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WASTEWATER TREATMENT OPTIONS FOR A PALM OIL INDUSTRY IN SIERRA LEONE

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Abstract

Palm oil production and consumption worldwide increased from 1 to 58 million tons over the last 40 years. This huge boost resulted in a positive economic transformation of underdeveloped tropical countries, mainly Malaysia and Indonesia, into newly industrialised market economies. The fast expansion of palm oil plantations also created ecological and social problems. To mitigate issues associated with this exponential growth, the Roundtable for Sustainable Palm Oil (RSPO) was founded to promote sustainable social, ecological and economical practices in the oil palm industry. One of the major challenges for oil palm industries is waste management. The majority of the solid waste is burned into boilers, and converted to renewable energy. The treatment of wastewater with high chemical oxygen demand (COD) concentration is more challenging, due to the large quantities produced.

In this thesis, a case study focusing on the waste management of an operational plantation in Sierra Leone is evaluated. Three innovative palm oil mill effluent (POME) treatments (dissolved air flotation (DAF), anaerobic digestion and sequence batch reactors (SBR)) were tested on lab scale. A new waste management approach was proposed to optimise waste treatment and energy recovery.

During the DAF treatments, the effect of different coagulants, flocculants and pressured water quantities on POME (raw and digested) were investigated. Via the anaerobic digestion treatment, the maximum amount of biogas produced per gram volatile solids of POME was analysed. The fed batch treatment was included to determine the biogas yields when feeding was done intermittently under semi-continuous conditions, over a period of 50 days. It was possible to obtain a > 90 % COD removal efficiency with raw POME using the DAF, however, the amount of chemicals needed to achieve this made it economically unfeasible. The coagulation worked best at lower temperature (< 30 °C). The DAF treatment obtained a COD removal rate of > 80 % with digested POME. The AD treatment showed a COD conversion efficiency to methane of 53 % after 21 days. During the fed batch reactor treatment, a 40 % COD conversion to methane was observed. This research indicates that a DAF treatment as pre-treatment before a covered anaerobic lagoon (CAL) is not advisable, due to the high amount of chemicals needed and reduced biogas production at the anaerobic digestion stage. The DAF as a post treatment after the covered anaerobic lagoon achieved a COD below 250 mg/L, matching required international standards, with 73 % less chemical use than DAF as a pre-treatment. Further research is needed for more efficient DAF treatment, and to review the implementation of a cost effective SBR system.

Samenvatting

De productie en consumptie van palmolie steeg werelwijd van 1 tot 58 miljoen ton over de laatste 40 jaar. Deze enorme expansie resulteerde in een positieve economische transformatie van onderontwikkelde tropische landen, vooral Maleisië en Indonesië, tot nieuwe ge-industrialiseerde markt economieën. De snelle uitbereiding van palmolie plantages creerde echter ook ecologische en sociale problemen. Om deze exponentiele groei te controleren, is de Roundtable of Sustainable Palm Oil (RSPO) opgericht om duurzame sociale, ecologische en economische praktijken in de palmolie industrie te promoten. Eén van de voornaamste uitdagingen voor de palmolie industrie is het beheer van het geproduceerde afval. Het merendeel van het vaste afval wordt verbrand in verbrandingsovens waar groene energie wordt opgewekt. De behandeling van afvalwater met een hoge chemical oxygen demand (COD) is meer problematisch, aangezien er grote hoeveelheden van worden geproduceerd.

In deze thesis wordt een case study geëvalueerd die focust op afvalbeheer van een operationele plantage. Drie innoverende POME behandelingen (dissolved air flotation (DAF), anaerobe vergisting en sequence batch reactoren (SBR)) werden getest op labo schaal. Een nieuwe aanpak voor afvalbeheer optimalisatie en hernieuwbare energie werd voorgesteld.

Tijdens de DAF experimenten werd onderzoek gedaan naar het effect van verschillende hoeveelheden coagulanten, flocculanten en de waterdruk op POME (rauw en verwerkt). Aan de hand van anaerobe vergisting experimenten werd de maximale hoeveelheid biogas geproduceerd per gram organische droge stof van het POME geanalyseerd. Het fed batch experiment werd uitgevoerd om de biogas productie te bepalen wanneer POME alternerend werd toegevoegd onder semi-continue condities voor 50 dagen. Meer dan 90 % van de COD kon worden verwijderd uit het rauw POME, alhoewel de hoeveelheid chemicaliën die hiervoor gebruikt werd niet economisch haalbaar was. De coagulanten werkten ook beter bij lagere temperaturen (< 30 °C). Het DAF experiment kon meer dan 80 % van de COD verwijderen voor reeds verwerkte POME. Het anaerobe vergisting experiment bereikte een COD conversie naar methaan met een efficiëntie van 53 % na 21 dagen. Tijdens het fed batch reactor experiment werd een COD conversie naar methaan genoteerd van 40 %. Deze thesis toont aan dat DAF als voorbehandeling voor een afgedekte anaerobe lagune (CAL) af te raden is, wegens de grote hoeveelheden chemicaliën nodig. De DAF inzetten na een gesloten anaerobe lagune bereikte goede resultaten, met effluent waarden die onder de vereiste internationale waarde blijven, maar de chemicaliën kost was nog altijd (te) hoog. Extra onderzoek is vereist om tot een meer kostefficiënt DAF en SBR systeem te komen.

List of abbreviation

ATU	Aerobic treatment unit
BMP	Biochemical methane potential
BOD	Biological oxygen demand
CAL	Covered anaerobic lagoon
CD	Charge density
COD	Chemical oxygen demand
DAF	Dissolved air flotation
EFB	Empty fruit bunch
FFB	Fresh fruit bunch
GHG	Greenhouse gas
HRT	Hydraulic retention time
KCP	Kernel crushing plant
MW	Molecular weight
NTU	Nephelometric turbidity unit
OLR	Organic loading rate
PAC	Poly aluminium chloride
PKC	Palm kernel cake
PKS	Palm kernel shell
POME	Palm oil mill effluent
PPF	Palm pressed fibres
RSPO	Roundtable on sustainable palm oil
SBR	Sequence batch reactors
TS	Total solids
TSS	Total suspended solids
UASB	Upflow anaerobic sludge blanket
VFA	Volatile fatty acids
VS	Volatile solids
VSS	Volatile suspended solids

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1. Introduction

a) The oil palm industry

The annual production of palm oil worldwide is 58 million tons, planted over 14 million hectares (FAO 2014). Oil palm is the most efficient oil bearing crop in the world. It can produce up to 3.8 tons of oil per hectare, which is 9 times more than soya and 6 times more than sunflower (Marti 1999). Because of its low production cost, palm oil is the most consumed vegetable oil. It is found in about half of all packaged products sold in super markets from the biscuit to the soap aisle (Spinks 2014).
Malaysia and Indonesia are producing 86 % of the total palm oil production worldwide (FAO 2014). Currently, these countries are reaching a land limitation, which guides the focus of the oil palm industry to new continents, like Africa and South America. Although oil palm is originating from West Africa, the production of the continent is minimal with only 2 million tons per year, which corresponds to 4 % of world production of palm oil (FAO 2014). However, Africa possesses a lot of non-cultivated land that constitute a huge potential to plant more palm trees.

With the fast expansion of palm oil production over the last 40 years, ecological and social problems emerged. There is a major concern about the lack of sustainable practices rising from the deforestation and usage of peat soil in Asia (Rival & Levang 2014). In Africa, “land grabbing” practices of palm oil producers and other community issues have caused many frictions with local farmers, which increased the critical involvement of environmental organisations. Referring to the Asian example, these organisations have successfully raised the international public awareness, demonstrating that the uncontrolled industrial expansion of oil palm not only leads to environmental catastrophes, but also towards intensification of social and economic problems. Nevertheless, palm oil cannot be excluded from the current consumer market, on the contrary, the worldwide demand for palm oil is expected to further increase to 240 million tons by 2050 (Campbell et al. 2016). In response to these challenges, the oil palm sector has created the Round table for Sustainable Palm Oil (RSPO). The RSPO is a multi-stakeholder certification body that aims at a better balance between social, ecological and economic practices. With 3,000 members worldwide representing sectors along the palm oil supply chain, it insures that the large scale palm oil buyers, such as Nestlé, L’Oreal and Unilever, commit to sustainable palm oil by deciding to only purchase palm oil from industries that have been RSPO certified (Mazzoni 2014). In 2016, about 17 % of all palm oil producers worldwide are RSPO certified, following its principles and criteria of social engagement towards local population, no deforestation on high biodiversity plots and waste management to ensure sustainable production.

One of the main challenges to achieve a sustainable, ecological palm oil production is the controlled discharge of the waste products. Approximately 70 % of the harvested fruit bunch is waste (Prasertsan & Prasertsan 1996). Palm oil waste production can be categorized as solids waste, which includes empty fruit bunches (EFB), palm pressed fibres (PPF), palm kernel shells (PKS) and palm kernel cake (PKC), and as liquid the palm oil mill effluent (POME). In 2008, Malaysia alone produced 44 million tons of POME (Wu et al. 2010). The environmental degradation caused by the improper disposal of POME is surface water and soil pollution. Consequently, waste management techniques are indispensable to minimize the severe environmental impact that accompanies the fast expansion of oil palm plantation in the tropics.

b) The issues with oil palm production

Deforestation

Extensive industrial palm planting occurs at the expense of biodiversity and ecosystems. Animals are injured, killed or displaced during deforestation, and the loss of their habitats can lead to their extinction. While palm oil production provides employment to many people, it is also, in some cases, destroying the forest land that local populations depend on, sometimes leaving no choice to these communities but to become plantation workers (Marti 1999).

Waste management

The production of palm oil leads to an important quantity of waste. Among the waste are the solids, which include: PPF, PKS, PKC and EFB, accounting for respectively 30, 6, 3 and 28.5 % of the Fresh fruit bunch (FFB) (Pleanjai et al. 2004).

The milling process also generates wastewater through sterilization of fresh oil palm fruit bunches, clarification of palm oil and effluent from hydro-cyclone operations (Borja et al. 1996). The wastewater (POME) is produced at the rate of 0.675 m^3 per tons of FFB processed (Baharuddin et al. 2010). It is considered a major source of water pollution, because of its high chemical oxygen demand (COD), biological oxygen demand (BOD), total suspended solids (TSS) and turbidity. As shown in Table 1, POME is also acidic and discharged at high temperature. If discharged untreated into waterways, it can be particularly harmful to aquatic organism, contaminate drinking water and cause eutrophication.

Table 1 : Palm oil mill effluent general characteristics from three different studies (Tabassum et al. 2015; Rupani et al. 2010; Abdullah et al. 2013)

Parameters	Units	Literature values[1]
pH	-	3.8 - 4.7
Temperature	°C	80 - 90
COD	mg/L	50,000 - 75,000
BOD	mg/L	25,000 - 27,000
Oil & Grease	mg/L	4,000
Total solids	mg/L	40,500 - 100,000
Total volatile solids	mg/L	24,000 - 50,000
Total suspended solids	mg/L	18,000 - 50,000

c) Existing waste management techniques in oil palm industries

Waste management of solid waste in oil palm industries includes mainly waste to energy or fertilizer techniques. Fibres and shells have high dry matter content and are, therefore, used in a biomass boiler as fuel for steam production (Sing & Aris 2013). The empty fruit bunches (EFB) on the other hand, are a poor fuel, because it contains 60 – 70 % of moisture that leads to incomplete combustion and flue gases emission problems (Kim et al. 2013).

The treatment of the POME includes biological treatments, both anaerobic and aerobic, but also physicochemical treatments.

Open pond system is a conventional and widely spread anaerobic treatment system. In Malaysia, 85 % of palm oil producing companies have adopted ponding treatment (Wu et al. 2010). The system consists of multiple ponds connected in series. First, the effluent remains in an acidification pond for 4 days, then the resulting treated effluent overflows into the following pond, and after an averaged hydraulic retention time (HRT) of 45 days it can be further treated in a series of facultative ponds (Wu et al. 2010). The installation of this system is low-cost, but it also has a number of disadvantages, such as the vast area each pond occupies, the lack of operational control, the long HRT requirement and the regular presence of solid accumulation and short circuiting (Wu et al. 2010). The anaerobic degradation occurring in open air pond leads to emissions of carbon dioxide (CO_2), methane (CH_4), a gas with a GHG potential 25 times higher than CO_2, and hydrogen sulphide (H_2S), which is a

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[1] The ranges given are based on values reported in three studies on POME by Tabassum, Zhang, and Zhang 2015; Rupani et al. 2010; Abdullah et al. 2013

source of odour (USEPA n.d.). The increased public awareness on environmental pollution along with governmental or international pressures require the palm oil industry to treat the POME with respect to the parameters listed in Table 2. Open pond systems often fail to meet discharge limits for discharging into rivers, such as a BOD concentration of 50 mg/L (Chin et al. 1996)

Table 2: Standard discharge limits for effluent from vegetable oil processing set by the International Finance Corporation

Parameter	Unit	Guidelines values
pH	-	6 - 9
BOD_5	mg/L	50
COD	mg/L	250
Total nitrogen	mg/L	10
Total phosphorous	mg/L	2
Oil and grease	mg/L	10
Total suspended solids	mg/L	50
Temperature increase	°C	< 3

Covered anaerobic lagoons (CAL)

An advantage of CAL over open ponds is the capture of methane that can be used as fuel in a biomass boiler reducing the oil palm production costs. The steam produced by the boiler, as a result of burning biogas, can be used directly in milling operation or sent to a turbine to produce electricity. Palm oil mills in Africa are generally located in regions with no electricity provider, and the industry itself has the possibility to provide electricity to the local population. In these cases, the capture of biogas can generate considerable savings in fossil fuels.

The anaerobic pond digestion of POME has resulted in COD removal efficiencies as high as 98.7 % (Yacob et al. 2006). However, starting with an influent COD concentration of 67,000 mg/L, a 97.8 % removal only results in an effluent COD of 1,474 mg/L and another 83.0 % additional removal is needed to reach the 250 mg/L COD permitted discharge.

Aerobic treatment

In an engineered aerobic treatment, oxygen is supplied to aerobic micro-organisms that consume the organic matter contained in the wastewater as part of their metabolic activity, resulting in the degradation of the organic compounds into inorganic compounds such as NH_3, NH_4, NO_3, SO_4 and PO_4 (Buchanan & Seabloom 2004). Similarly to an anaerobic treatment, a favourable environment (temperature, pH, dissolved oxygen) must be provided to the microorganism to achieve a high rate of removal of organic pollutants. In general, as temperature and dissolved oxygen increase, microbial activity increases (Buchanan & Seabloom 2004). Although, organic wastewater is treatable at a wide pH range, the ideal pH for aerobic microbial growth is 6.5 - 7.5 (Benefield & Randall 1981). Aerobic treatment unit (ATU) configurations include extended aeration, suspended growth bioreactors, attached growth bioreactors, rotating bioreactor contactors, and sequence batch reactors (SBR). The ATU's are considered more efficient than CAL units at treating POME, because they can accomplish the same removal efficiencies at a lower HRT, that is 95 % removal in 10 - 14 days vs 20 days in anaerobic treatment (Wu et al. 2010). For the treatment of POME, aerobic treatment could also be a post-treatment to the anaerobically treated POME, as CAL systems alone fail to reduce the COD to discharge standards. An aerobic digester used as a post-treatment of different anaerobically digested POME can be effective for COD, TSS and volatile suspended solids (VSS) removal, when operated at a HRT up to 7.2 days (Chou et al. 2016). The ATU's do not only reduce the COD by aerobic digestion, they also contribute to the conversion of ammonia by nitrification, and have additional chambers for the removal of nutrients, suspended solids and pathogens from the effluent (Buchanan & Seabloom 2004).

Dissolved air flotation

In a recent study, Faisal, et al. (2016) evaluated the effect of combining baffled air flotation with an anaerobic membrane bioreactor to treat POME. The initial COD content of the POME was approximately 20,000 mg/L. The air flotation system was used as a pre-treatment for the removal of suspended solids and fat/oil, thereby increasing the performance of the membrane by reducing the occurrence of clogging. The study reports that the air flotation process achieved a COD removal efficiency of 35.5 % at an HRT of 5 days and an air flow rate of 0.66 m^3/h. The effluent was subsequently passed through an anaerobic membrane bioreactor and the recorded total (air flotation & membrane) COD removal efficiency was 97 %. Membranes are advantageous, because an effluent of higher quality is obtained in a shorter time period and smaller area then CAL systems (Faisal et al. 2016). On the other hand, treating a wastewater high in suspended solids such as POME with membranes may result in a decreased membrane life and high maintenance costs due to the frequent clogging (Metcalf & Eddy 2003).

