Testing a Pilot Wastewater Treatment Plant in Laboratory Scale

Miguel Guillermo Rodríguez del Egido

Bachelor's thesis
July 2015
Degree Programme in Environmental Engineering

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ABSTRACT

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The aims of this project were taking the laboratory-scale wastewater treatment plant into use, make test runs by changing several factors and analyze the wastewater quality focusing on nutrient removal.

Experiments were carried out in the laboratories of TAMK, concretely in the environmental and process laboratories, during 4 months.

The information and results obtained can be used for laboratory courses for future students and new projects related to water treatment.

At the plant, biological, chemical and mechanical processes are carried out. The system consists of different modules in which there are processes of denitrification and nitrification, aeration and clarification.

The results show that phosphorus removal of the system is done properly, while regarding to nitrogen removal seems to be confused because of the different forms in which nitrogen appears in the process.

Finally, the testing project should be continued. Further experiments may be performed by changing various factors, analyzing different parameters and using others methods of analysis.

Key words: denitrification/nitrification, nutrients removal, wastewater, laboratory scale

CONTENTS

ABBREVIATIONS AND TERMS

P	Phosphorus
N	Nitrogen
NH_4^+	Ammonia
NO_3^-	Nitrate
NO_2^-	Nitrite
BOD	Biological Oxygen Demand
COD	Chemical Oxygen Demand
PAO	Phosphorus Accumulating Organisms

1 INTRODUCTION

Wastewater is any water that has been modified by human action making it inappropriate or dangerous for human consumption, industry, agriculture, etc. Different types of wastewater can be defined according to the source and contaminants of them: domestic wastewater, industrial wastewater, agricultural wastewater, and storm wastewater. (Torre, R., 2014)

Domestic wastewater contains mainly organic matter (proteins, lipids, carbohydrates, etc), phosphorus, solids, ammonia, pathogens; industrial wastewater is characterized by chemicals, hydrocarbons, etc; agricultural wastewater contains mostly pesticides and fertilizers; and storm wastewater is based on rainfall and snowmelt. (Torre, R., 2014)

The main problem of the wastewater is the high amount of carbon, nitrogen and phosphorus, which causes the consumption of dissolved oxygen and the eutrophication of lakes and rivers. Eutrophication means a nutrient enrichment of an ecosystem, it is a massive supply of nutrients in an aquatic ecosystem. Eutrophication comes from the excess of supply of nutrients, which leads the extremely fast growth of plants and algae that, when these organisms die, they consume oxygen in the water, creating an anoxic state.

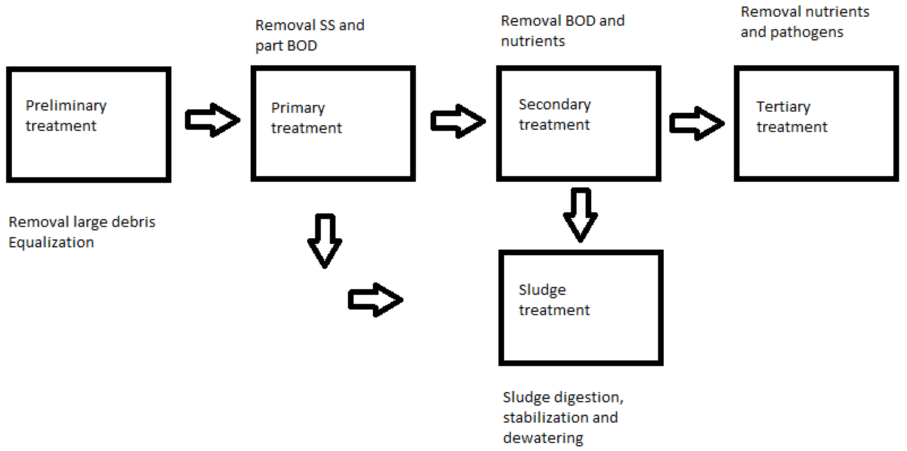
This is the reason why wastewater treatment is necessary. (Torre, R., 2014)

1.1. Wastewater Treatment

Wastewater treatment is a standardized process, which usually consists of 3 phases:

The process starts with a preliminary treatment which is based on the elimination of large and heavy solids. Homogenization tank to avoid fluctuations is used. Next stage is the primary treatment, this part of the process is characterized by the removal of suspended solids and a part of Biological Oxygen Demand (BOD). Physical operations are used such as sedimentation or flotation. In sedimentation, solids and BOD are removed by gravity. Flotation consists of separation of suspended solids by introducing air bubbles. Air bubbles adhere to the particles. The overall particle-bubble rises to the surface where they are collected by mechanical systems (scrapers). (Torre, R., 2014)

The secondary treatment is based on the removal of the organic matter (BOD) and nutrients. Chemical and biological processes are used in this phase. The common processes used are lagooning (small scale), activated sludge systems and trickling biofilters. Finally there may be a tertiary treatment in which pathogens are removed.



Picture 1. Block diagram (Picture modified of Raul Muñoz, 2014)

1.2. Removal of Nitrogen by biological Nitrification/Denitrification

The removal of Nitrogen in standard wastewater treatment plant is based on the conversion of organic and inorganic N (NH_4^+, NO_3^-, NO_2^-) into N_2, which is released into the atmosphere.

