clear waters along with *Arcella sp*, *Philodina sp* and *Carchesium sp*.



Picture: *Pristina sp* is a worm found under similar conditions to *Dero sp.*



Picture: *Beggiatoa sp* is a filamentous bacteria present under hypoxic to anaerobic conditions it proliferates at $\text{BOD} > 50 \text{ mg} \cdot \text{L}^{-1}$ and causes putrid smell.



Picture: *Tokophrya sp* feeds on *Aspidisca sp* and protozoa. It indicates $\text{BOD} < 20 \text{ mg} \cdot \text{L}^{-1}$.



Picture: *Opercularia sp* is a bacteriophage that lives under conditions of $\text{BOD} < 20 \text{ mg} \cdot \text{L}^{-1}$ and distinguishes by its capacity to survive high concentrations of metals.