Evaporation and adsorption

The treatment method of evaporation and adsorption is based on the physical and chemical characteristics of POME. It consists of boiling POME and collecting the vapour and the concentrated solution. A study by Kathiravale & Ripin (1997) reported applying a vacuum pressure of 0.47 bar to reduce the boiling point of POME to the temperature when exiting the mill (80 – 85 °C), thereby avoiding the addition of heat. The vapour produced is passed through an absorption column for the treatment of the distillate. Depending on the absorbent (synthetic Zeolite or natural) the COD removal efficiency is in the range of 60 – 40 %. The method is advantageous, because it leads to the recovery of 80 % of the water (equivalent POME volume reduction) and the concentrated effluent analysis showed a high content of nitrogen, phosphorus and potassium which can be used as feed material for the making of fertiliser (Kathiravale & Ripin 1997). However, with this method the investment and operational costs can become high, considering the prices of distillation and absorption columns and, especially, the energy requirement.

d) Proposal for the improvement of Goldtree wastewater treatment

The objective of this study is to recommend options for the improvement of Goldtree’s wastewater treatment. The challenge is to propose a system that is mechanically robust, not sensitive to unexpected circumstances, and not too technologically advanced, while still meeting the discharge COD limit of 250 mg/L. Avoiding a high level of technological sophistication is necessary to keep not only the investment cost low but also operation and maintenance routines as simple as possible.

This proposal focuses on building units in series as follows:

- A dissolved air flotation (DAF) unit upstream to remove the majority of suspended solid reducing the COD content to an expected 90 % (Figure 1). The efficiency of removal of the suspended solids in fresh POME has to be evaluated, but considering that the temperature of the incoming wastewater is 60 – 70 °C, it is also necessary to investigate the effect of temperature on coagulation/flocculation processes to propose cooling towers if needed. While the floating sludge blanket is collected and sent to the plantation as fertilizer, the liquid phase is sent to a covered anaerobic lagoon.
- A covered anaerobic lagoon (CAL) to further treat the liquid phase of DAF and recover biogas that can either be flared or sent to the mill biomass boiler. Here, a biomethane potential (BMP) test is essential to evaluate the fraction of ingoing COD in POME that is converted to methane under optimum condition whereas a fed batch reactor test is

necessary to evaluate the biogas production under normal conditions of temperature and feeding. Estimating the biogas production is useful for the calculation of potential savings in the production of electricity associated with running the biomass boiler followed by a turbine on biogas rather than a generator on fossil fuel.

- A sequence batch reactor (SBR) would only follow the covered lagoon if the COD of the anaerobic digestion effluent is still above the target discharge limit.

The most suitable location to build a new wastewater treatment would be located close to the current system. It is ideally located to:

- Receive POME directly from the oil recovery tank via gravity
- Send the DAF skimmed sludge as well as excess of anaerobic sludge to the plantation
- Send the biogas directly to the biomass boiler at the mill

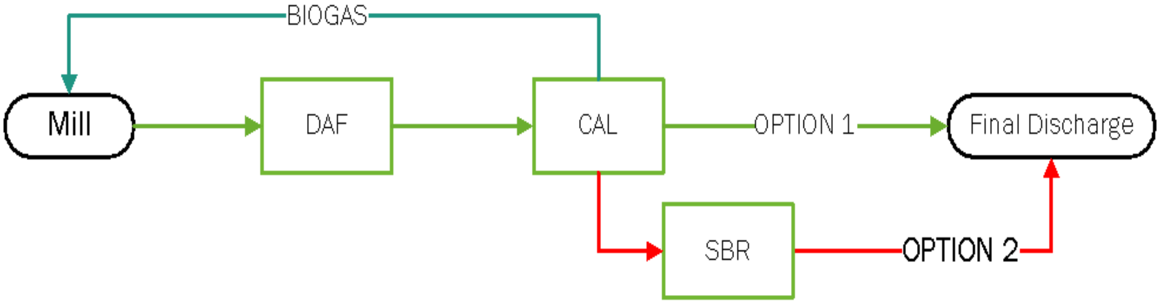

Figure 1: Proposal 1. Flow diagram of first proposed wastewater treatment plant. The DAF unit is placed upstream to decrease the suspended solid content by 90 %, The liquid phase of the DAF is send to a CAL for further treatment and capture of the biogas. The SBR installation is optional, and only necessary if after the CAL installation the COD level remains above the target COD concentration of 250 mg/L.

Considering that more biogas can be recovered if the POME is not pre-treated with air flotation, an additional option would be to use a DAF unit as a post treatment (Figure 2).

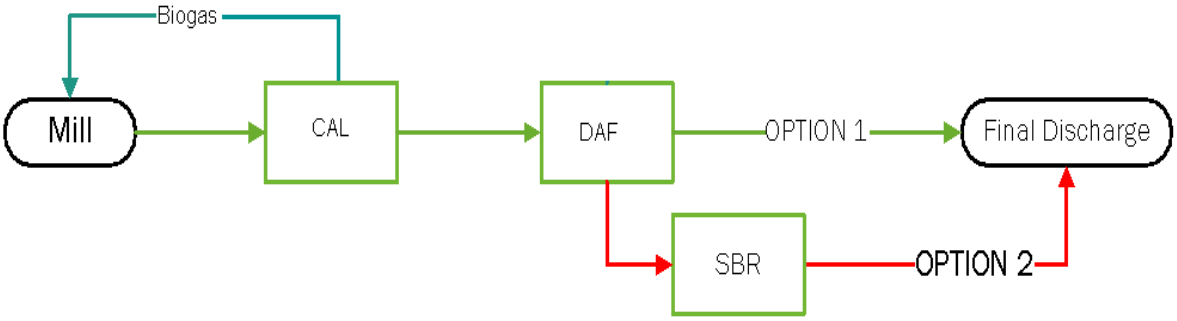

Figure 2: Proposal 2. Flow diagram of an alternative option where the DAF unit is used as a post-treatment to anaerobic digestion to allow higher biogas recovery. The SBR installation is optional, and only necessary if after the CAL installation the COD level remains above the target COD concentration of 250 mg/L.

2. Case study

a) Goldtree

Goldtree is a fully integrated agro industrial site, including a rehabilitated oil palm plantation and mill located in Daru in the Eastern Province of Sierra Leone. The rainfall in the region is favourable for oil palm trees, with a yearly average temperature and precipitation of 28.5 °C and 2,300 mm, respectively (Goldtree Ldt. 2016, personal communication, September 3).

Goldtree plantation consists of 1,500 hectares, with a potential crop production of +20,000MT FFB. Additionally fruits are purchased from surrounding local farmers spread over 5,000 hectares. The crop yield distribution, which is directly linked with waste production, follows a peak (January - June) and lean season (July – December). Up to 70 % of the annual crop will be harvested and processed during the first 6 months of the year (Figure 3).

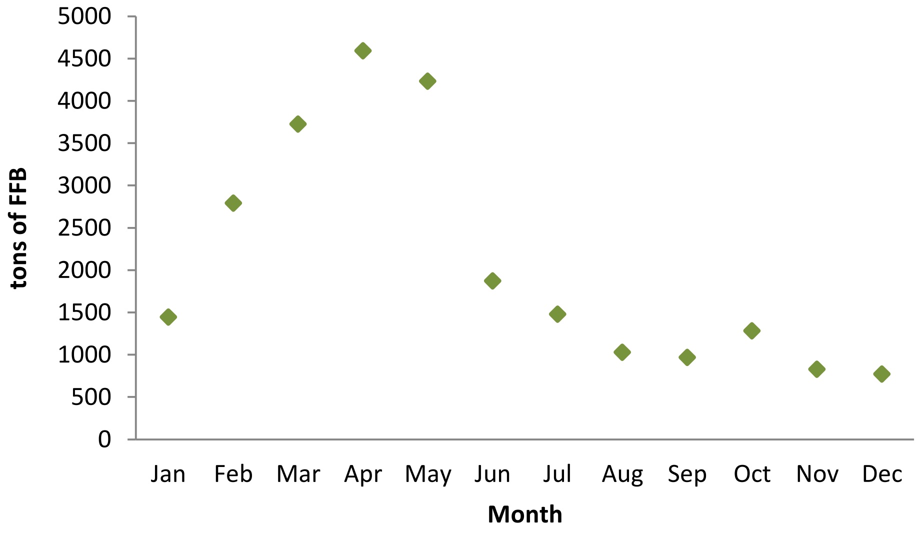

Figure 3 : Goldtree processed palm fruit bunches (MT) in 2015. It shows an increase in crop production from January to June with a peak in March and the start of the lean season in July lasting until December.

For the next 10 years, Goldtree foresees a steep increase in production of fruit from its own estates due to the expansion from 1,500 planted hectares to 3000 hectares (Table 3). There will also be an increase in fruits purchases form local farmers that will be benefitting from a World Bank program to expend by additional 1,000 hectares (Goldtree Ldt. 2016, personal communication, October 7). With the increase in crop yield, an increase of crude palm oil (CPO) and POME production will take place, as well as higher energy needs.

Table 3: Projected fruits yield from 2014 to 2025.

Year	**2014**	**2015**	**2016**	**2017**	**2018**	**2019**	**2020**	**2021**	**2022**	**2023**	**2024**	**2025**
TFF	8,907	11,649	16,062	19,150	22,292	26,924	38,768	48,775	60,882	74,114	82,455	90,110

Goldtree palm oil mill is equipped with a CPO extraction process (20 T/h milling capacity) design with vertical sterilisers and presses for extraction. It also has a kernel crushing (3 T/h capacity) and bottling plant (Figure 4).

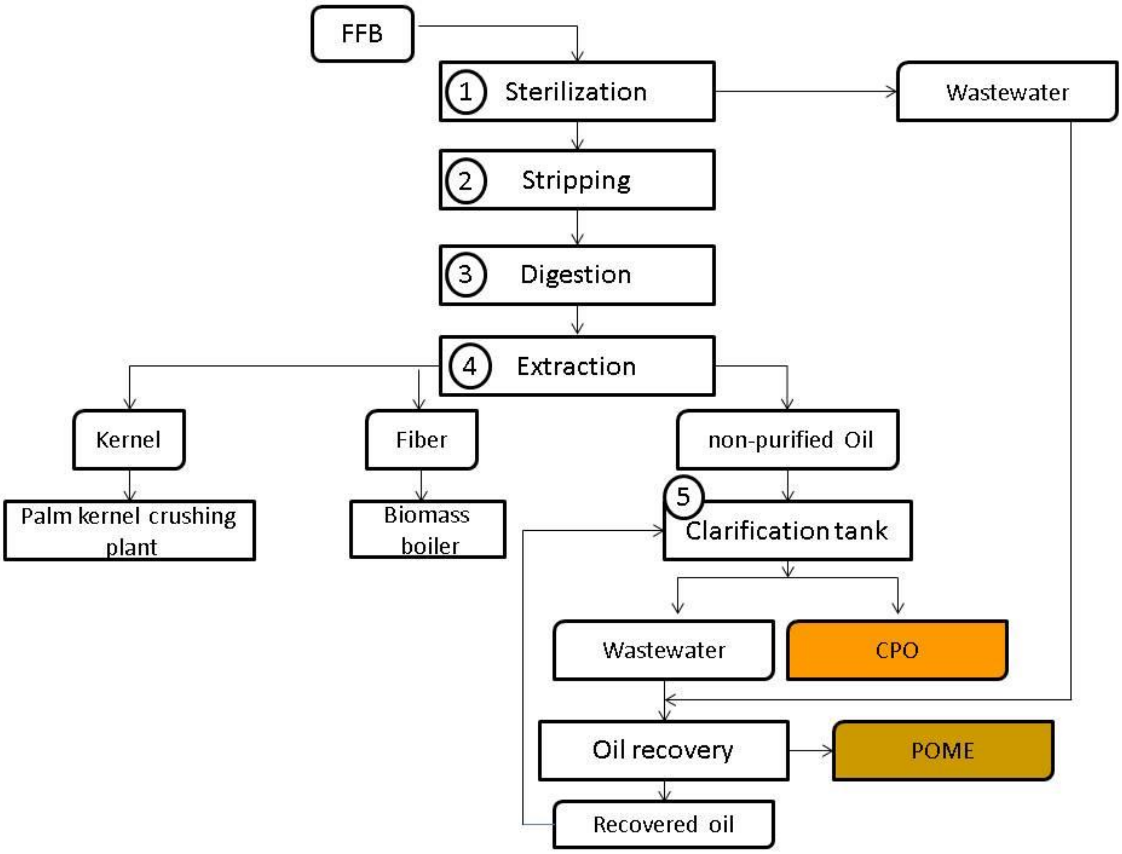

Figure 4: Goldtree mill flowchart describing the processing of FFB and production of POME. The major processes steps are: 1- Treatment of FFB at high temperature and pressure steam for disinfection and for improving detachability of the fruits from the bunch. 2- Mechanical separation of the fruits from the bunch. 3- Reheating the fruits to 80 – 90 °C to loosen the mesocarp from the nuts. 4- Digested fruit is pressed without breaking the kernel using the screw press. 5- Oil purification.

The mill is not connected to the grid but has a biomass boiler manufactured by BOILERMECH, with the capacity of 12 tons of steam per hour at 20 bar. This boiler is able to combust fibres, shells and, EFB. The boiler supplies steam to both the mill heating system and a 0.6 MW back pressure turbine that services the mill, the kernel crushing plant (KCP), the local administration and housing estate. When the mill is not in operation, the rest of the estate relies on a back-up power diesel generators (200 kW and 400 kW).

A decanter is used for oil recovery from wastewater, and POME is sent from the decanter to the wastewater treatment system. The current effluent treatment system consists of a series of 4 open ponds (Figure 5). Pond 1 presents clear signs of acidification and overloading with banks collapsing. Pond 2 and 3 show signs of sludge saturation. The inlet connection is a temporary piping and the final discharge is done by pumping the content of pond 4 in the nearest sections of the plantation.

Figure 5: Goldtree pond system. The picture on the left hand corner is a bird-eye view showing 3 of the 5 ponds in use. It also shows that there is no connection to a final discharge, which is actually performed by an external pump discharging into the plantation, as shown in the top right corner. The inlet to the ponds is also a temporary installation, as shown in the top further right. The bottom right show signs of acidification and overloading.

The COD data of each pond listed in Table 4 shows that the pond system can remove 98 % of COD. Nonetheless, Goldtree production in 2016 was only 16,062 tons of fresh fruits (TFF), which implies that the high removal rate is only due to a high retention time. With the existing plans to increase production by 500 % in 2025, the effect of retention time will be lost, and the effluent treatment system will have to be improved to meet the required effluent standards.

Table 4: Average effluent COD concentrations of samples collected from all 4 ponds of Goldtree pond systems over 2 years. Sampling and COD analysis are performed twice a year.

Location	**COD (mg/L)**
Pond 1	65,088 ± 11,367
Pond 2	16,264 ± 324
Pond 3	1,712 ± 858
Pond 4	1,052 ± 316

b) Dissolved air flotation

Physico-chemical treatment methods, such as membrane separation, have been applied to anaerobically treated POME with significant removal efficiency of suspended solids (Faisal et al. 2016). Other approaches, such as flocculation and electroflotation, on anaerobically treated POME also showed high removal efficiencies of 97 % (Poh et al. 2014).

Dissolved air flotation (DAF) units are one of these physico-chemical techniques used in wastewater industries, mainly for the removal of suspended solids. In a DAF system micro bubbles (diameter less than 100 µm) are created by dispersion water or high-pressure (40 – 70 mwp) water saturated with dissolved air (Kiuru 2001).

The DAF tank is divided into two zones a contact zone and a separation zone and a baffle divides the two (Figure 6). The purpose of the contact zone is to provide opportunity for collisions and attachment of floc particles and air bubbles while in the separation zone the micro-bubbles rise to the top, carrying the suspended particles present in the water (Edzwald 2010). It results in the formation of a sludge blanket at the surface of the tank, which is then skimmed off while the clarified water is withdrawn from the bottom of the tank. The clarified water is also partly recycled back to a saturator where air under pressure is dissolved into the recycled flow. The total amount of air delivered to the contact zone depends on the saturator pressure and the recycle flow (Edzwald 2010). Depending on the equipment used for the injection of air to the water, there is a specific size distribution of the micro-bubbles and the most recent equipment achieves a micro-bubble diameter lower than 100 µm (Kiuru 2001).