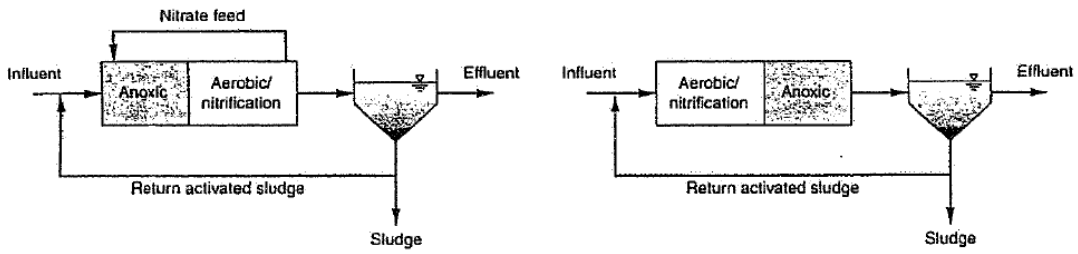
This process is the most widely used because of: high potential removal efficiency, high process stability and reliability, relatively easy process control, low land area requirements and moderate cost. (Tchobanoglous & Burton, 1991)

Nitrification is a biological process in which ammonia or ammonium are oxidized to nitrite or nitrate. This process is slow and sensitive to environmental conditions such as low temperatures, presence of heavy metals and high concentration of NH_3.
Regarding to pH, optimal nitrification happens between 7.5 and 8, and a pH of 7 to 7.3 is usually used.
Two groups of bacteria are necessary; in a first step, ammonia is oxidized to nitrite by *Nitrosomomas* and in a second step, nitrite is oxidized to nitrate by *Nitrobacter*. Both

groups of bacteria are autotrophic. The impact of the nitrification during the aerobic treatment is the increase of the demand of O_2 and the increase of the production of sludge (less significant). (Torre, R., 2014)

$$2\,NH_4^+ + 3\,O_2 \rightarrow 2\,NO_2^- + 4\,H^+ + 2\,H_2O \quad (Nitrosomonas)$$
$$2\,NO_2^- + O_2 \rightarrow 2\,NO_3^- \quad (Nitrobacter)$$
$$NH_4^+ + CO_2 + O_2 \rightarrow Biomass + NO_3^- \quad (Total)$$

Denitrification is based on the reduction of nitrate to nitrous oxide, nitric oxide and nitrogen gas. Two kinds of denitrification process can be used as it can be observed in Picture 2. The most common process is that which consists of an anoxic chamber where denitrification occurs followed by an aerobic chamber where nitrification happens and a final chamber to separate effluent and sludge, a part of this sludge will be recirculated and the other part is a system outflow. The other process consists of an aerobic tank before the anoxic tank and, as in the previous process, a chamber to separate effluent and sludge. (Tchobanoglous & Burton, 1991)



Picture 2. Different nitrogen removal processes (Tchobanoglous & Burton, 1991)

1.3. Removal of Phosphorus

Phosphorus removal can be done in two ways, chemically and biologically.
In the biological removal, phosphorus is incorporated into cell biomass, so phosphorus is removed as a sludge waste. The clue is that microorganisms are exposed to alternatives conditions, aerobic and anaerobic. Biological phosphorus removal (BPR) is based on the ability of a certain group of microorganisms, Phosphorus Accumulating Organisms (PAOs), to accumulate polyphosphate as a source of energy under aerobic conditions.

It occurs in two stages. First stage is an anaerobic process: Acetate is released as a product of fermentation of organic matter or chemical oxygen demand (COD). PAOs assimilate acetate as polyhydroxybutyrate (PHB) using the stored energy in the links of P present in the internal polyphosphate reserves, and PO_4^{3-} is released to the extracellular medium. The second stage is an aerobic process where the stored PHB is metabolized as an energy source in the presence of O_2 and new cellular material is formed. The energy produced is stored as polyphosphates. The biomass removal process involves the removal of P. (Torre, R., 2014)

There are several typical processes in the phosphorus treatment such as Anoxic/Oxic (A/O) Process, PhoStrip Process and Sequencing Batch Reactor, etc.

Regarding to the chemical elimination, chemicals are added to wastewater in order to produce insoluble salts when reacting with phosphorus. The typical chemicals used to achieve the aim are alum, sodium aluminate, ferric chloride or sulphate and lime.

Factors which affects the choice of the chemical for the treatment are: influent phosphorus level, wastewater suspended solids, alkalinity, chemical cost, reliability of chemical supply, sludge handling facilities, ultimate disposal methods and compatibility with other treatment processes. (Tchobanoglous & Burton, 1991)

Reactions during the precipitation and chemical species in sludge according to Tchobanouglus & Burton,1991.

- Lime

$$5\,Ca^{+2} + 3\,PO_4^{-3} + OH^- \leftrightarrow Ca_5(PO_4)_3(OH)$$

$$Mg^{+2} + 2\,OH^- \leftrightarrow Mg\,(OH)_2$$

$$Ca^{+2} + CO_3^{-2} \leftrightarrow CaCO_3$$

- Alum

$$Al^{+3} + PO_4^{-3} \leftrightarrow AlPO_4$$

$$Al^{+3} + 3\,OH \leftrightarrow Al\,(OH)_3$$

- Iron Fe (III)

$$Fe^{+3} + PO_4^{-3} \leftrightarrow FePO_4$$

$$Fe^{+3} + 3\,OH^- \leftrightarrow Fe\,(OH)_3$$

The use of lime is decreasing due to the rise of the sludge to be treated in comparison with the other methods, and their respective operation problems. About metal salts, these can be added in different points of the process, however because of orthophosforus is easily removed, these salts are added after the secondary treatment. (Tchobanoglous & Burton, 1991)

2 OBJECTIVES

The aims of this project are taking the laboratory-scale wastewater treatment plant into use, make test runs by changing factors of pH, flow rate, oxygen concentration and dosing of coagulant and analyze the wastewater quality focusing on nutrient removal, and making user instructions for the device.

As it is previously discussed in Introduction Chapter, the nutrient removal is carried out by chemical and biological processes.

This bachelor thesis was commissioned by Tampere University of Applied Sciences TAMK, and it is the university itself which will benefit from it.

3 SYSTEM DESCRIPTION

3.1. Description of the system

As in the real wastewater treatment plants, in the pilot plant wastewater is purified by chemical, biological and mechanical processes. Four modules can be identified:

The first chamber is the pre-clarification, this module is used when wastewater contains a large amount of organic and solid matter, which sediments by gravity. Due to the recipe used to create synthetic wastewater, this does not contain large amounts of organic and solid matter so this chamber is bypassed.

Next step is the denitrification chamber, as the name of the module expresses, the process of denitrification happens here by which nitrate (NO_3^-) and nitrite (NO_2^-) become in nitrogen. Denitrification involves anoxic conditions which are fulfilled as the oxygen concentration is around 0.13 mg/L.

Chemical action is carried out in this chamber since coagulant and sodium hydroxide are added.

The coagulant used is a coagulant based on ferric sulphate, its trade name is PIX. Using this coagulant, the highest maximum adsorption of phosphorus is achieved. (Smoczynski et al., 2014)

Biomass formed in the aeration tank and inorganic precipitates from the clarification module are recirculated to this chamber.

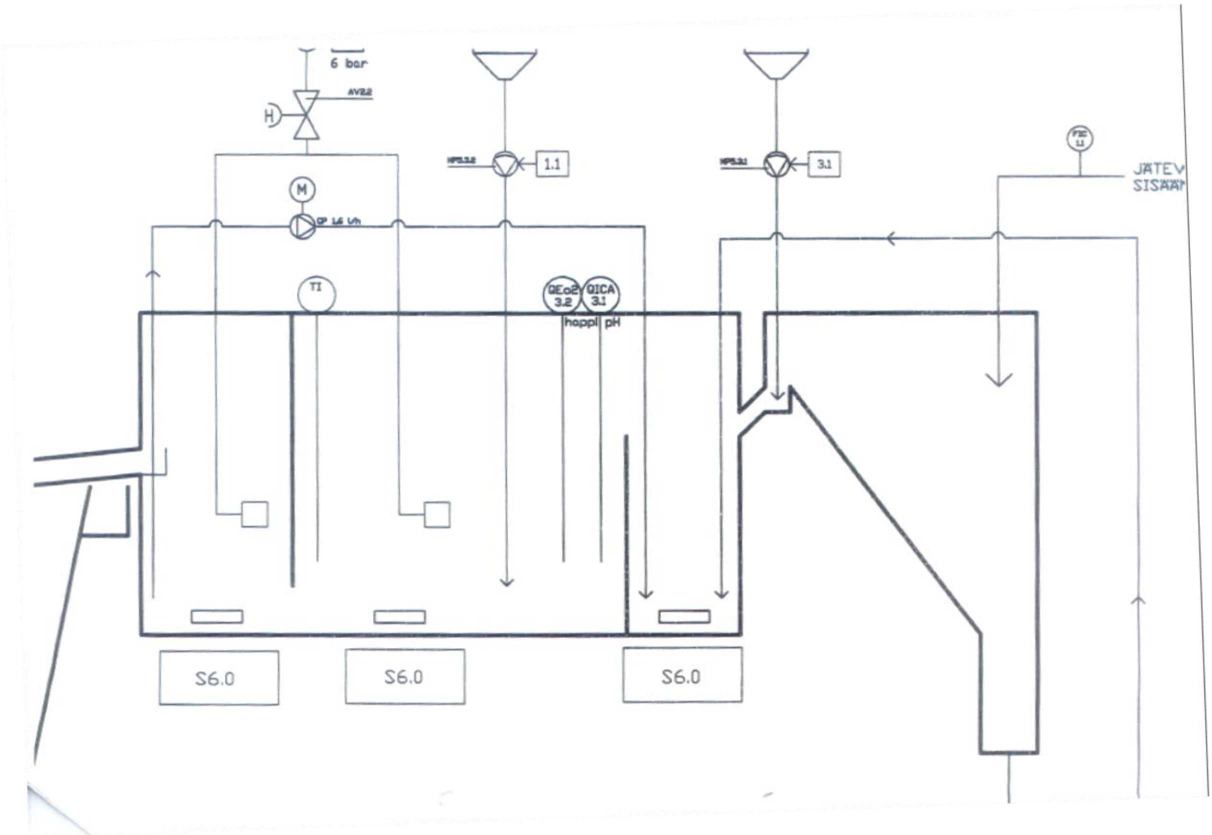
Third module is the aeration/nitrification chamber where activated sludge is found, optimal condition for growth is an oxygen concentration of 3-4 mg/L according to instructions given by pilot wastewater treatment plant manual, in order to fulfil this condition airflow is continuously bubbled into the module. Air flow is controlled by a valve and the pressure that arrives from air system. Activated sludge is collected from Hatanpää wastewater treatment plant.