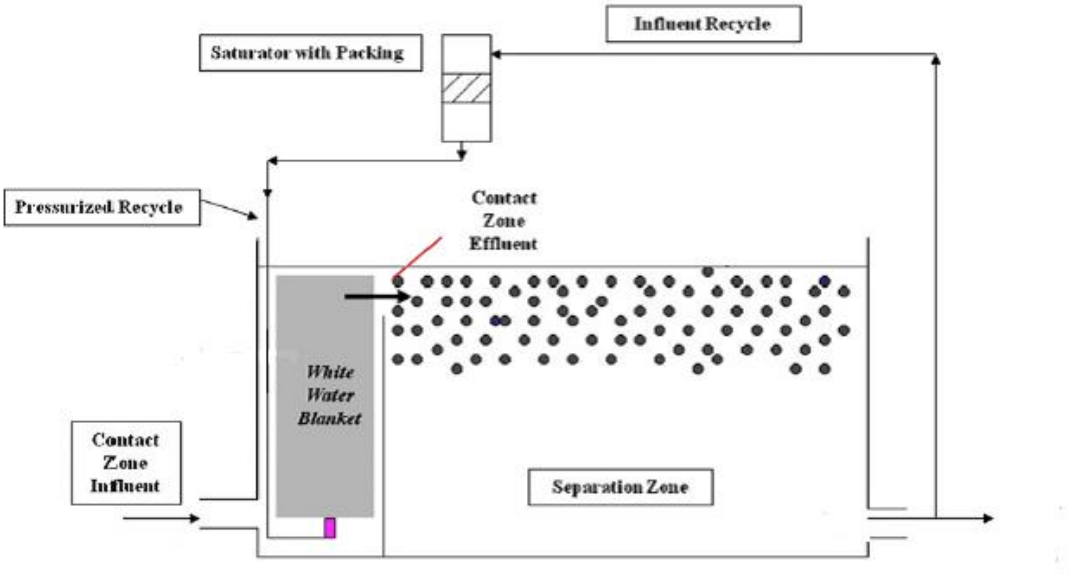

Figure 6: Separation and contact zone in DAF units (Edzwald 2010)

The DAF units with micro-bubbles in the order of 100 μm and less coupled with coagulant have a removal efficiency of more than 90 %, with the efficiency improving with decreasing bubble size, since it leads to larger surface area and longer HRT (Poh et al. 2014). Poh, et al. (2014), reported that the micro-bubbles alone can remove a significant amount of organic compounds or oil and grease (O&G), without the addition of any other chemicals. In this study, a centrifugal pump and an ejector type venturi device were used to generate micro-bubbles. The pump was operated at three different under different bubbling times (i.e. 2.5, 5, 10 and 12.5 min) to investigate the effect of bubbling time on the efficiency of contaminant removal and for a bubbling time of 12.5 min the COD content was reduced by 53.7 %. No chemicals addition is environmentally advantageous but also cost effective. However, in this same study, Poly-Aluminium Chloride (PAC) coagulant was added after micro-bubble flotation to meet COD discharge values which in agreement with a previous study by Van Le et al. (2012) demonstrating that the addition of coagulant was necessary to obtain an efficient removal of contaminants in anaerobically digested POME.

The addition of chemical coagulants in the inlet compartment of a DAF unit, in the presence of micro-bubbles of air, results in a highly efficient separation process since floatability increases with the size of the particles which is enhanced with various chemical coagulants (Wang et al. 2005). Metal coagulants based on aluminium and iron are commonly used in wastewater treatment plant (aluminium chlorides, ferrous sulphate, aluminium sulphates or ferric chlorides (Ødegaard 2001). Metal coagulants can destabilize and aggregate colloids using different mechanisms.

Electrostatic coagulation

It is the process where metal salts are applied to the stable colloidal suspension so that the counter ions (positively charged) cause neutralization of the particles (negatively charged). However, increasing the amount of salts past the zero zeta potential will lead to a shift to positively charged particles that become once again stable in suspension (Van Nieuwenhuijzen 2002).

Precipitation coagulation

Precipitation or sweep coagulation requires a higher coagulant dose than charge neutralization where excess leads to the formation of metal coagulant precipitates (*e.g.* $Al(OH)_3$ or $Fe(OH)_3$) dragging down the suspended particles (Van Nieuwenhuijzen 2002).

Polymers can also be used to destabilize colloidal particles. In wastewater treatment they are mainly of synthetic nature, and can be categorized as cationic, anionic and non-ionic but also based on molecular weight and, in the case of polyelectrolytes (ionic polymers) on charge density (CD) (Bolto & Gregory 2007). The CD of polymers indicates the amount of charges available for destabilization, while the molecular mass reflects the amount of monomers or the length of the polymer chain (Van Nieuwenhuijzen 2002).

The mechanisms of actions are:

Electrostatic interaction

These are interactions where the destabilization is obtained by a polyelectrolyte strongly attaching to that of the colloidal surface due to the attraction between oppositely charged ionic groups (a cationic polymer attachment to a negatively charged colloidal particles) (Bolto & Gregory 2007).

Bridging flocculation

Bridging refers to the attachment to particles by long chain polymers causing a “bridging” effect (Figure 7). An important requirement for bridging flocculation is that there should be sufficient unoccupied site on a particle for attachment of segments of polymer chains adsorbed on other particles (Bolto & Gregory 2007). Consequently, there is an optimal dose of flocculant to each solution below which there is insufficient aggregation and above which there is re-stabilization of the particles (Hubbard 2006).

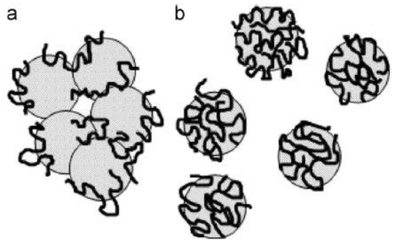

Figure 7: Schematic of a) bridging flocculation and b) re-stabilization due to an overdose of flocculant (Bolto & Gregory 2007).

A polymer can also be used as a flocculant, after coagulation with a metal salt, to proceed to the further grouping of the now neutralized particles. Polymers are less sensitive to pH variations than metal coagulants but, when an inorganic salt and an organic polymer are used as coagulants and coagulant aids, respectively, it is important to maintain pH control (Wang, Fahey, and Wu 2005).

Turbidity and colour are monitored in jar test experiments to determine the optimum dosage of coagulant and flocculants for the removal of suspended solids (Wang et al. 2010). A Standard Jar Test Apparatus is a four- or six-place gang stirrer set at the same speed that is used to mimic mixing and settling conditions at, for example, different chemical dosages and pH values. A compressed air/water tank can be used to obtain the micro bubble; this tank is modified by removing the nozzle from its hose and fitting a pressure gauge into the tank if not already incorporated (Wang et al. 2010).

c) Anaerobic digestion

Anaerobic digestion is a suitable method for wastewater that has a high organic content; that is a wastewater with a BOD/COD ratio of 0.5 or higher, which is easily treated biologically (Metcalf & Eddy 2003). Anaerobic digestion is defined as the engineered methane producing decomposition of organic matter, which involves different species of anaerobic microorganisms that degrade organic matter in the absence of molecular oxygen (Cote et al. 2006). The biological conversion of the organic substrate occurs by hydrolysis, followed by acidogenesis, acetogenesis and methanogenesis to convert the intermediate compounds into methane (CH_4) and carbon dioxide (CO_2), which are the two main end products (Guerrero et al. 1999). Anaerobic digestion has two major drawbacks, which are the long retention times and long start-up period (De Mes et al. 2003). The problem of long retention times can be rectified by using high-rate anaerobic bioreactors, such as upflow anaerobic sludge blanket (UASB) reactors, up-flow anaerobic filtration and fluidized bed reactors (De Mes et al. 2003). As for the long start-up period, it can be shortened by seeding the reactor with sludge from the same process and maintaining a favourable environment (pH and temperature) for the rapid growth of microbial consortia in the case of high-rate anaerobic reactors (Yacob et al. 2006; Liu et al. 2010).

Factors affecting anaerobic digestion are the reactor pH, organic loading rate and agitation.

pH: The microbial communities present in anaerobic digesters are sensitive to low pH values and abrupt changes, especially methanogens (Leslie et al. 1999). Methanogenic activity will decrease when pH in the digester deviates from the optimum values favouring growth, *i.e.* between 6.8 and 7.2 and the major cause of pH decrease is the increase in volatile fatty acids concentration, resulting in inhibited methanogenesis (Gerardi 2006).

Organic loading rate (OLR): An increase in the OLR results in an increase in biogas production until the consortium of microorganism cannot work fast enough and the efficiency of COD removal decreases (Bala et al. 2014).

Mixing/Agitation improves contact between microbial communities and substrates and stabilizes environmental conditions, avoiding layers of material with stages of different pH and temperature (Leslie et al. 1999). Mixing can be done mechanical using impellers, through biogas recirculation or through slurry recirculation. A study by (Karim et al. 2005) on study the effect of mixing (via biogas recirculation, impeller mixing, and slurry recirculation) on biogas production with three sets of experiments using cow manure slurry feed showed that slurry recirculation yields more biogas production compared to the other two mixing modes.

The authors explain that the higher biogas production associated with slurry recirculation may be attributed to the fact that the particles, chunks and flocks were exposed to higher shear and were crushed while passing through the hub of the recycling pump used.

Intermittent mixing is preferable over continuous mixing, while rapid mixing is not recommended as methanogens can be less efficient in this case (Bala et al. 2014). Whitmore et al. (1987) explain that very rapid mixing disrupts flocs structure thereby disrupting the syntrophic relationships between organisms.

POME has a high organic content (Table 1), and represents a source of high biogas production. Anaerobic treatment of POME coupled with the capture of the released biogas from the process has a strong economic advantage. To evaluate the potential profit from biogas production, a biochemical methane potential (BMP) test can be performed. It allows the determination of the biodegradability of a substrate, but also the equivalent potential of methane production during anaerobic digestion (Owen et al. 1979).

Two studies, that are reviewing the potential of POME as a source of methane, show a range of methane yield between 0.278 - 0.348m^3/kg COD (Table 5). One of the studies, Kim et al. 2013 reports the mesophilic co-digestion of POME with EFB by performing biochemical methane potential (BMP) test on both POME and EFB, first separately and subsequently compares the biogas yields to the one obtained in the co-digestion. The other, (Basri et al. 2010), is a scale-down study using a reactor of 0.05 m^3 where the experiment was conducted in semi-continuous mode.

Table 5: Comparison common parameters of studies on biogas production form POME

Parameters	(Kim et al. 2013)	(Basri et al. 2010)
COD concentration (mg/L)	49,200	50,000
Methane yield (m^3 CH_4/kg COD)	0.301	0.278 - 0.348

It should be noted that stoichiometrically 1 kg of COD removed leads to 0.35 m^3 of methane, thus the relevant information giving a real idea of the methane potential is the fraction of ingoing COD that is converted to methane; in which case Kim et al. (2013) report a conversion of 0.397 ± 0.009 m^3 CH_4/kg VS.

d) Sequence batch reactors

Aerobic treatment units, such as the sequence batch reactors (SBR) system, rely on high-rate oxidizers of soluble organic and nitrogenous compounds (the conversion of organic nitrogen and ammonia into nitrate). The SBR's are mixed tanks operated at constant volume. However, the influent flow is intermittent, since the system is closed to the influent during the treatment cycles that are: filling, reacting, settling, decanting, and idle (Buchanan & Seabloom 2004) (Figure 8).

Filling: Pre-treated wastewater is added to the reactor. Aeration can be turned off during this phase to alternate periods of high or low dissolved oxygen. Alternating low and high levels of oxygen and substrate (fresh wastewater) increases the efficiency of the microorganisms in treating the wastewater (Henry & Heinke 1996).

Reaction: Aeration is provided for the rapid biodegradation of organics and nitrogenous compounds.

Settling: Aeration is stopped, allowing anoxic conditions enabling denitrification, but also allowing efficient liquid-solid separation.

Draw: The clear supernatant is removed. When in excess, the solids at the bottom of the tank must also be removed (Idle stage).

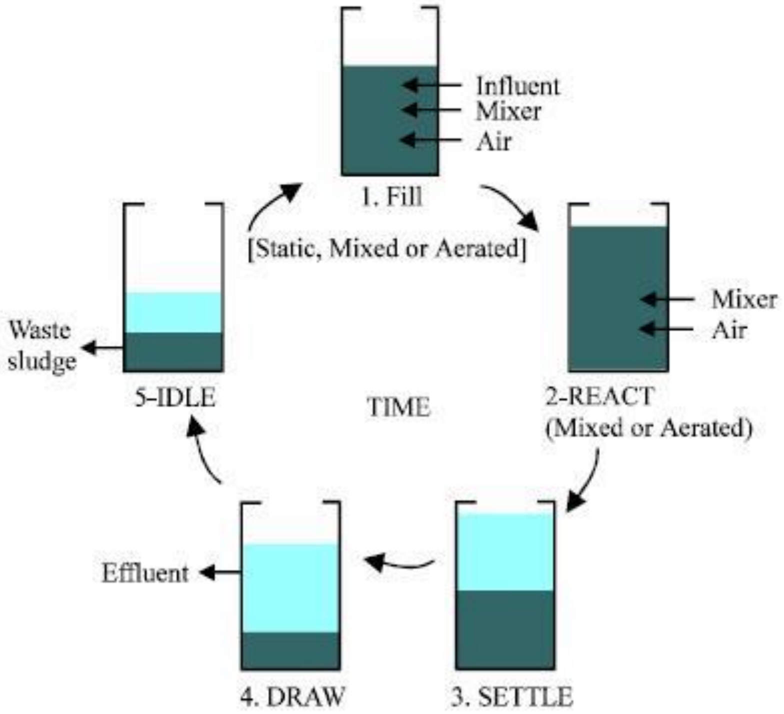

Figure 8: Sequence batch reactors stages (IWA Publishing)

The treatment cycle listed above can be changed to promote aerobic, anaerobic or anoxic conditions; for instance, a static filling (without aeration) allows minimum energy input and high substrate concentration at the end of the filling, which promotes a high food to microorganism (F/M) ratio favouring floc formation and good settling (Buchanan & Seabloom 2004). Moreover, bacteria are degrading the organic matter using residual oxygen or nitrate (alternative electron acceptor) in anoxic conditions, which leads to denitrification (Buchanan & Seabloom 2004).

The SBR has been proven a cost-effective treatment system for palm oil refinery effluent (PORE) that has a COD content ranging from 1,000 mg/L to 5,000 mg/L (Vijayaraghavan et al. 2017). Thus, it shows the potential of SBR as post-treatment for Goldtree anaerobically treated POME that has a COD ranging from 1,465 mg/L to 700 mg/L at the facultative pond 4. A study on SBR for the post treatment of anaerobically digested POME reports a COD removal efficiency ranging from 91.2 % to 95.6 %, starting with a CAL system delivering an anaerobically digested POME with an initial COD content of 1,170 – 2,390 mg/L reduced to 724 – 1,290 mg/L after the SBR (Chan et al. 2010).

3. Material and method

a) DAF experiments

POME samples

Samples of POME were provided by Goldtree (SL) limited. To obtain reliable data representative of the day-to-day fluctuating COD content in POME due to the milling process, fresh samples were taken on every milling day. The samples were collected in a 20-litre jerry can at the oil recovery tank where the temperatures vary from 30 to 70 °C depending on milling. The samples were stored at the Goldtree laboratory at room temperature but no longer than a day. Before each experiment the samples were homogenised thoroughly to avoid settling effect. For experiment 1 and 3, these samples were cooled to room temperature prior to each jar test. For experiment 2 they were heated, when necessary, to the desired temperature. For the first phase of experiment 4, samples of anaerobically digested POME were collected at pond 4. This pond was in operation during sampling therefore receiving daily new inflow of digested POME from upstream ponds. The samples were taken at approximately 20 cm below the surface and at 20 meters away from the inlet. Samples were stored in Goldtree laboratory at room temperature, but no longer than a day. For the second phase of experiment 4, the samples of anaerobically digested POME came from the reactor bottles used in the fed -batch reactor experiment.

Coagulants, flocculants and other chemicals

Coagulants (liquids) listed in Table 6 are metal salt of positive charge added to the wastewater to overcome the repulsive charge and "destabilize" the suspension of colloidal particles provoking aggregation into “pin” flocs. The flocculants are cationic and anionic polymer (powdered) used for further grouping of pin particles into larger floc (Table 7). Coagulants and polymers provided by Nijhuis Indutries (NL) are Kemira Water Solutions, Inc. products. As recommended by the manufacturer, each coagulant was used without dilution.

The exact values of MW and CD of the polymers used are not provided in the product data sheets but some estimation could be found in literature (Bolto & Gregory 2007; Colic et al. 2005).