Final step is the clarification module in which biomass formed in the aeration tank and inorganic precipitates are separated from the water by gravity and some of the precipitates are recirculated to the denitrification chamber. The clarified water is obtained by overflow and goes directly to the sink.

3.2. Flow diagram

In this chapter, the system is presented as a flow diagram in which the different flows between chambers and instrumentation can be observed in Picture 3.

The instrumentation in the system consists of a valve, pumps, sensors indicators and aerators, which are represented by their symbols.



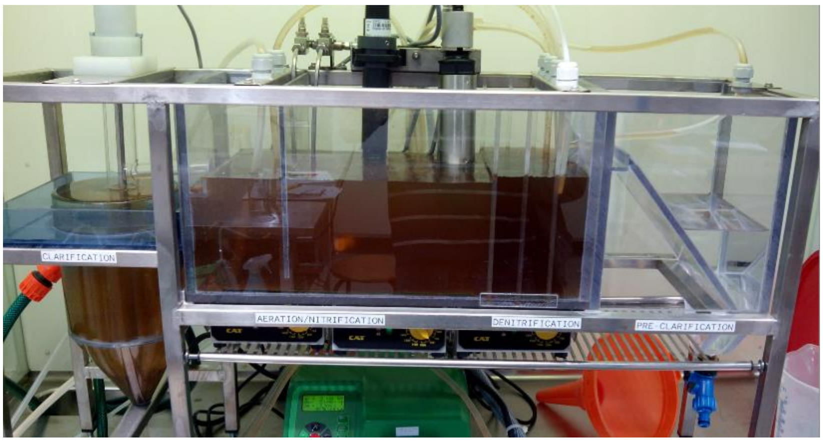
Picture 3. Flow Diagram (Saimaa University of Applied Sciences, Manual for the Lab Scale Wastewater Treatment Plant, 2013)

3.3. Description of equipments

In this section, all the equipment is described. All the equipment has a purpose which will be explained in the individual description of each equipment. The system equipment can be observed in Picture 4.

Picture 4. Working place (Miguel Rodríguez del Egido, 2015)

The different parts that make up the system are: The Pilot Plant which is the main part of the system where the purification process is carried out, it is divided in different chambers as it can be observed in Picture 5.



Picture 5. Pilot plant (Miguel Rodríguez del Egido, 2015)

There are different kind of pumps, one of them is the Solenoid Metering Pump, Prominent Beta b 5, which is the pump used to inject air into the system in order to keep clean the aerators and provide an extra supply of air, it can be seen in Picture 6. Another are the Peristaltic Pumps, Watson Marlow 520u which are used to boost the synthetic water along the system, they can be observed in Picture 7. The other type of pump is Grundfos Alldos dda, these pumps are used to boost the coagulant and NaOH to the denitrification chamber and are shown in Picture 8.

Picture 6. Air pump (Miguel Rodríguez del Egido, 2015)

Picture 7. Synthetic water pump (Miguel Rodríguez del Egido, 2015)

The rest of the system consists of a bucket, with a volume of 80L, which is the space from which synthetic waste water is injected into the system. The Controller Hach Lange sc200 is the controller which collects data of oxygen and pH from different sensors and displays it. The dissolved oxygen sensor, Hach Lange LDO Sensor is the instrument which measures the oxygen in the aeration/nitrification chamber. The last equipment is a pH Sensor, Hach Lange phD sc Digital Differential pH/ORP Sensor is the device which measures the pH in the aeration/nitrification chamber.

Picture 8. Coagulant and NaOH pump (Miguel Rodríguez del Egido, 2015)

4 PROCESS AND METHODS

4.1. Collecting inoculum of activated and return sludge

An important action is to collect the inoculum, from the conventional wastewater treatment plant, which will be used in the pilot plant and containing the necessary microorganisms to carry out the biological process. Half a litre of activated sludge and return sludge were collected each time.

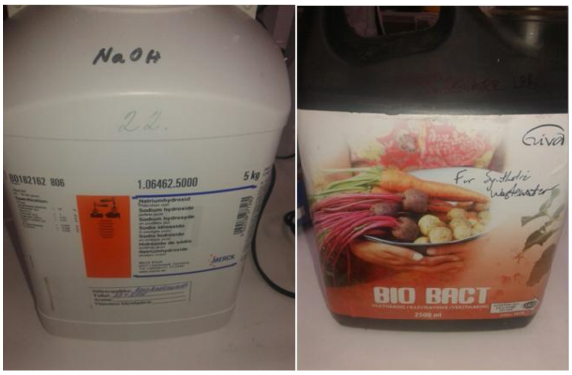
For this objective, containers such as those that appear in Picture 9 are used.

Picture 9. Collecting samples in wastewater plant (Google Images)

4.2. Synthetic waste water

During the realization of the experiments, two different recipes have been used to make synthetic water. The first one was composed, for 6 litres, of 1300 grams of treacle, 22 grams of urea, 25 grams of dishwater and fill with water to 6 litres. The second recipe was composed, for 900 litres, of 900 grams of glucose or sugar, 42.9 grams of urea, 1161.7 grams of a commercial product called BioBact, 40 grams of sodium hydrophosphate and fill with water to 900 litres.

From these recipes, it is necessary to extrapolate to the appropriate amounts for the different experiments. In Picture 10 some of the compounds of the recipe are shown.



Picture 10. Some components of the recipe (Miguel Rodríguez del Egido, 2015)

4.3. Methods

The methods used have helped to measure nitrate, phosphorus, and pH and dissolved oxygen. Whatever it was the substance to be measured, there have always been a number of common steps.

The first step is to collect a representative sample of the different modules from the pilot plant as it is shown in Picture 11.The numbers observed in the beakers correspond to a simplified way to differentiate the samples that have been taken from different chambers. The number 1 is the synthetic wastewater, number 2 is denitrification chamber, number 3 is aeration/nitrification chamber and finally number 4 is from clarification chamber. The following step is to prepare the sample to bring them to the centrifuge, they are taken to the centrifuge so that the solids settle to the bottom, and thereby the solids do not interfere with the results. The samples have been treated in the centrifuge with 2000 rpm for 5 minutes.

The following analyzing methods have been used to measure several parameters.

Picture 11. Samples of the different modules (Miguel Rodríguez del Egido, 2015)

4.3.1 Nitrate

Nitrate has been analysed with Spectrophotometer HACH Lange DR 2800 according to Cadmium Reduction Method by which cadmium metal reduces nitrates in the sample to nitrite. Complete instructions can be found in HACH DR 2800 Procedures Manual, Method 8039, Nitrate. This device can be observed in Picture 12.

Picture 12. Spectrophotometer HACH (Miguel Rodríguez del Egido, 2015)

4.3.2 Orthophosphate

Orthophosphate has been analysed with Spectrophotometer HACH Lange DR 2800 according to PhosVer 3 (Ascorbic Acid) Method by which orthophosphate reacts obtaining finally an intense blue colour. Complete instructions can be found in HACH DR 2800 Procedures Manual, Method 8048, Phosphorus Reactive (Orthophosphate). This spectrophotometer can be observed in Picture 12.

4.3.3 Dissolved oxygen and pH

These parameters are continuously measured and monitored. They have been analysed with Hach Lange LDO Sensor and Hach Lange phD sc Digital Differential pH/ORP

Sensor, and monitored with Hach Lange sc200. The controller displays the data on a screen like the one shown in Picture 13.

Picture 13. Monitoring Screen (Miguel Rodríguez del Egido, 2015)

5 RESULTS

The results obtained during the experiments will be developed in this section.

Oxygen and pH have been measured in aeration/nitrification chamber. These variables are less important in others chambers, even though they have been measured. The measured oxygen in the denitrification module was 0.13 mg/l. The influent conditions were 0.14 mg/l O_2 and pH 6.25. The conditions under which the experiments have been carried out can be observed in Table 1.

Table 1. Working conditions

Date	pH	Oxygen (mg/l)	Flow (rpm)	PIX (ml/h)
15/05/2015	7.06	4.55	3	30
12/05/2015	7.49	3.89	4	50
11/05/2015	7.33	3.72	6	50
08/05/2015	6.82	3.77	6	30
07/05/2015	7.12	6.75	6	30
06/05/2015	7.07	4.42	6	50
05/05/2015	6.95	5.44	6	40
04/05/2015	6.73	5.41	6	40
30/04/2015	7.51	3.18	6	30
29/04/2015	8.88	5.46	6	20
24/04/2015	6.5	3	6	20

The nitrate and phosphate values obtained from the samples taken from the different modules are shown in Table 2. In the case of phosphorus, the spectrophotometer gives the measurement in PO_4 and it has been calculated the desired value.

Table 2. Concentrations obtained from the different modules

Date	SAMPLE	NO_3-N (mg/l)	PO_4 (mg/l)	PO_4-P (mg/l)
15/05/2015	Influent	11	38.4	12.52
	Denitrification	6	29.4	9.58
	Aeration	9	27.8	9.06
	Effluent	7	28.4	9.26
12/05/2015	Influent	5.25	15.2	4.96
	Denitrification	4	21.6	7.04
	Aeration	5	23.2	7.56
	Effluent	3	24.8	8.08
11/05/2015	Influent	3.5	24.8	8.08
	Denitrification	4	26.2	8.54
	Aeration	8	26.4	8.61
	Effluent	4	30	9.78
08/05/2015	Influent	2.75	31.9	10.39
	Denitrification	5	31.3	10.20
	Aeration	11	34.9	11.38
	Effluent	7	29.4	9.58
07/05/2015	Influent	2.5	37.9	12.36
	Denitrification	6	29.1	9.49
	Aeration	12	30.3	9.88
	Effluent	9	30.6	9.98
06/05/2015	Influent	2.25	40.4	13.17
	Denitrification	5	30.9	10.07
	Aeration	9	28.8	9.39
	Effluent	4	27.7	9.03
05/05/2015	Influent	6	66	21.52
	Denitrification	4	20.5	6.68
	Aeration	5	21.2	6.91
	Effluent	2	24.3	7.92
04/05/2015	Influent	4.25	352	11.48
	Denitrification	1	30.5	9.94
	Aeration	-	23.6	7.69
	Effluent	-	24.7	8.05
30/04/2015	Influent	3.25	33.2	10.82
	Denitrification	2.25	17.7	5.77
	Aeration	1,5	13.9	4.53
	Effluent	1.5	13	4.24
29/04/2015	Influent	3.25	33.2	10.82
	Denitrification	2.25	9.1	2.97
	Aeration	1.5	9.4	3.06
	Effluent	0.75	9.6	3.13