Table 6 Coagulants specifications

Name	Commercial name	Product specification
Polyaluminium chloride	PAX18/14	Al = 9.0 ± 0.2 % Al_2O_3 = 17.1 ± 0.4 % Fe < 0.01 %
Sodium aluminate	SAX14	$NaAlO_2$ = 29 - 46 % NaOH = 5 %
Ferric chlorides	PIX311	$FeCl_3$ = 37 - 42 % HCl <= 1.0 %

Table 7 Flocculants specifications

Commercial name	Type	Concentration (g/L)	MW	CD
Superfloc C492	Cationic	2.5 - 5	High	Very low[2]
Superfloc A130	Anionic	2.5 - 5	High[3]	Medium

Dissolved air flotation experiment on POME

General procedure for experiment 1 - 3

The DAF experiments, as described below, were performed according to the instructions provided by Nijhuis industries during a training session.

The equipment used were 1,000 mL beakers, a VELP SCIENTIFICA JLT4 flocculator as well as a BIRCHMEIER Spray-Matic 5S to pressurize water to 4 - 6 bar thereby simulating the micro-bubbles addition system of a DAF unit. The COD was measured using the HANNA HI839800-02 COD Reactor, HI93754C-25 COD vials high range (1,000-15,000 mg/L), and HI83224-02 COD and Multiparameter Photometer with bar code vial recognition. The turbidity was measured using a Secchi disk, and the % floating sludge blanket was evaluated using a ruler to measure the cm of sludge blanket over the cm of total volume (sludge blanket + liquid).

Each sample of 200 mL of POME was first mixed using the flocculator at the rotation speed of 250 rpm for 1 minute during which the pH and temperature of the sample were measured. While maintaining the speed of 250 rpm, a coagulant was added followed by 1 mL of sulphuric acid (on average) to adjust the pH of coagulation to 3.5 but only when adjustment

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[2] Approximated charge density 10 - 25 mol % (Bolto & Gregory 2007)

[3] Estimated molecular-weight > 7,000,000 D (Colic et al. 2005)

was necessary. The pH of untreated POME is approximately 4.5 and the addition of metal salts was often enough to drop the pH of the solution to 3.5. Solutions of sodium hydroxide were used to neutralize the sample to a pH of 6.5. For the flocculation phase, rotation speed of the flocculator paddles was first lowered to 150 rpm, the polymer was then added using a 10 mL syringe inserted 1 cm bellow the liquid surface. The flocculant was added at the same time as pressurized water (varying quantities of water depending on the experiment). The flocculator was turned off 1 minute after the addition of flocculant. The pH, turbidity and COD of the liquid were measured and efficiency of removal was calculated using initial and final COD of the sample.

Experiment 1: Effect of coagulant/flocculant combinations and microbubbles

In this experiment, we compared combinations of coagulant and flocculant with different volumes of pressurized water to determine a combination resulting in a stable separation of the solids from the liquid, regardless of the COD content of the POME ranging from 35,000 - 60,000 mg/L.

The coagulants were used undiluted (as recommended by the supplier) maintaining the concentrations listed in Table 6 and the volumes were kept at 4 mL per samples of 200 mL. Polymer solutions of 5 g/L were prepared and 20 mL was added to each tested sample of 200 mL. Since very little research has been done on raw POME treatment with DAF and multiple coagulants (PAX18, PAX14, $FeCl_3$ and SAX14) and flocculants (A110, A130, A150, C492, C496, C498, C577 and C587) were available, it was crucial to follow a practical, efficient screening method. Thus, a trial and error set of experiment, working from diluted to pure samples of POME, was performed prior to experiment 1 to determine the volumes of coagulant and flocculant mentioned above. The results of the set trials are not discussed here.

The effect of micro-bubbling addition is tested by comparing 3 volumes of pressurized water (200 mL, 400 mL and 600 mL). This experiment is repeated for samples of low COD content (31,680 mg/L) and high COD content (59,520 mg/L). Considering the limited amount of COD kit tests tubes available, the parameters of comparison which were turbidity and percentage of floating sludge blanket, were both assessed visually, as mentioned earlier.

From the results of this experiment, the combination of coagulant/flocculant/pressurized water that yielded similar result for different COD concentration of POME is selected for the remaining 3 jar test experiments.

Experiment 2: Effect of PAX18 and A130/C492 on the solids separation at different temperatures

The POME that comes out of sterilisers has a temperature between 60 – 70 °C. The effect of temperature on COD removal was investigated to recommend or rule out the need of a cooling tower prior to the DAF unit. In this experiment the initial temperature of the "high temperature" samples was 60 – 70 °C while the "low temperature" samples were kept at room temperature *i.e.* 25 – 27 °C. The coagulants were used undiluted (as recommended by the supplier) and their volumes were kept at 4 mL. Polymer solutions of 5 g/L were prepared and 20 mL was added to each tested sample

Experiment 3: Effect of different volumes of PAX18 and C492/A130 on solids separation

The goal of this experiment was to estimate the optimum volumes of coagulant to obtain a COD removal efficiency of 90 % or higher. Three different volumes of PAX18 (3 mL, 4 mL & 5 mL) combined with two volumes (20 mL & 30 mL) of 5 g/L of A130 and C492 were tested according to the procedure described above.

Experiment 4: Effect of different volumes of PAX18 and A130/C492 on solids separation for samples of anaerobically digested POME.

To determine the possibility of using a DAF unit as a post treatment to anaerobic digestion, additional jar tests using samples of anaerobically digested POME were performed. This experiment investigates whether the use of chemicals can be reduced via solids separation of anaerobically treated POME with a DAF. In a first phase, samples were collected from pond 4 (initial COD content of 1,052 ± 316 mg/L) in order to evaluate what to expect when using a DAF as a post-treatment to the current wastewater treatment installation. In a second phase, samples are collected from the fed batch reactor test bottles representing the digested effluent of a CAL system (see section on anaerobic digestion).

For these DAF simulations, each sample of 200 mL of anaerobically digested POME was first mixed using the flocculator at a rotational speed of 250 rpm for 1 minute during which the pH and temperature of the sample were measured. While maintaining the speed of 250 rpm, 5 mL of sulphuric acid was added to lower the pH from 7.0 to 4.0. Next, a coagulant is added (0.5 - 1 mL) further reducing the pH to 3.5. To neutralize the sample to a pH of 6.5 after coagulation, 2 mL of sodium hydroxide was added. After lowering the rotation of the flocculator paddles to 150 rpm, 5 mL of C492 (2.5 - 5 g/L) is added using a 10 mL syringe inserted bellow the liquid surface at the same time as 200 mL pressurized water. The flocculator was turned off 1 minute after the addition of flocculant. The pH and COD of the liquid phase were registered, and the efficiency of removal was calculated using the initial and final COD content of the sample. Sets of trial-and-error experiments were run on anaerobically digested POME prior to experiment 4 to determine the volumes and concentrations of chemicals listed above. The results of these trials are not discussed in this dissertation as they were purely based on whether or not a separation is obtained which was evaluated visually.

b) Anaerobic digestion

Biochemical methane potential (BMP) test

The biochemical methane potential (BMP) tests were performed in batch reactors to evaluate the maximum amount of biogas or bio-methane produced per gram of volatile solids (VS) contained in the POME. Each flask had a working volume of 80 mL. The experiment was operated for 21 days at mesophilic conditions (35 °C). The inoculum came from the wastewater treatment plant Ossemeersen in Ghent. First, the inoculum was characterized by performing VFA, conductivity, TS and VS analysis. Based on the VS data and to obtain a final VS concentration of 10 g VSS/L, 32.18 mL of inoculum was added to each penicillin bottle. Next, the amount of substrate needed per vessel was calculated based on the COD content of POME and 11.84 mL of POME was added for each sample serum flask (excluding the negative controls) to obtain a substrate to inoculum ratio of 0.5 g COD/g VSS. Finally, 336 mL of tap water was added to acquire a total liquid volume of 80 mL in each bottle. Triplicate negative controls were run, containing the inoculum at a concentration of 10 g VSS/L, to estimate endogenous methane production. After adding the inoculum and substrate the serum flasks were sealed and connected to gas columns by means of air-tight tubing and needle. To avoid the CO_2 in the biogas from dissolving the columns were filled with the distilled water at $pH < 4.3$ and placed in a water bath of the same distilled water/acid composition. The biogas production was evaluated by measuring the water displaced in the gas columns. The measurements were recorded on a daily basis for 21 days. Biogas composition was evaluated at the end of the experiment.

Fed batch reactors

The goal of this experiment was to determine the biogas production potential of POME under intermittent feeding conditions in semi-continuous reactors. Three replicate reactors were set up in a temperature-controlled room at 34 ± 1 °C, and fed with POME 3 times per week. Measurements of biogas yield and composition were recorded three times per week. The inoculum used came from the wastewater treatment plant Ossemeersen in Ghent while the POME was provided by Goldtree. The characterization of both inoculum and POME included measurements of total solids, volatile solids, VFA and COD (for POME only). The experimental set up consisted of Schott bottles with a total volume of 500 mL. They were connected to plastic tubes for gas collection (Figure 9). Each reactor bottle contained 400 mL of inoculum and substrate feeding was done three times per week on Monday, Wednesday and Friday. To acclimatize the microbial community in the inoculum to POME, feeding volumes started from 20 mL to 30 mL. Next, a regular feeding of 40 mL on Mondays and Wednesday and 60 mL on Friday (to incorporate the extra day on weekends) was followed for 50 days. This feeding regime resulted in a HRT of 20 days.

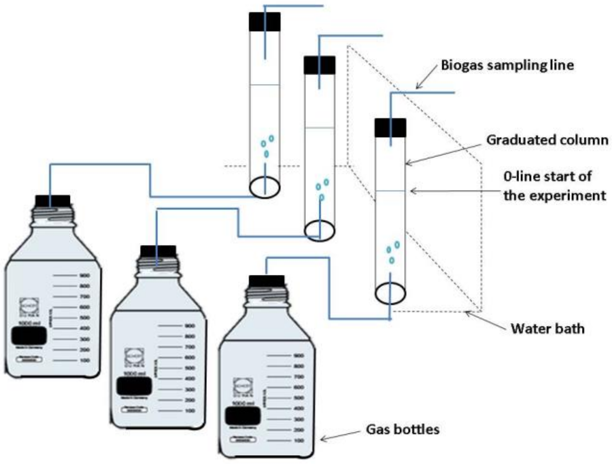

Figure 9: Fed batch reactor set up. The material used included 3 glass Schott bottles (reactors 1, 2 & 3), a 10 L water basin (pH kept below 4.3 to avoid CO_2 from dissolving) fitting three DN250 mm PVC pipes, rubber stoppers, and column caps. The reactor bottles contained the inoculum (400 mL) and substrate (increasing volumes) and the gas collection columns were graduated (0 – 4 L) and filled up to the 0-line with water.

The TS, VS, VFA concentrations and conductivity of the inoculum of all three reactors were monitored once per week. For every feeding, the pH of the reactors and biogas volume and composition were measured.

c) Sequence batch reactors

The effect of SBR systems on COD removal are simulated in the experiment described below. The system involved periods of filling wastewater, aeration, settling, and refilling. In order to satisfy both short and long term options, SBR tests were performed using as substrate both the supernatant from the BMP test bottles and anaerobically digested POME form pond 4.

Aerobic sludge preparation

To obtain aerobic sludge used as seeds for the reactor, 6 L of anaerobic sludge were collected from activated sludge ponds of Goldtree. The anaerobic sludge was fed 400 mL of anaerobically digested POME while being aerated using a HITACHI BEBICON compressor at 0.2 bars for 3 to 5 hours a day for 7 days at the ambient temperature of 28 °C ± 2. Seven days was considered enough time for acclimatization as the sludge was accustomed to the type of wastewater. During the acclimatization phase, the sludge was monitored with respect to TS and pH. Appropriate sludge content was indicated by the effluent reaching a TS content of 0.25 g TS/L.

Procedure

The experiment started with biologically seeding each reactor with 500 mL of aerobic sludge. The reactors were 2L plastic bottles that were covered to minimize water loss due to aeration and evaporation. A single reactor per substrate was used to conduct the sequences of fill, aerate, settle, and withdraw. A volume of 500 mL of BMP supernatant / anaerobically digested POME were added to 500 mL aerobic sludge giving a final a working volume of 1L. The mix was then aerated for 3 hours after which it is left to settle for 20 hours. After, the 20 hours settling phase, a sample was collected and analysed for COD and new cycle was started. Because the constraints on the electricity source (biomass boiler and steam turbine only in operation during milling), the cycle frequency was kept at once a day for a period of 15 days.

Reactor performance

The SBR was operated at fixed time of aeration and substrate volume. The performance of the SBR was evaluated based on COD and suspended solids removal. The clarified supernatant was thus tested for COD and TDS after each cycle. In order to determine removal efficiency the substrate/aerated sludge mix was also analysed for COD and TDS.

d) Analytical techniques

COD analysis

COD measurement performed in Goldtree industrial site (DAF experiment 1 - 4 and SBR) were done using a HANNA COD reactor and photometers. All the other COD measurements (anaerobic digestion) were performed in Cmet analytical laboratory by back titration.

COD analysis through titration

The COD was performed according to Standard Methods for the examination of water and wastewater (5220-C, APHA, 1992). The COD analysis by back titration is an oxidation method using $K_2Cr_2O_7$ in an acidic, high temperature environment and in the presence of silver sulphate as catalyst. After oxidation of dissolved and suspended organic matter, the excess (unreduced) $K_2Cr_2O_7$ is titrated with a solution of $FeNH_3SO_4$. Ferroin is added as indicator which led to the colour change from green to blue to the endpoint red/brown during the titration. Samples of untreated POME were diluted at the start of the experiment; all other samples analysed were not. A blank consisting of 10 mL water instead of sample was also included.

COD analysis using HANNA COD reactor and photometers

The COD measurements were performed following the equipment user manual. It consisted in preheating a heating apparatus (HANNA HI839800-02 COD Reactor) to 150 °C, diluting the samples of untreated POME (COD vials ranged from 1,000 to15,000 mg/L) and transferring 5 mL of each sample into a COD vial. The digestion at 150 °C lasted 2 hours and cooling to room temperature lasted 20 - 30 mins. The measurement of COD content was done using HI83224-02 COD and Multiparameter Photometer.

VFA analysis

The C2 - C8 fatty acids (including isoforms C4 - C6) were measured by gas chromatography (GC-2014, Shimadzu®, The Netherlands), equipped with a DB-FFAP 123-3232 column (30 m x 0.32 mm x 0.25 µm; Agilent, Belgium) and a flame ionization detector (FID). Detection limits were 30 mg/L for acetate, 10 mg/L for propionate, and 2 mg/L for the other VFA, with an upper detection limit of 1,000 mg/L. The liquid samples were conditioned with sulfuric acid, sodium chloride and 2-methyl hexanoic acid as internal standard for quantification.

Diethyl ether was used for extraction. The prepared sample (1 µL) was injected at 200 ºC with a split ratio of 60 and a purge flow of 3 mL/min. The oven temperature was increased by 6 ºC/min (110 ºC to 165 ºC) where it was kept for 2 min. The FID had a temperature of 220 ºC. The carrier gas was nitrogen at a flow rate of 2.49 mL/min.

Solids content analysis

The total solids (TS) and volatile solids (VS) tests were performed according to the Standard Method 2540G (APHA, 1992). To determine TS samples were dried in crucibles at 105 °C for minimum 24 hours. The VS was determined by ashing the crucible at 550 °C for 1.5 h in a muffle furnace (Nabertherm® LE6/11/B150, Germany).

To determine the TS in g/L, the weight of the crucible was subtracted from the weight after drying at 105 °C, and divided by the volume of the original sample. To determine the VS, the weight of the crucible ashing at 550 °C was subtracted from the weight after drying at 105 °C, and dividing by the volume of the original sample.

Gas analysis

The biogas samples were collected using a vacuum pump, a gas collection tube and airtight syringes. The composition of 1-2 mL of each sample was analysed with a Compact GC (Global Analyser Solutions, Breda, The Netherlands), equipped with a Molsieve 5A pre-column and Porabond column (CH_4, O_2, H_2 and N_2) and a Rt-Qbond pre-column and column (CO_2, N_2O and H_2S). Concentrations of CH_4, H_2S, O_2, N_2, H_2 and CO_2 were determined by means of a thermal conductivity detector with a lower detection limit of 100 ppm for each gas component.

Conductivity and pH measurement

Conductivity measurements were performed using a Consort C6010 conductivity meter. The pH measurements performed in Goldtree were done using a HANNA portable pH/EC/TDS Meter (HI9813-5) (DAF and SBR experiments), all other pH measurements were done with a Metrohm 744 pH meter.

4. Results

a) DAF experiments

Experiment 1: Effect of coagulant/flocculant/pressurized water combinations

The goal of this experiment was to determine the combination of coagulant and flocculant that would guarantee a clear separation of the liquid and solids, independent of the changes in COD content of the POME. The amount of micro-bubbles needed to observe such separation was also investigated. Series of trial and error experiments with samples of 200 mL of POME were necessary to determine that approximately 4 mL of coagulant and 20 mL of A130/C492 were needed to observe a separation with a clear liquid phase and a floating blanket. In this experiment, 4 coagulants (PAX18, PAX14, SAX14 and $FeCl_3$) are tested combined to polymers A130 and C492. The results were based on a visual assessment of turbidity. At first look, the more pressured water added the lower the turbidity (Figure 10).

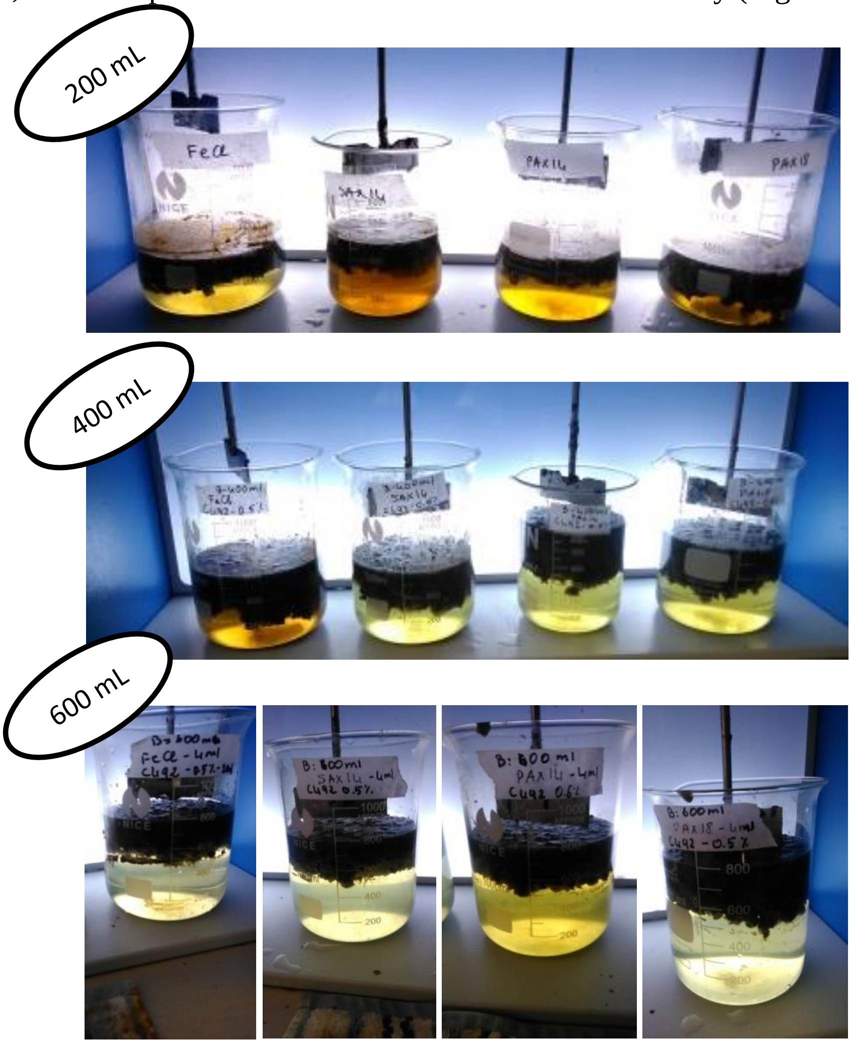

Figure 10: For the same sample of 200 mL POME, 4 coagulants are combined to the polymer C492. The jar test is repeated for 3 volumes of pressured water added (200 mL, 400 mL and 600 mL). The turbidity of the bottom jar solutions improves with increasing volume of added pressurised water.

Combining PAX18 and PAX14 to A130 or C492 resulted in the lowest turbidity values while $FeCl_3$ gave the highest values, and often failed separation at higher initial COD content (Figure 11). Adding 200 mL of pressurized water to a sample volume of 200 mL gave the highest turbidity and poorest separation at lower COD content, with even no separation at higher COD content. When comparing the results of all 4 coagulants combined to A130 where 600 mL of pressurized water was added to the ones where 400 mL was added, the 600 mL resulted in a lower turbidity than 400 mL, with a reduction of 27.50 ± 0.25 % (Figure 12). As for combinations to C492, a reduction in turbidity of 27.08 ± 0.25 % was observed (Figure 13).

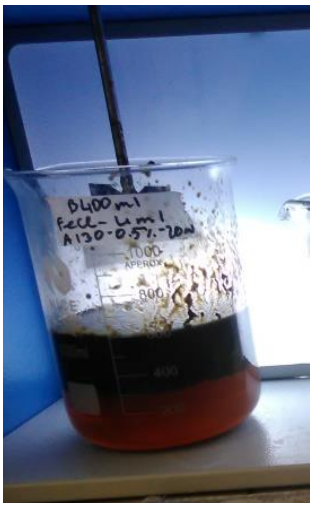

Figure 11: Demonstrating the high turbidity results obtained for a Jar test with $FeCl_3$/A130/400mL of pressurized water on POME sample with initial high COD content (59,520 mg/L).

The results of the percentage of floating sludge confirmed the results obtained for turbidity. A volume of 200 mL of pressurized water gave the lowest quantities of sludge blanket, while 400 mL of added water gave the highest quantities. The PAX18 gave higher percentages of floating sludge compared the other coagulants, especially when coupled to C492 which is an indication of better separation. When combined to C492, PAX18 had a percentage of sludge floating, 19 % higher than PAX14 (second best) and 41 % higher than $FeCl_3$ (poorest results). Considering these results, the remaining DAF experiments were conducted using 400 mL of pressurized water and PAX18 combined to A130/C492, since there was no obvious difference between the two flocculants.

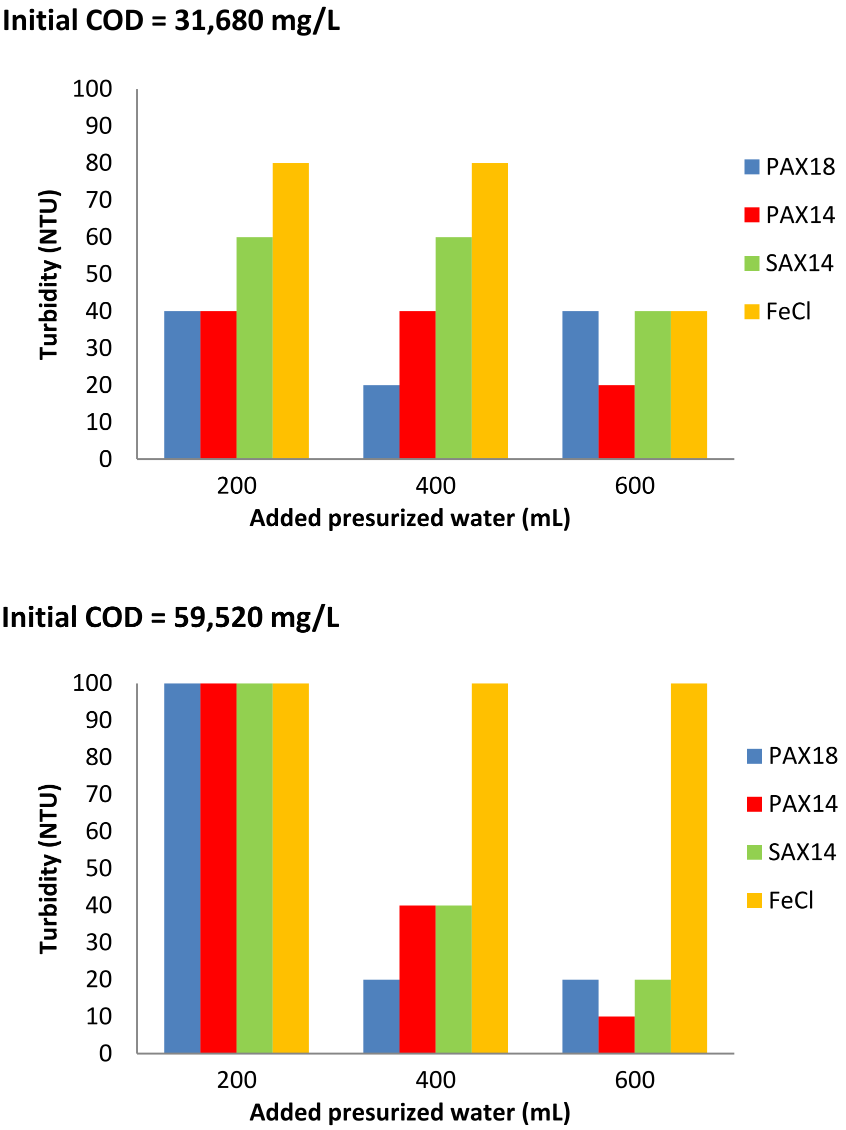

Figure 12: Comparing 4 coagulants combined to the flocculant A130 for 3 different volumes of added pressurized water. The experiment was repeated for POME samples at low (31,680 mg/L) and high (59,520 mg/L) COD content. The comparison assessment was visual and turbidity was measured using a Secchi disk. A volume of 200 mL of pressurized water gave the higher turbidity and poorest separation at high COD content. A volume of 600 mL of water resulted in a 27.50 ± 0.25 % lower turbidity than 400 mL. The PAX18 showed, in general, lowest turbidity and $FeCl_3$ the highest.

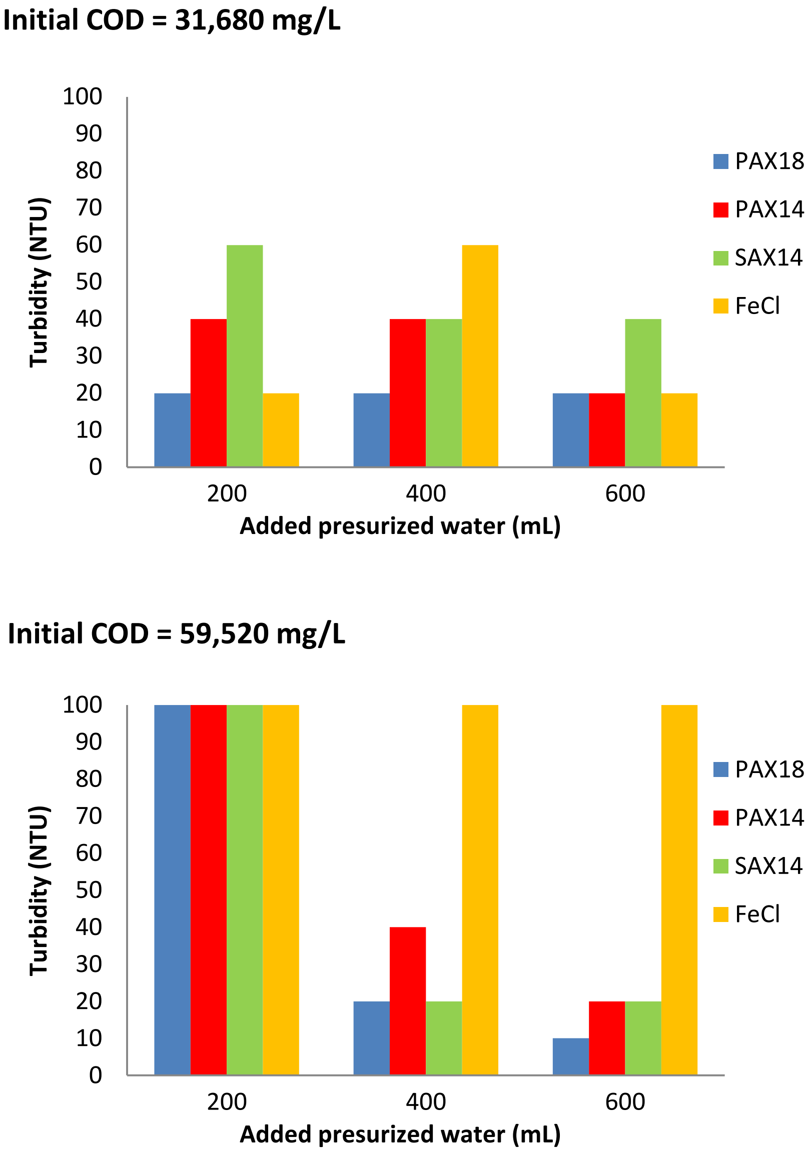

Figure 13: Comparing 4 coagulants combined to the flocculant C492 for 3 different volumes of added pressurized water.

The experiment was repeated for POME samples at low (31,680 mg/L) and high (59,520 mg/L) COD content. The comparison assessment was visual, and the turbidity was measured using a Secchi disk. A volume of 200 mL of pressurized water resulted in a higher turbidity and poorest separation at high COD content. A volume of 600 mL of water gave a lower turbidity than 400 mL. The reduction in turbidity was in average of 27.08 ± 0.25 %. The use of $FeCl_3$ resulted in the highest turbidity, while PAX18 showed the lowest values.

Experiment 2: Effect of PAX18 and A130/C492 on the solids separation at different temperatures

In this experiment, the effect of temperature on the removal of COD was investigated. For each samples of 200 mL, 4 mL of PAX18 was added, as well as 20 mL of A130/C492 and 400 mL of pressured water. The low temperature samples were kept at room temperature, while the high temperature samples were heated to 60 – 70 °C. At lower temperature, the COD removal efficiency was higher than at the high temperature samples by 3.05 ± 0.02 % for PAX18/A130 and 2.16 ± 0.03 % for PAX18/C492 (Figure 14). A slight difference in solution turbidity and colouring between low and high temperature can also be observed visually (Figure 15).

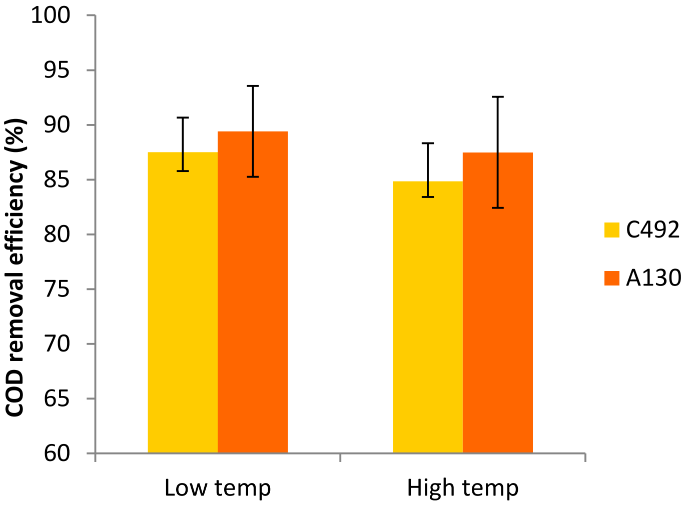

Figure 14: This experiment is evaluating the effect of temperature on COD removal (3 replicates), comparing samples at high temperature (60 – 70 °C) vs samples kept at room temperature (25 – 27 °C). At room temperature the COD removal efficiency is higher than at high temperature but only by an average of 3.05 ± 0.02 % for PAX18/A130 and 2.16 ± 0.03 for PAX18/C492. Error bars represent standard deviations.

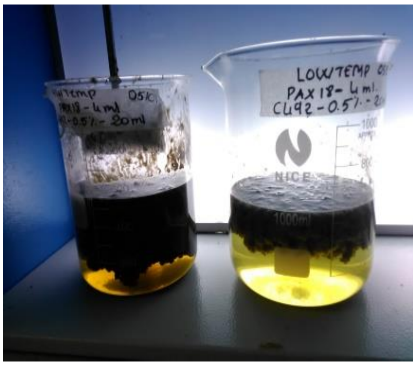

Figure 15: The difference in turbidity and colouring of the solution for a jar test evaluating the effect of temperature on COD removal. The sample at high temperature (60 – 70 °C) on the left vs the sample kept at room temperature (25 – 27 °C) on the right.

Experiment 3: Effect of different volumes of PAX18 and C492/A130 on the solids separation

The goal of this experiment was to estimate the minimum volumes of coagulant and flocculant leading to a COD removal of 90 % or above. Considering the results of experiment 1, with 4 mL of coagulant, 20 mL of flocculant and 400 mL of pressured water giving 40 NTU turbidity or lower, the selected volumes of PAX18 were 3, 4 and 5 mL and the volumes of flocculant were 20 and 30 mL.

The more coagulant added, the higher the removal efficiency, but the effect of increasing coagulant dose is minimal (Figure 16). Adding 4 mL of PAX18, which corresponds to dosing 85 % more aluminium than 3 mL in a 200 mL sample, led to only a 1.57 ± 0.09 % higher COD removal efficiency than 3 mL. The addition of 30 mL of flocculant led to a removal efficiency lower than 20 mL by 4.05 ± 0.21 % for C492 and 2.65 ± 0.12 % for A130.