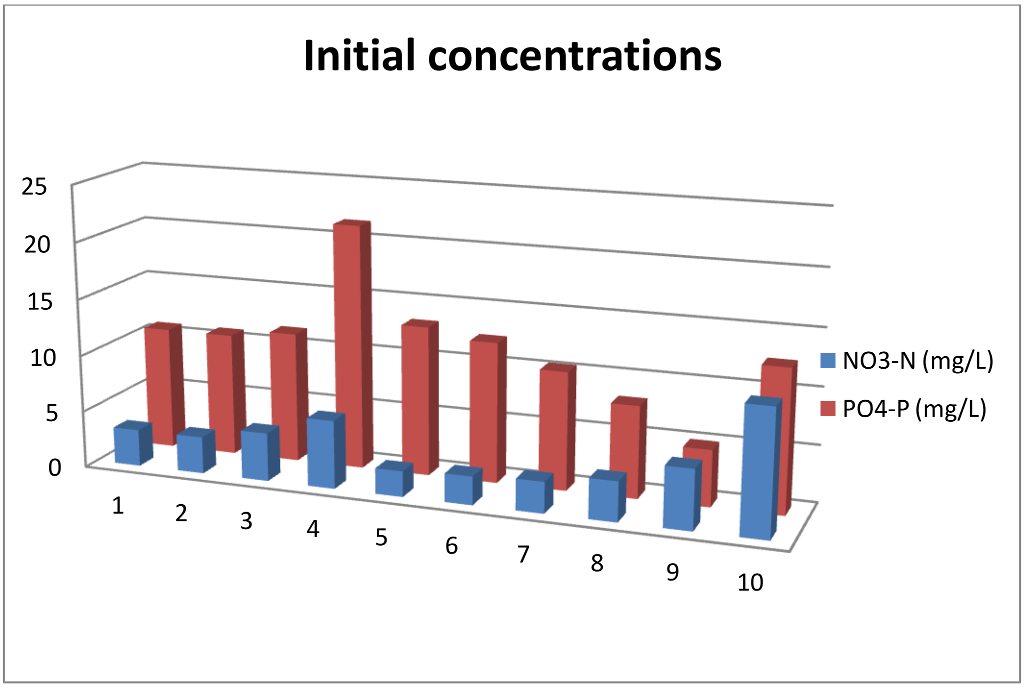
Once the values have been obtained, the percentage of removal is calculated and is shown in Table 3. As it can be seen in the following table, there are positive and negative percentages which mean if removal exists or not being a negative percentage that there is no removal.

Regarding to phosphates, in most experiments there is elimination, either higher or lower, while in the case of nitrates there are more variations between the results.

Table 3. Percentage of removal of nitrates and phosphates

Date	Removal NO_3-N (%)	Removal PO_4-P (%)
15/05/2015	36.36	26.04
12/05/2015	42.86	-63.16
11/05/2015	-14.29	-20.97
08/05/2015	-154.55	7.84
07/05/2015	-260.00	19.26
06/05/2015	-77.78	31.44
05/05/2015	66.67	63.18
04/05/2015	-	29.83
30/04/2015	53.85	60.84
29/04/2015	76.92	71.08

The initial conditions of phosphate and nitrate are shown in Picture 14. As it can be seen, the initial values are not completely the same, having some different magnitude, although uniformity is appreciated. Experiment 1 corresponds to date 29/04/2015 and so on until experiment 10 which corresponds to date 15/05/2015.



Picture 14. Initial NO_3-N and PO_4-P concentrations

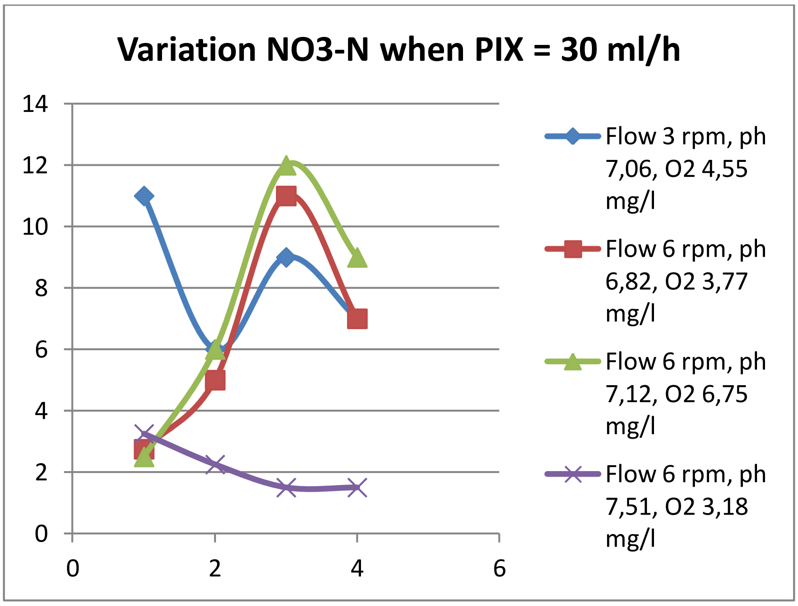
6 DISCUSSION AND CONCLUSIONS

Many experiments have been performed giving some results that are discussed below, but many more should be done to a greater understanding of the system.