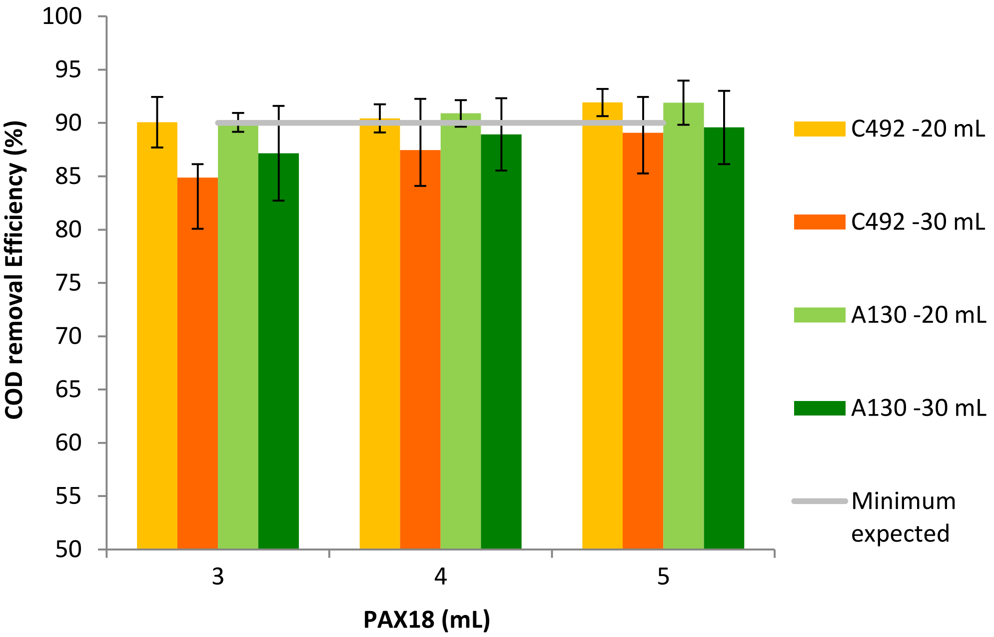

Figure 16: Comparing three different volumes of the coagulant PAX18 combined to two volumes of the flocculants A130 and C492 (3 replicates). Error bars represent standard deviations. The parameter of comparison is COD removal. Increasing the volume of coagulant caused only a slightly higher removal efficiency. Increasing the volumes flocculants decreased the COD removal efficiency.

Experiment 4: Effect of different volumes of PAX18 and A130/C492 on solids separation for samples of anaerobically digested POME.

As demonstrated in experiment 3, 3 mL of PAX18 was necessary to obtain 90 % of COD removal efficiency for the treatment of 200 mL of untreated POME at the lab scale. When converted to industrial scale, the corresponding needed amount of aluminium led to conclude that the option of using DAF as a pre-treatment to anaerobic digestion is environmentally and economically unfeasible. The goal of experiment 4 was to evaluate the potential of DAF as a post-treatment to the actual ponding system, and the future CAL installation.

In a first phase, samples of pond 4 overflows (initial COD = 1,052 ± 316 mg/L) were collected and analysed. Using 0.5 mL of PAX18 achieved on average 82.70 ± 5.87 % removal efficiency when combined to 5 mL of C492 at 2.5 g/L and 85.57 ± 4.46 % for 5 g/L of C492 (Figure 17). Using 1 mL of PAX 18 achieved higher results with 86.83 ± 7.22 % removal efficiency for a concentration of C492 at 2.5 g/L and 88.27 ± 4.46 % for 5 g/L of C492.

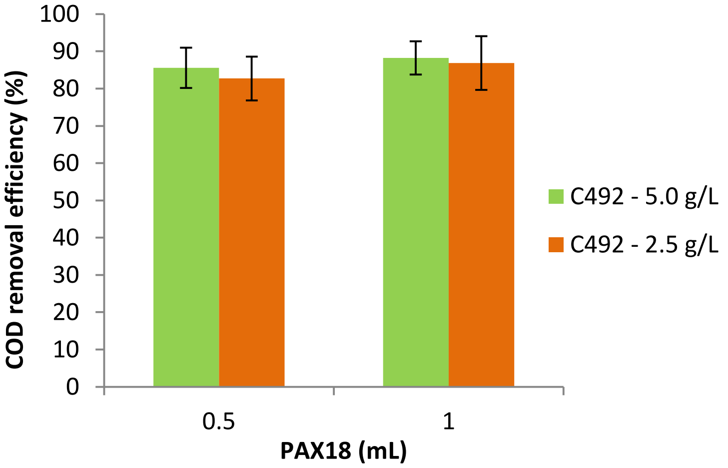

Figure 17: Comparing 0.5 - 1 mL of PAX18 and 5 mL of C492 at 5 g/L and 2.5 g/L on samples of 200 mL from pond 4 overflow (3 replicates). The addition of 0.5 mL of PAX 18 resulted in on average 82.70 ± 5.87 % removal efficiency combined to 5 mL of C492 at 2.5 g/L and 85.77 ± 4.46 % when combined to 5 mL of 5 g/L of C492. Using 1 mL of PAX 18, higher removal efficiency were obtained with 86.83 ± 7.22 % for a concentration of C492 at 2.5 g/L and 88.27 ± 4.46 % for 5 g/L of C492. Error bars represent standard deviations.

The initial COD content of samples from pond 4 was 1,052 ± 316 mg/L. Even though the removal efficiencies obtained were below 90 %, the effluents COD content meet the required 250 mg/L (Figure 18). Samples in jar 1, 2 and 3 were treated with 0.5 mL of PAX18 and 5 mL of C492 at the concentration of 5.0 g/L, while jar 4, 5 and 6 were treated with 0.5 mL of PAX18 and 5 mL of C492 at the concentration of 2.5 g/L.

Jar 1, 2 and 3 returned an effluent with a COD content bellow 250 mg/L (average removal efficiency 85.57 ± 4.46 %), while jar 4 contained effluent with a COD value above the discharge limit, with a corresponding removal efficiency of 77 % only (5 % below the average 82.70 ± 5.87 %).

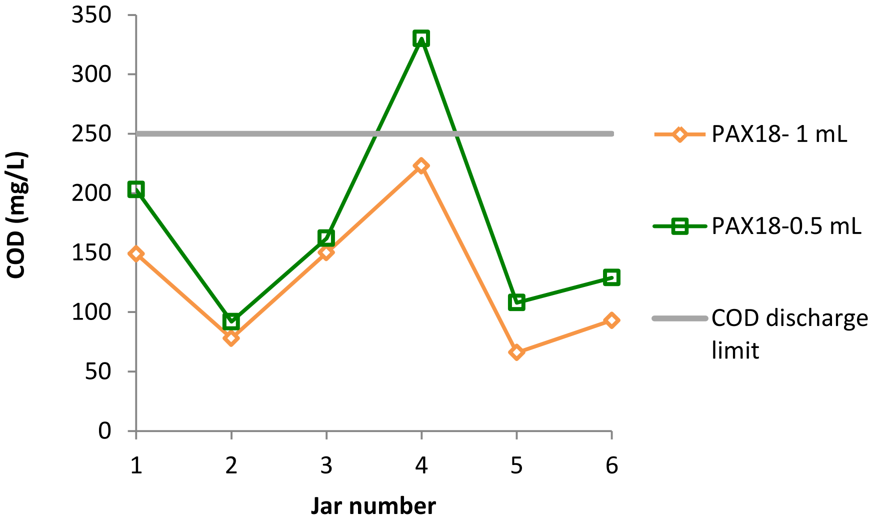

Figure 18: Comparing 0.5 - 1 mL of PAX18 and 5 mL of C492 at 5 g/L and 2.5 g/L on samples of 200 mL from pond 4 overflow. The removal efficiencies obtained are enough to meet the required COD content of 250 mg/L at discharge.

In a second phase, anaerobically digested POME collected from the fed batch reactor experiment bottles was treated with 0.5 mL of PAX18 and 5 mL of C492 at 5 g/L. The initial COD content of the sludge was 5,691 ± 694 mg/L and the removal efficiency 81.0 ± 3.1 %. The resulting effluent had a COD content of 1,094 ± 300 mg/L.

Another observation made in experiment 4 is the decrease in the thickness of the sludge blanket produced (Figure 19). When treating raw POME with PAX18 at 90 g/L and of C492 at 5 g/L and 200 mL of pressurized water, 53 ± 27 % of the total occupied volume of the jar was the sludge blanket (experiment 1). Treating anaerobically digested POME with 90 g/L and of C492 at 5g/L and 200 mL led to 16 ± 8 % of sludge produced or 70 % less sludge. A reduction of sludge production corresponds to a reduction in operational cost, and is discussed in the CAPEX and OPEX calculations section.

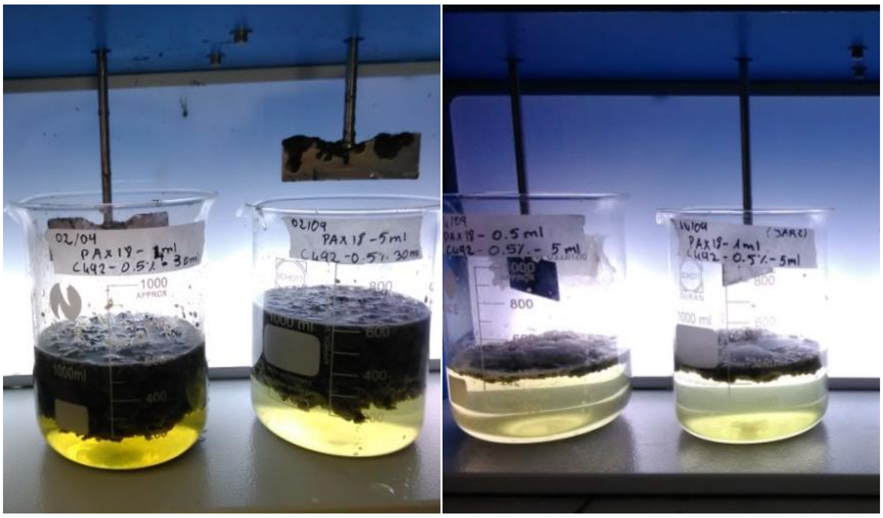

Figure 19: Showing the difference in sludge production when treating untreated POME (left picture) and anaerobically digested POME (right picture) with 90 g/L of PAX18/5 g/L of C492.

b) Anaerobic digestion

Biochemical methane potential (BMP)

The goal of this experiment was to evaluate the maximum yield of methane produced per gram of VS of POME. The BMP test ran for 21 days during which the methane volumes were measured daily (Figure 20). The substrate characterization gave a VS and COD content of 21.41 ± 0.45 g VS/L and 33.80 ± 2.32 g/L, respectively. A value of 291 ± 17 mL CH_4/g VS was observed, corresponding to 53 ± 0 % COD conversion efficiency to methane.

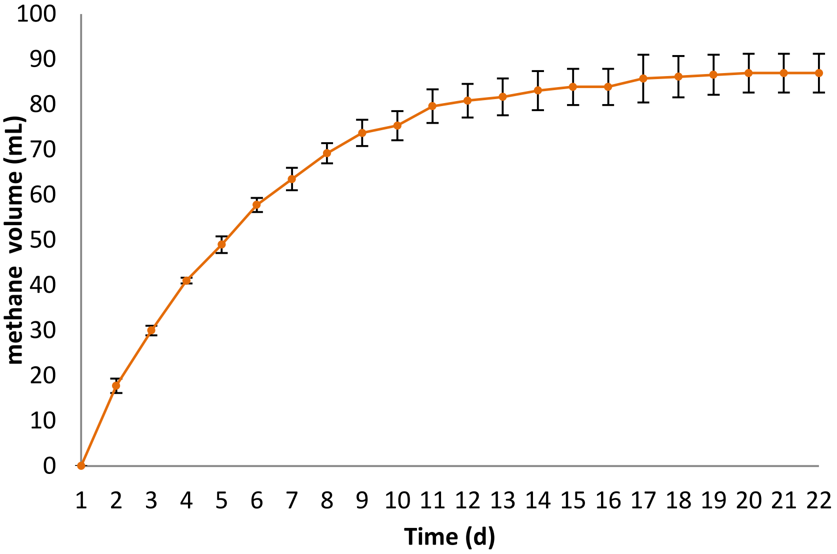

Figure 20: Methane production (mL) over 21 days. Three penicillin bottles (replicates) contained 32.18 mL of inoculum 11.84 mL of POME each. These volumes of inoculum and substrate are based on the characterisation of both the inoculum and substrate. A maximal conversion efficiency of 53 ± 0 % was obtained, which corresponds to 290 mL CH4/ g VS. Error bars represent standard deviations.

Fed batch reactor

The fed batch reactor experiment lasted 50 days and feeding was performed as to maintain a HRT of 20 days. The biogas yield measurements collected on Monday, Wednesday and Fridays show a decrease from days 25 to 3 (Figure 21). This is can be explained by the overloading of reactors during feeding on days 13, 15 and 17 where 60 mL of POME was fed all three times instead of 40 mL for feeding 6 and 7 and 60 mL for feeding 8. A decrease in pH (Figure 22) and increase in acetate and propionate (Figure 23) were also observed. The observed COD removal efficiency, calculated using the COD of the POME before and the COD of the POME after digestion, is 83.14 ± 11.59 %, and an average methane yield of 222 ± 77 mL CH_4/g VS corresponding to 40 ± 0.14 % COD conversion efficiency to methane was obtained.

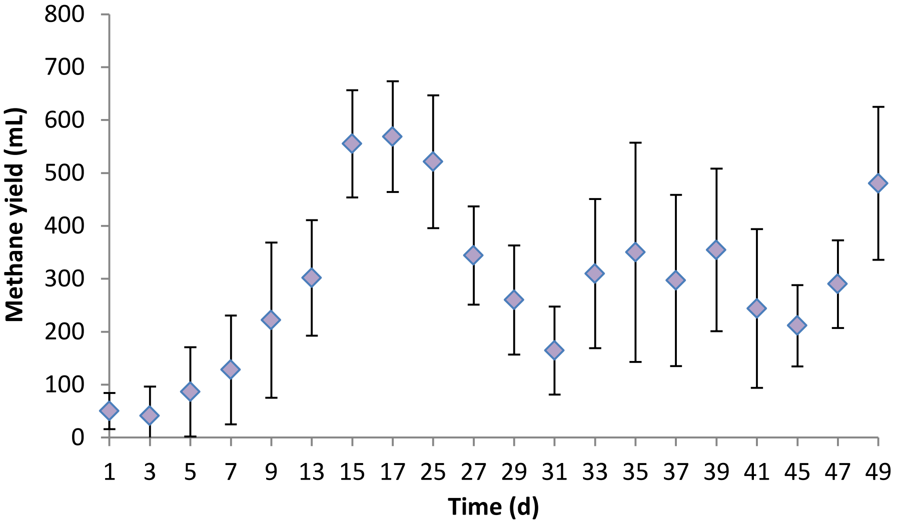

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Figure 21: Measured methane yield (mL) (triplicates). This experiment ran for 50 days with intermittent feeding and biogas yield measurements (Collected on Mondays, Wednesdays and Fridays). Error bars represent standard deviations.

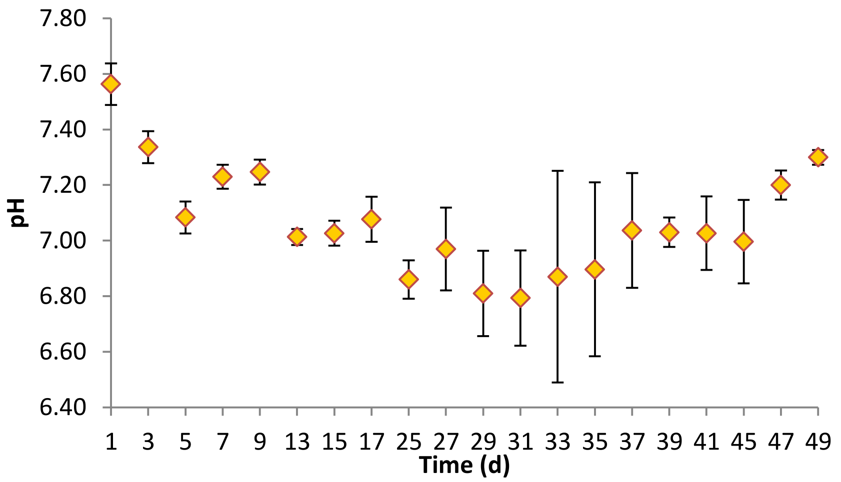

Figure 22: Digester pH. Error bars represent standard deviations.