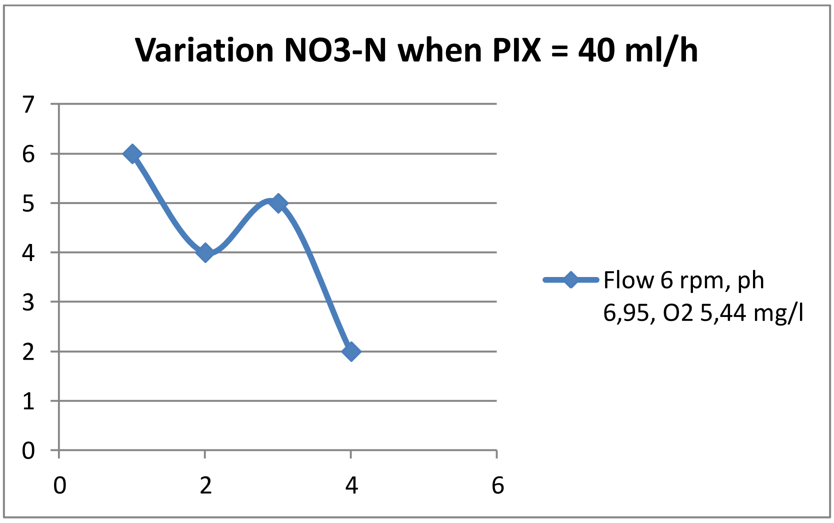
Although it cannot be measured or observed by any values presented, in any of the experiments, the increase of coagulant PIX used has direct results on the visible purification of the wastewater. The more coagulant used, the water is more transparent.

Oxygen must be regulated accordingly to prevent anoxic conditions in denitrification module and to maintain aerobic conditions in aeration/nitrification module. These anoxic conditions are given with an oxygen concentration below 0.4 mg/l. A special attention to this parameter is required since oxygen needs change as the process advances since there is an evolution of microorganisms. Initially microorganisms need a certain amount of oxygen and as they grow this amount changes being increased until stabilize.

According to Pictures 15, 16 and 17 nitrogen has variable results; it can be seen that there is no elimination of nitrates in some cases but it increases in others. This does not mean that there is no nitrogen removal, it is simply that the nitrate concentration varies.
This low correlation between what has to happen in theory, which is the nitrogen removal, and what happens, that is ambiguous and unrepeatable results, may be due to several reasons: first, it may be due to a unsatisfactory sample collection, not having collected a representative sample from the pilot plant; second, it may be caused by inefficient working conditions; third, it may be due to an improper use of methods of analysis.
In waste water treatment process, there are different forms of nitrogen and the concentrations of these different forms of nitrogen vary depending on where we measure.
The evolution of concentrations appear in the following graphs referred to numbers, these numbers correspond to different chambers of the pilot plant; nº 1 corresponds to influent, nº 2 is the denitrification chamber, nº 3 refers to aeration chamber and nº 4 is the effluent.

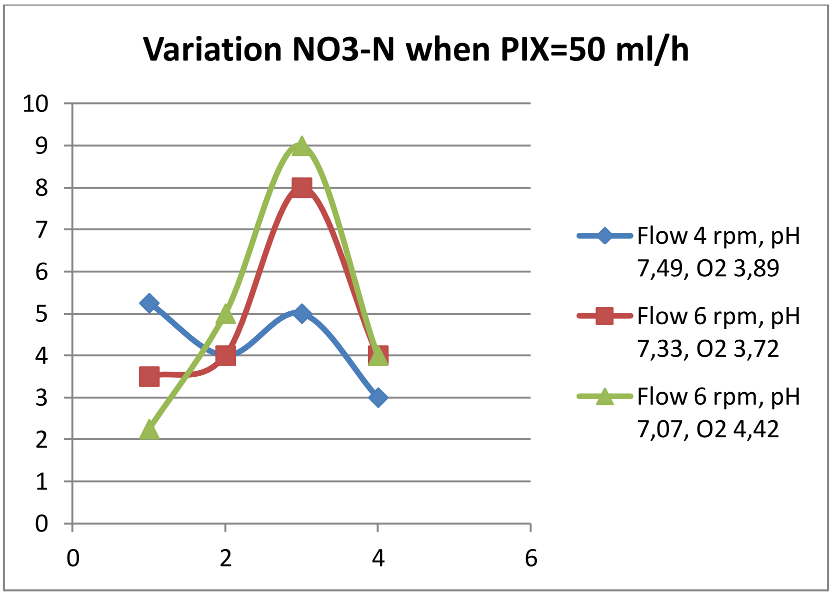


Picture 15. Tracing NO_3-N concentration when PIX=30mg/l.



Picture 16. Tracing NO_3-N concentration when PIX=40mg/l.

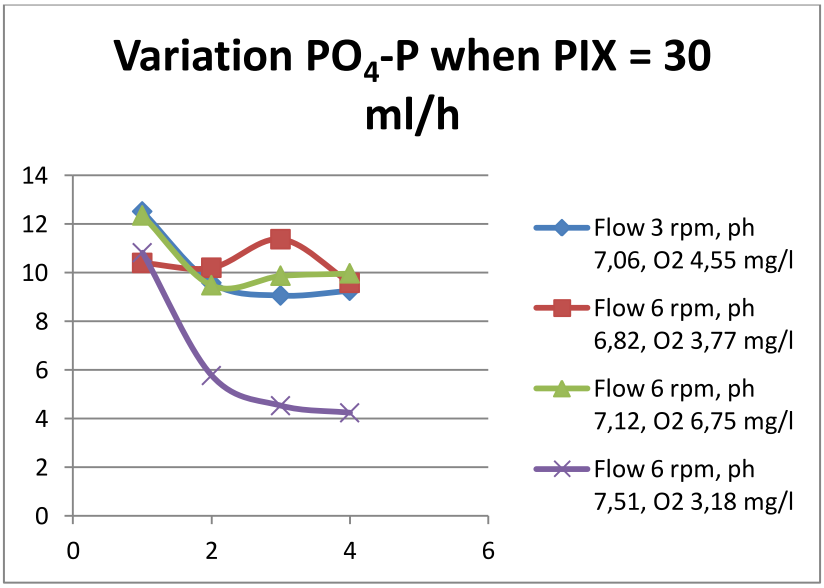
It can be observed the different tracing in each experiment.



Picture 17. Tracing NO_3-N concentration when PIX=50mg/l.

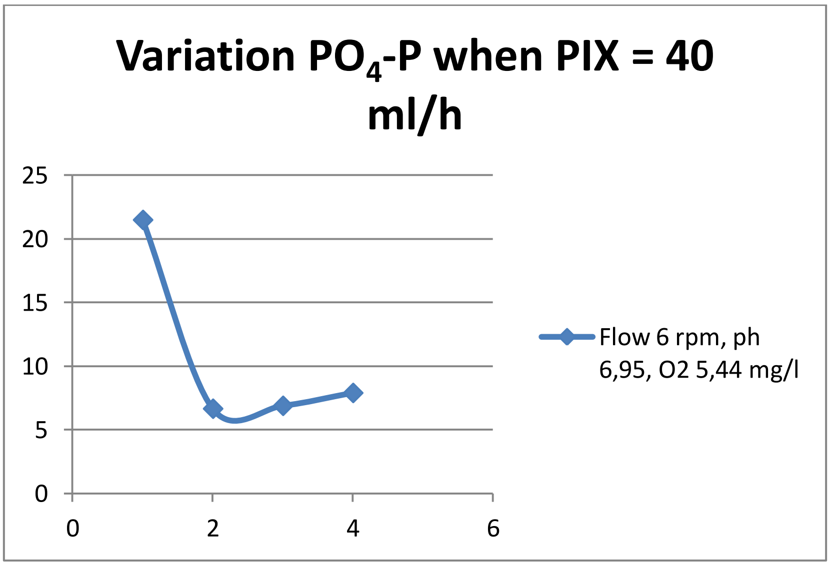
The longer retention time of the wastewater in the pilot increases the clarity of the result water, this is because microorganisms have more time for the process.

As it is shown in Pictures 18, 19 and 20, in most experiments, phosphorus removal has been carried out with adequate success. In contrast to the case with nitrogen, there is no possible confusion with the forms of phosphorus so the system works properly.

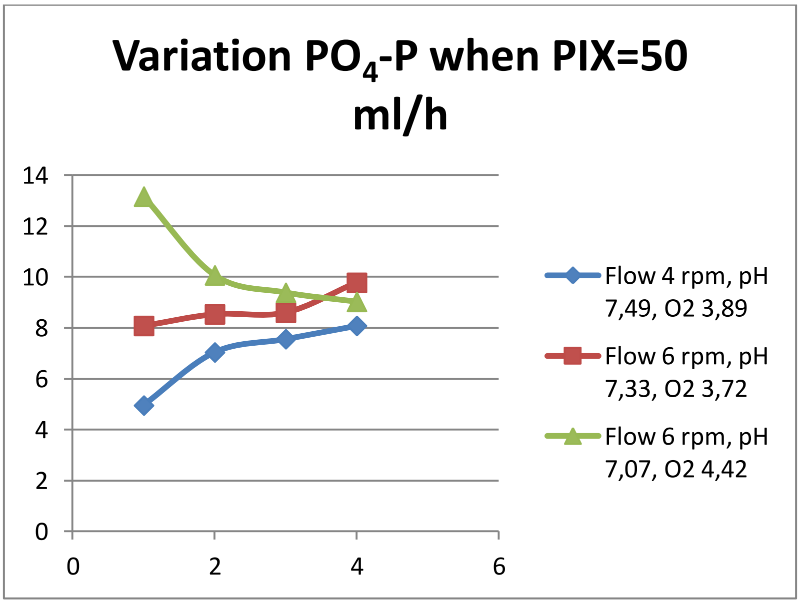


Picture 18. Tracing PO_4-P concentration when PIX=30mg/l.

Among the small differences in working conditions, a better removal is achieved when pH is around 7 and 7.5, and an oxygen concentration between 3.5 and 5 mg/l.



Picture 19. Tracing PO_4-P concentration when PIX=40mg/l.



Picture 20. Tracing PO_4-P concentration when PIX=50mg/l.

This experimental project should be continued. As it is said before, nitrogen results are ambiguous, this is because only nitrate has been measured. Nitrate has been measured but not ammonia or total nitrogen so we do not have a clear impression of how the system is able to remove the different forms of nitrogen in each step.

To avoid confusions it is necessary to measure ammonia or total nitrogen. Other methods of analysis and more data and measurements are required.

Regarding to phosphorus, good results about removal percentage are achieved, however as in the case of nitrogen, more information is needed so more experiments are required.

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