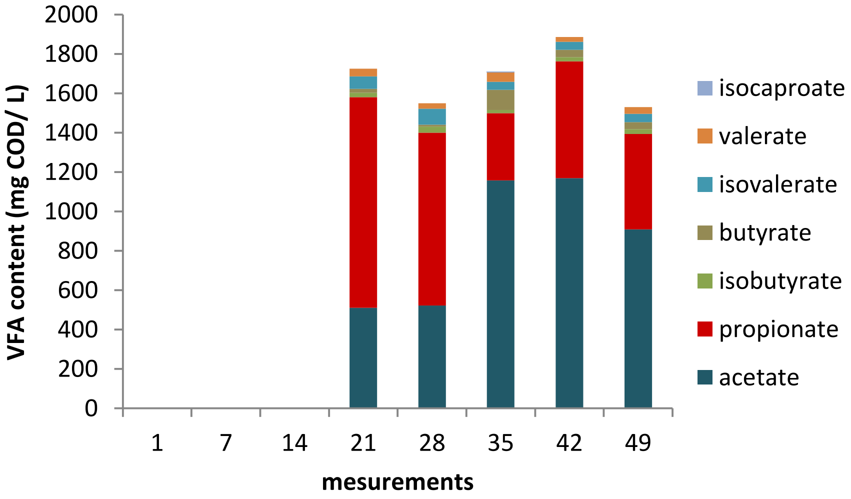

Figure 23: Total VFA concentration (mg COD/L) per week. The increase in VFA is caused by an overloading of the reactors.

c) Sequence batch reactors

The goal of this experiment was to determine the COD removal efficiency of a SBR system on anaerobically digested POME. For 15 days, a daily cycle of filling, aeration and settling was performed and COD content of the clear supernatant was measured after 3 cycles. After a steep decrease in COD until day 6 and 9 for samples from pond 4 and BMP reactors respectively, an increase in COD content to end of the experiment was observed (Figure 24). Considering the minimum COD level obtained (on day 6 and 9), the removal efficiencies were 64.78 % and 67.01 % for pond 4 and BMP reactors samples respectively.

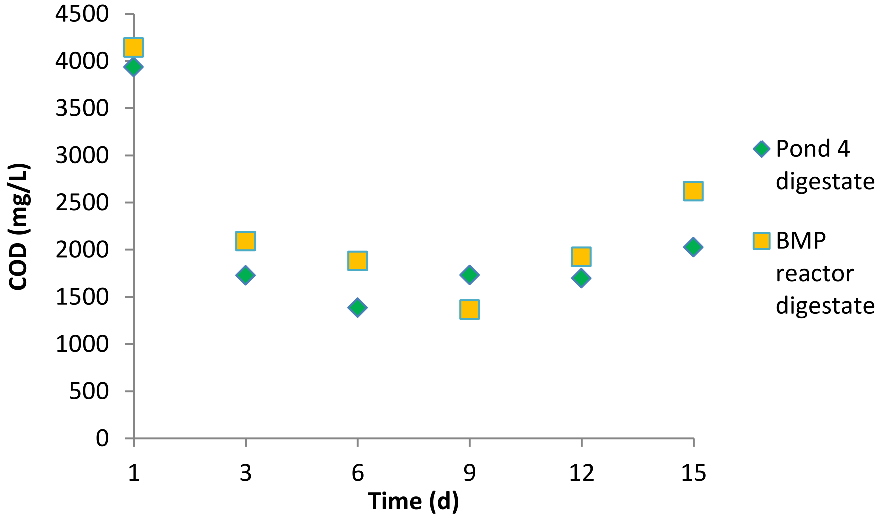

Figure 24: COD measurements (mg/L) performed every 3 days which corresponds to 3 cycles of filling, aeration, settling.

5. Discussion

a) DAF experiments

Experiment 1: Effect of coagulant/flocculant/pressurized water combinations

In practice, the total amount of air delivered to saturate the water depends on the saturator pressure and the recycle flow, which is typically 10 % (Edzwald 2010). Considering that the sample size was 200 mL, the addition of 400 mL and 600 mL of pressurized water (twice and three times the initial volume) is quite high, but it should be noted that this experiment is a laboratory batch test aiming at demonstrating that the separation of suspended solids in POME is possible via air flotation. Optimizations of theses volumes can be achieved at the pilot or industrial scales. It is understood that the water saturation step is energy consuming (operating the air compressor), and the lower the recycle ratio the lesser the operational costs. Considering the current results, an increase of 33 % in the input of pressurized water for a 30% decrease in the turbidity of the subnatant that will probably still need to be anaerobically digested, is not efficient.

One of the requirements from Goldtree was a treatment system that is low in chemical use to lower operational cost, but also to avoid frequent shipment, due to the difficulty of access to the plantation. The goal of this experiment was to determine the combination of coagulant and flocculant that would guarantee a separation independently of the changes in COD content of the POME keeping in mind Goldtree request. The combinations tested were matching a single coagulant to a single flocculant. A, possibly better, test method for ensuring flocs formation in wastewater of varying concentration of pollutants, would have been to combine anionic and cationic flocculant. Multiple studies report the advantages of dual polymer conditioning:

- *Chitikela and Dentel* (1998) report a significant reduction in optimal doses of chemicals used in dual system compared to the dose requirements for either chemical used separately.
- The study by *Fan, Turro, and Somasundaran* (2000) that investigated the effect of combining an anionic flocculant and a high molecular weight cationic copolymer for the removal of residuals after coagulation with aluminium salts, showed that the addition of the second, higher molecular weight polymer led to formation of larger floc and reduction of the optimum polymer dosage.

Experiment 2: Effect of PAX18 and A130/C492 on the solids separation at different temperatures

The studies on the effect of temperature on coagulation mostly report on the effect of temperature ranging from above water freezing point to room temperature. In a study by *Fitzpatrick, Fradin, and Gregory* (2004) the effect of temperatures ranging from 6 °C to 29 °C for a suspension of kaolin clay in tap water using alum, ferric sulphate and three polyaluminium chloride (PACl) as coagulant was investigated. Floc formation was slower at lower temperatures (6 ± 1°C) for all coagulants. Another study supports that at low temperature the viscosity increases, resulting in inhomogeneous distribution of the coagulant in the water with a poor coagulation efficiency (Kang & Cleasby 1995). The effect of temperature is also linked to alkalinity, and the best coagulation-flocculation results were obtained at a constant pH and at a temperature between 5-20 °C (Hanson & Cleasby 1990). No references where found on the effect of temperature above room temperature. Water temperature can affect fluid and particle motion, particle-particle interaction, rate and extent of hydrolysis of metal salt coagulants, and adsorption and precipitation rates (Kang & Cleasby 1995). One can speculate that with increasing temperature, the colloidal particles motions increase, affecting the efficiency of coagulation, but such claims will have to be further investigated for confirmation. A detailed cost analysis will have to be done to review the benefits of cooling towers vs. the additional cost of chemicals to obtain the same COD removal efficiency at higher temperature.

Experiment 3: Effect of different volumes of PAX18 and C492/A130 on the COD content of POME

The more coagulant was added, the higher the removal efficiency but the effect of increasing coagulant dose was minimal. A study by *Al-Shamrani, James, and Xiao* (2002) on the effect aluminium and ferric sulphates as destabilising agents for oil–water emulsions concluded that the destabilisation of the emulsions is more sensitive to pH variation than the increasing coagulant concentration, and the optimum pH conditions for destabilisation of the oil–water emulsion were close to the neutral pH.

Increasing volumes of flocculants from 20 mL to 30 mL led to lower removal efficiencies for both C492 and A130. This decrease in COD removal efficiency can be caused by the overdosing of flocculants. An overdose of polymer is an issue, because it creates a system considerably more stable than the initial system, because polymer adsorption is effectively irreversible (Hubbard 2006).

By adding 3 mL of PAX18, the expected COD removal of 90% was reached for a sample of 200 mL of POME. Considering that the concentration of aluminium in PAX18 is 90 g/L, the sample of 3 mL of PAX18 needed for 200 mL of POME contains 0.27 g Al. Therefore 1.33 g Al is needed to treat 1 L of POME. During peak season, Goldtree processes 4,000 tons of fruits during 24 working days in a month. The daily processing is then 167 tons/d. The production of POME per ton of fresh fruits bunch produced is 0.675 m^3 (Baharuddin et al. 2010). The daily production of POME is then 113 m^3/day. This production will require 150 kg Al/d. In terms of coagulant dosage, 20 mL of 5 g/L for a sample of 200 mL corresponds to 0.45 g/L and 51 kg to treat 113 m^3 of untreated POME. Taking into account the amount of aluminium needed to treat raw POME, using a DAF as a pre-treatment unit to anaerobic digestion is not environmentally or economically feasible. Although the COD removed reaches 9 0%, high quantities of Aluminium would remain in the effluent and sludge layer.

Experiment 4: Effect of different volumes of PAX18 and A130/C492 on solids separation for samples of anaerobically digested POME.

For a sample of 200 mL of anaerobically digested POME, 0.5 mL of PAX18 with a concentration of 90 g Al/L was needed. The corresponding aluminium content is 45 mg Al. It means that 225 mg of aluminium is needed to treat 1L of anaerobically treated POME. Comparing to the amount of aluminium needed to treat “raw” POME, it corresponds to 71 % less aluminium needed to treat the daily production of POME at Goldtree during a peak season. The quantities of polymer used are also reduced. In Experiment 3, 20 mL of C492 at 5 g/L were needed vs. 5 mL in Experiment 4, which is equivalent to 56.5 kg of polymer compared to 14.1 kg or 75 % less polymer.

The COD removal efficiencies obtained ranged from 81 to 86 %, including results obtained with anaerobically digested POME from pond 4 and fed batch reactor test bottles. These efficiencies were lower than 90 % obtained with untreated POME. In a pilot study by Wang et al. (2015), anaerobically digested POME with a COD content of 3,587 mg/L was treated via DAF and the COD removal efficiency reported was 88.65 %.

From experiment 1 - 4, it can be concluded that using a DAF as a post- treatment to anaerobic digestion has for advantages to:

- Eliminate the need for an SBR unit, since the wastewater has a COD content below the maximum limit for discharge.
- Reduce the use of metal coagulant chemicals, which makes the technology more environmentally and economically feasible.
- Reduce the production of sludge.
- Higher biogas recovery from the anaerobic digestion.

b) Anaerobic digestion

Biochemical methane potential (BMP)

A study by Kim et al. (2013) that investigated mesophilic co-digestion of POME with EFB, first performed BMP tests on POME that had a VS content of 37.0 ± 4.6 g VS/L and COD content of COD 49.2 ± 6.2 g/L. The BMP result reported was 397 ± 9 mL CH_4/g VS. The result obtained in this study was 291 ± 17 mL CH_4/g VS. The difference in methane yield of this experiment compared to Kim et al. (2013) can be attributed to the inoculum selection. De Vrieze et al. (2015) demonstrated the effect of the selected inoculum on BMP tests results. The comparison of the bio-methane potential of four substrates (molasses, bio-refinery waste, liquid manure and high-rate activated sludge) determined using four different inoculums resulted in significant differences on BMP results for two out of the four substrates. The authors assert that inoculum effect could be due to the abundance of methanogens in a given inoculum or a potential inhibiting effect of another inoculum. It is possible to obtain a higher methane yield using inoculum from an industrial biogas plant anaerobically digesting POME, rather than domestic wastewater treatment plant, since the microbial community in the industrial plant will be more accustomed to the digestion of POME.

Another possible cause is the low oil and grease content in Goldtree POME. The oil content is 190 mg/L, while values up to 4,000 mg/L are reported in literature (Goldtree Ldt. 2016, personal comunication, August 17; Rupani et al. 2010). These disparities could be due to the current processing conditions at Goldtree. Since the mill is only running at 30 % of its capacity, the retention times at the clarification is longer, and less oil is wasted in POME. In a study by Lauwers et al. (2012) the biodegradability of fat, oil, and grease (FOG) was tested in biochemical methane potential experiment. The FOG is theoretically suitable for anaerobic treatment, due to its high methane production potential, *i.e.* 900 - 1,400 mL CH_4/g VS (Alves et al. 2009). This study reported that co-digesting FOG and activated sludge quadrupled the biogas yield for a FOG loading around 50 % of the substrate VS. The degradation of FOG can also lead to an accumulation of VFA in which case it results in the inhibition of the methanogenesis and digester failure (Lauwers et al. 2012).

Fed batch reactor

The COD removal efficiency obtained is on the lower side of the 80 – 95 % expected in the "POME to energy" plant (Rahayu et al. 2015). A number of factors can explain these lower values. The main potential causes could be the increase in organic loading rate (OLR) and subsequent decrease in pH, which points to overloading. Bala, Lalung, and Ismail (2014) explained that the ORL is positively related to the biogas production, until a stage when methanogens cannot work fast enough to convert acetic acid to methane and the biogas production decreases. In anaerobic digestion, the microbial community is affected by pH changes and the methanogens are the most sensitive (Leslie et al. 1999). The optimum pH is between 6.8 and 7.2 (Gerardi 2006). The pH averages observed were within this range, despite the accumulation of VFA. Several cases of reactor failure reported in studies of wastewater treatment are due to the accumulation of VFA, causing a drop in pH, which inhibits methanogenesis (Bala et al. 2014). Fatty acids are believed to be toxic to methanogenic archaea, but the exact mechanism behind this toxicity is not yet fully known (Lauwers et al. 2012). Although, reactor failure is not concluded in this experiment, the increase in acetate and propionate form day 21 coincides with the decrease in pH and biogas production observed on day 25 to 31.

c) Sequence batch reactors

The maximum COD removal efficiencies observed were 64.78 % and 67.01 % for pond 4 and BMP reactors samples respectively. These values are lower than expected. A study by Chan, Chong, and Law (2010) that had for objective to investigate the aerobic treatment of anaerobically digested POME via sequence batch reactors (SBR) showed that COD removal efficiencies as high as 95 % to 96 % were possible. Various factors could have contributed to the lower removal rates. In Chan, Chong, and Law (2010) experiment a continuous stirring at a speed of 350 rpm was provided to homogenised the distribution of the wastewater which is not the case here. A new feed was filled into the mixed liquor of the previous cycle in Chan, Chong, and Law (2010) experiment when here the refill was done with the clarified effluent of the previous cycle since time limitation allowed only one BMP test from which the digested POME could be collected. Most importantly, the water losses through evaporation contributed to not only the lower COD removal rates but also to observed COD increase towards the end of the experiment.

6. CAPEX and OPEX calculations

The scope of the thesis was to review the possibility to upgrade the water treatment system at Goldtree to a cleaner, more efficient system. The technical data obtained from experiments 1 to 4 are indicating that the POME can be treated to internationally accepted COD content norms at discharge. These treatment options can be combined to the production of biogas, which will provide a major economic benefit to Goldtree. The anaerobic digestion tests, although demonstrating low conversion rates, show a potential for energy recovery. A biogas plant with a DAF as post treatment is the ideal combination to merge ecological and economic benefits together.

In the next four to five years, Goldtree will be investing in the expansion of its plantation, and profits are only forecasted for 2023 (Figure 25), it is then important to review the investment and running costs of both projects, and synchronise with the Goldtree business plan.

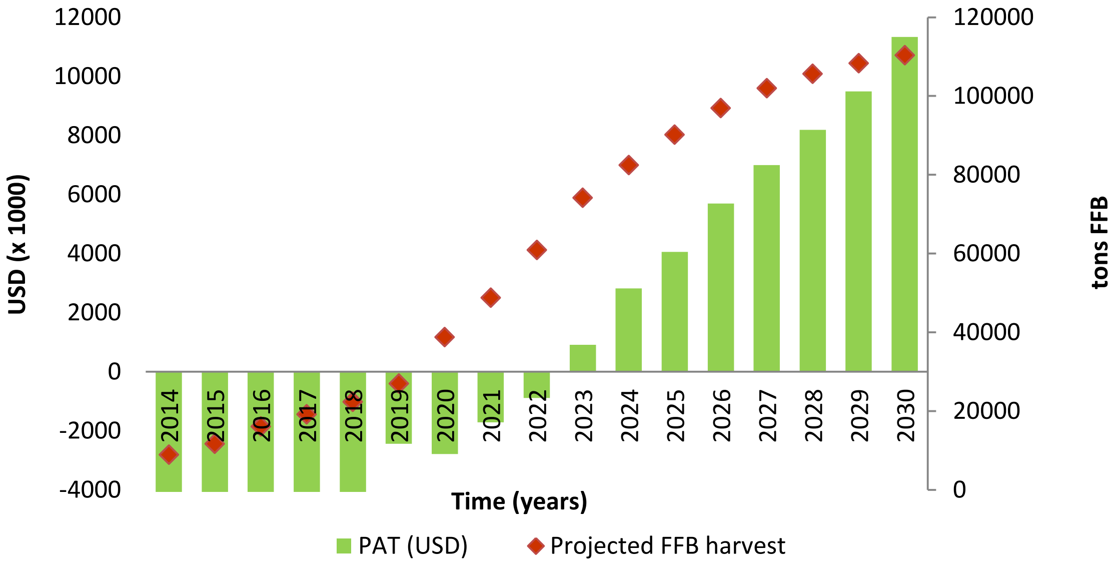

Figure 25: Goldtree profit after taxes (PAT) in US dollars. This graph shows that the company is investing in expending its plantation, and profit from the sale of oil palm is foreseen for 2023.

The calculations below follow the proposed option of installing a DAF as a post-treatment unit to the current ponding system (short term option) and a DAF after a CAL (long term option).

a) Dissolved air flotation

Goldtree is currently running at 20,000 tons FFB/year, which will increase to 100,000 tons FFB/year in the next 10 years. During the peak season which last 4 months (February to May), 14 % of the yearly harvest is processed. The values listed consider 0.675 m^3 POME/ tons FFB, 24 days of processing per month and 8 hours of processing a day (Table 8). It is assumed that the inlet flow to the current ponding system or CAL installation in the future is equal to the outlet flow (inlet to DAF).

Table 8: Max POME production considering a peak month at the expected harvest of 2025. These values are calculated considering 0.675 m^3 POME/ tons FFB, 24 d/month and 8 h/d.

Parameter	Unit	Value
TFFB	ton/yr	101,971
POME production	m^3/month	9,636
	m^3/d	401
POME flowrate	m^3/h	50
COD inlet concentration	kg/m^3	33.80 ± 2.32

Running costs of a DAF unit are mainly determined by power, chemical usage, spare parts and overheads. Table 9 is an example of the electrical running costs for a 50 m^3/h DAF unit (Eddie Broeders 2016, personal communication, October 27).

Table 9: Example of electrical running cost associated with operating a 50 m^3/h DAF unit

Process Part	Relative power output due to lower frequency	kW	Total (kW)
Feed pump	0.50	3.00	1.50
Recirculation pump	0.60	5.50	3.30
Scraper drive	0.25	0.37	0.10
Polymer concentrate pump	0.10	0.75	0.10
Dosing pump polymer	1.00	0.25	0.30
Sludge pump	0.10	1.50	0.20
Mixer poly make-up	0.10	0.75	0.10
Total			5.40
kWh/m^3			0.11

The greatest operating expense of an industrial wastewater treatment system is in chemical usage (FRC Systems 2015). When finding ways to reduce operating cost, starting with decreasing the chemical usage is a good place to start, but attempting to save money by eliminating the use of chemicals altogether in the case of POME is not possible. Attempts at separating the suspended solids in jar tests without coagulant and polymers failed. Industrial DAF systems can still remove a significant portion of these solids without added chemistry, but then the floating sludge is diluted and the effluent is not of good quality (FRC Systems 2015). Chemical costs are directly related to the flow rate and wastewater composition (*i.e.* solids concentration, oil content, pH), and can vary widely from application to application (FRC Systems 2015). Experiment 1 to 4 show that there is a reduction of 73 % in chemical use when treating anaerobically digested POME compared to untreated POME. The values obtained in these batch experiments can be optimized further during a pilot or full scale operation. The unit price of aluminium chlorides range from 400 – 2,200 EUR/ton, while polymers powders are sold at 2.75 EUR/kg (Gebbie 2006). The values listed in Table 10 consider the treatment of 10,000 m^3 of anaerobically treated POME, corresponding to the monthly production of POME during peak season in approximately 10 years.

Table 10: Estimated chemical cost for the treatment of 10,000 m^3 of POME per month

Parameter	**PAX18 Liquid (concentration = 100%)**	**C492 Powder (concentration = 100%)**
Concentration	90 g/L	5 g/L
Quantities needed	2,250 kg	1,250 kg
Unit Price	400 - 2,200 EUR/tons[4]	2.75 EUR/kg[5]
Cost	**900 - 4,950 EUR/month**	**3,438 EUR/month**

The chemical cost obtained at this point is not economically viable. However, these are calculated based on the volumes needed to obtained separation while jar testing. Since the batch test values can be optimized, the prices listed can be reduced.

There is a wide range of DAF suppliers providing different DAF systems and price/quality ratio must be reviewed carefully, since Goldtree requires a system that is robust enough to be operated in remote areas with limited supervision, while limiting cost. The purchase cost of a 50 m^3/h DAF unit is estimated at 70,000 - 130,000 EUR (Goldtree Ltd. 2017, personal communication, May 26). Shipping, duties, installation, commissioning cost can easily double this price.

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[4] (Gebbie 2006)

[5] Unit prices were provided by Nijhuis industries via internal communication

b) Covered anaerobic lagoon

The CAL systems are much more common than DAF in the oil palm sector. In Africa, three CAL systems were installed and commissioned in Ghana, Gabon, and Nigeria since 2015. An estimated CAPEX and OPEX cost for Goldtree extrapolated from BIOTEC and CIRAD feasibility studies on Presco Plc, Nigeria CAL installation is given in Table 11 - 14. Calculations on potential saving are based on the results of the BMP and fed batch reactor tests. Some assumptions are made using values reported by Rahayu et al. (2015).

Table 11: Anearobic lagoon characteristics

Parameter	Units	Value	Remarks
Biodigester size	m^3	8,000 - 10,000	Calculated based on the influent flow and an HRT = 20 - 25days
Hydraulic retention time	d	20 - 50	Typical in commercial biogas plants for POME (Rahayu et al. 2015)
COD removal efficiency	%	83	Based on fed batch reactor test
Influent COD load	T/d	13.6	Calculated based on flowrate and COD concentration

Table 12: Estimated OPEX cost extrapolated from a feasibility study from BIOTEC and CIRAD

Parameter	Unit	Value	Remarks
Operation			
Human resource	€/year	15,000	
Consultant supervision	€/year	10,000	
Lab analysis	€/year	5,000	
Insurance	€/year	5,000	
Subtotal	**€/year**	**35,000**	
Maintenance			
Bio-digester	€/year	30,000	
Biogas handling area	€/year	30,000	
Subtotal	€/year	60,000	
Total O&M cost	**€/year**	**95,000**	

Table 13: Estimated CAPEX cost extrapolated from a feasibility study from BIOTEC and CIRAD

Digester system installation cost			**Remarks**
Civil woks	€	200,000	
Equipment	€	100,000	
Electrical works	€	100,000	
Engineering	€	100,000	
Subtotal	**€**	**500,000**	
Biogas system installation cost			
Civil and hydraulic works	€	150,000	
Equipments	€	400,000	
Electrical works	€	150,000	
Engineering	€	150,000	
Shipping and insurances	€	50,000	
Subtotal	**€**	**900,000**	
Management cost			
Project management	€	50,000	
Contingency budget	€	40,000	
Profit	€	100,000	
Subtotal	**€**	**190,000**	
Total Project cost	**€**	**1,590,000**	

The prices listed in Table 12 and Table 13 can vary depending on the contractor providing the engineering plans, the insurance provider and shipping and custom costs. According to Rahayu et al. (2015), the investment costs of covered lagoon range from USD 1.5 – 3 million per MWe of power generation from biogas. The potential power generation for Goldtree, based on the BMP results (Table 14) is 0.69 MWe (Table 15), which corresponds to an investment cost between USD 1 - 3 million. The extrapolated values from Table 13 are within the ranges reported by Rahayu et al. (2015).

Table 14: Biogas production potential

Parameter	Unit	Value	Remarks
Conversion efficiency to CH_4	%	53	Based on BMP test result
Biogas captured	m^3/d	5,989	Based on kg of COD removed and conversion efficiency
Average CH_4 content	%	73	Based on the Fed batch reactor test
Methane captured	m^3/d	4,372	

Table 15: Potential cost savings calculation

Parameter	Unit	Value	Remarks
Methane caloric value	MJ/m^3 CH_4	35.7	Values reported in Rahayu et al. (2015)
Gas engine efficiency	%	38	
Potential electricity production	MJ/d	59,311	
Potential electricity production	KWh/d	16,480	
	MWe	0.69	
Biogas to diesel equivalence	L/ m^3biogas	1.03	
Diesel savings	L /d	4,503	Calculated using biogas diesel equivalence from Galicia and the methane captured estimation (Table 14)
Diesel cost	€/L	0.77	(Goldtree Ltd 2017, personal communication, May 15)
Saving	**€/d**	**3,467**	Calculated using diesel unit cost and the methane captured per day

The savings calculated are highly influenced by the use of biogas. A CAL system has limited biogas storage capacity, thus, if not used for the production of electricity, the biogas has to be flared in which case the savings decrease. For instance, if only 60 % of the biogas production calculated in Table 14 is converted to electricity, the potential savings are reduced to 2,080 EUR/d or 60 % of the initial saving in Table 15. The calculations above correspond to a maximum foreseen POME production and with lower POME production, the methane production decrease as well as the potential savings.

c) Sludge disposal

An important aspect with respect to the operation of a DAF and CAL is how to dispose the sludge produced in both units.

Experiment 1 and 4 show that 70 % less sludge is produced when treating anaerobically digested POME with air flotation. Reducing the sludge production decreases the operational cost. The DAF sludge has moisture content above 95 %, which makes transportation and disposal difficult and costly (Steele & West 1989). Disposal options for Goldtree are limited. Locally, there are no industries that would be interested in processing the sludge, thereby reducing the amount of wastes deposited. Rendering industries, processing agro-industrials waste, also encounter several problems with the treatment of DAF sludge such as the impaired drying due to the high moisture content inhibiting heat transfer, and the added polymer that can also create a coating in the cookers (Steele & West 1989).

A feasible option for both DAF and CAL excess sludge is land application in the form of compost. Besides the liquid waste, an oil palm industry has to dispose of its solid waste, especially EFB that represents 28.5 % of the FFB, and it cannot be incinerated, due to its high moisture content (60 - 70 %) (Pleanjai et al. 2004; Kim et al. 2013).

Gurmit, Manoharan, and Kanapathy (1981) reported an average EFB nutrient content of 0.8 % N, 0.1 % P, 2.5 % K and 0.2 % Mg on a dry weight basis, which makes it a suitable organic fertilizer. Various techniques have then been tested for the composting of EFB and sludge. In a study by Baharuddin et al. (2009), EFB was mixed with anaerobically treated POME from an anaerobic pond. The AD treated POME was sprayed onto the shredded EFB to maintain the moisture content between 50 to 60 % throughout the composting process. The composting piles were turned over 1 – 3 times per week. Maintaining proper aeration is important for increasing degradation rate, controlling the temperature and avoiding anaerobic conditions that can affect the quality of the compost pile (Michel 2008). The temperature was reported in Baharuddin et al. (2009), to fluctuate between 50 and 62 °C, before decreasing at a later stage of the process. The pH of the system (7.75 – 8.10) did not significantly change. The mature compost contained very low levels of heavy metals, and the N, P_2O_5 and K_2O content were 2.26, 3.30 and 2.25 % of the total matter, respectively. The reduction in volumes was 40 - 50 % (Epstein 1997). The transportation and distribution of EFB in the field, as well as the frequency at which the material is turned are factors that affect the cost of composting (Michel 2008). In Goldtree, the transportation services are outsourced, and running cost of composting are estimated in Table 16.

Table 16: Breakdown of operation cost associated with composting

Services	**Parameter**	**Unit**	**Value**
Transportation	**Trucks capacity**	ton/truck	7 - 8
	Unit Price	EUR/ton	5
	Cost	**EUR/truck**	**35 - 40**
Mixing	**Cost**	**EUR/h**	**32**

The DAF sludge will contain aluminium that is toxic to plant inhibiting root growth (Delhaize & Ryan 1995). There is considerable variability in Al tolerance within crops varieties. In a study aiming at investing the growth inhibition of oil palm varieties under Al stress, Supena et al. (2014) tested five oil palm varieties i.e. PPKS239, PPKS540, PPKS718, Simalungun, and Dumpy at five different concentrations (0, 75, 150, 225, and 300 ppm). The study reports that at the maximum concentration of Al tested (300 ppm), the length of Simalungun primary roots was stable while PPKS718 and PPKS540 varieties had their roots length decreased to 24.3 and 12.4 % respectively.

General conclusions

In this thesis, options for the improvement of an oil palm wastewater treatment in Sierra Leone were investigated. Initially, three systems were proposed (DAF, CAL and SBR) to reduce the COD level to the required limit of 250 mg/L. The SBR was considered optional, and only necessary if the targeted COD concentration in the effluent was not reached after the DAF and CAL. Lab-scale experiments for DAF and CAL and SBR were conducted.

The metal salt coagulant PAX18 and polymer A130 and C492 gave the best sludge/subnatant separation. The quantity of pressurized water added played a crucial role in achieving a clear separation. Where 600 mL gave the best results, 400 mL was chosen to follow the practical limitations of a DAF system. Lower temperatures (30 °C) are more beneficial to achieve the expected COD removal rates which imply that there is a need for a cost analysis for the installation of cooling towers. A COD removal rate of 90 % is achievable for raw POME using a DAF, but the chemical cost is too high to make it economic viable.

For anaerobically treated POME, high COD removal efficiencies and COD effluent concentrations below the international required standards were achieved with air flotation, resulting in 71 % less aluminium needed, 75 % less polymer needed, 50 % less water consumption, and 70 % less sludge produced compared with treating raw POME. The chemical cost of approximately 100,000 EUR/year to treat 40,000 m^3 of anaerobically treated POME is still on the high side.

A methane yield of 291 ± 17 mL CH_4/g VS corresponding to 53 ± 0.03 % COD conversion efficiency was obtained. As for fed batch reactor, the observed COD removal efficiency was 83.14 ± 11.59 %, and an average methane yield of 222 ± 77 mL CH_4/g VS was achieved, corresponding to 40 ± 0.14 % COD conversion efficiency to methane. All these values were much lower than the expected 70 – 80 % COD conversion efficiency to methane, which can be due to inoculum characteristics and/or the low oil content in Goldtree POME. In any case, the Goldtree POME with average COD of 50,000 mg/L can be considered suitable for a biomethanization installation.

The SBR operation did not reach the expected COD removal efficiencies (95 % to 96 %). The maximum COD removal efficiencies observed were 64.78 % and 67.01 % for pond 4 and BMP reactors samples respectively. These results are attributed to problems preventing evaporation and time constrains.

Based on the lab experiments and CAPEX study, it can be concluded that a DAF as post-treatment to anaerobic digestion is the best option, since it offers multiple advantages, such as:

- Reduction of the use of metal coagulant chemicals, which makes the process more environmentally friendly and economically feasible.
- Decrease in the production of sludge.
- Increase in biogas production, which can be recovered for renewable energy recovery.

The main challenge of DAF installation seems to be the high operational cost, due to high chemical cost. More research is needed to analyse suitable minimal chemical dosages on anaerobically treated POME. This should be done while also focusing on the optimal quantity of pressurized water to be added. It is possible that increasing the amount of pressurized water will contribute to decreasing the quantities of chemicals, which would result in a cheaper and more efficient DAF treatment.

Future perspectives

A number of factors have not been studied in this report, and these can be the object of future investigations. First and most importantly, the chemical dosage optimization should be investigated. Experiment 4 showed that anaerobically treated POME required less chemicals and less pressurized water at the air flotation stage than untreated POME but the conversion of the current quantities to the industrial scale led to high operational costs. Investigating the effect of cationic polymers for coagulation, instead of metal salts on POME, can also be an interesting option, since some of the advantages of using a polymer compared to aluminium are:

- Lower coagulant dose requirements,
- Decreased volumes of sludge production,
- Decreased ionic loads of the treated water,
- Reduced levels of aluminium in treated water,
- Increased cost savings up to 25–30% (Rout et al. 1999)

Combining anionic and cationic polymers can improve floc formation in wastewater of varying concentration of pollutants. Since the COD content in POME varies, depending on fruit milling processes, the effect of combining polyelectrolytes could be investigated.

Second, most of the studies on the effect of temperature on coagulation were focusing on the effect of low temperatures (1 - 6 °C). Since temperature affects the motion of particles, one can consider that with increasing temperature, the colloidal particles motions increases, possibly decreasing the efficiency of coagulation.

Third, since the BMP test did not give the expected high methane yields, another trial using inoculum that adapted to the digestion of POME could be carried out to verify if higher conversion efficiencies and methane yields can be obtained.

Lastly, it is noteworthy that a SBR treatment might be a good, cheaper, alternative for the DAF treatment. Further lab scale pilot trials will be needed on anaerobically treated POME to study the COD removal rate. If the SBR treatment does not lead to sufficiently low effluent concentrations, a combination of CAL – DAF – SBR or CAL – SBR – DAF could solve the problem, with DAF running at low chemical dosage.

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