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DEWATERING AND RECOVERY OF FATS, OILS AND GREASE (FOG) OF GREASE TRAP WASTE

A design-research of a new-built process

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OF GREASE TRAP WASTE**

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Master's Thesis
November 2013
Degree programme in Industrial Management
Oulu University of Applied Sciences (School of Engineering)

ABSTRACT

Oulu University of Applied Sciences
Degree programme in Industrial Management

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Title of thesis: DEWATERING AND RECOVERY OF FATS, OILS AND GREASE (FOG) OF GREASE TRAP WASTE. A design-research of a new-built process.

Supervisor: Hannu Päätaalo

Term and year of completion: Autumn 2013

Number of pages: 66

This thesis is a design science research study about the development of a process to dewater waste collected from grease traps (GTW). Grease traps are installed in professional kitchens and at food manufacturing sites to protect the sewage works from grease waste that can clog the sewer system and disrupt the biological treatment at wastewater treatments plants (WWTP). When traps are emptied hot water is used to clean the units. Therefore GTW can contain up to 99% water. During 2008-2009, the waste centres and WWTPs in the Oulu and Kainuu regions in Finland collected 6.6 kg GTW per capita annually.

GTW consists of water, partly hydrolyzed fats, oils and greases (FOG), food residues, and other contaminants as soap and metals. The grease is emulsified throughout the water phase and mixed with solids, which makes it difficult to dewater. Dewatering is important as it decreases the amount for waste disposal, which is expensive as GTW is classified as special waste. Additionally, the upcoming legalisation in 2016 forbids disposal of organic waste to landfills and therefore cost-effective alternatives have to be found.

A fairly simple process was designed to dewater GTW by using acid hydrolysis and moderate heating (65-70°C). The process was tested first on laboratory scale from where it was step-wise scaled up. In 2012, the process design was tested and evaluated by a pilot plant. The goal was to gather as much information and experiences as possible, in order to improve the process products' quality and the overall process efficiency for the next production design. The profitability of the design was tested by comparing estimated profit margins in different scenarios. The study contains also an overview of possible technologies to utilise or dispose the dewatered fraction (also called brown grease) resulting from the process.

The conclusion of the study is that the designed dewatering process is profitable when used at full operating capacity (3000 t/a) and a dewatering efficiency of at least 75%. In this case the estimated payback time is 3-6 years from start-up. The plant was not suitable for extended dewatering of the FOG fraction. An own unit is needed for this purpose. Otherwise, reuse alternatives should be search that can deal with the remaining water content, such as biogas production or utilisation as steam boiler fuel or low-grade soaps. The test design still needs technical adjustments before it can be taken in use. Further research is needed to find a solution how to destabilize the intermediate layer of organics, light solids and water formed between the FOG and water phase (rag layer). For the moment, the fraction remains a non-valuable waste.

Keywords: Grease trap waste, brown waste, FOG, dewatering, fatty acids, rag layer, reuse

CONTENTS

ABSTRACT.....	2
ABBREVIATIONS AND TERMS	5
1 INTRODUCTION	6
1.1 Business case.....	6
1.2 Process design	8
1.3 Research study.....	9
1.4 Design science research.....	10
2 THEORETICAL BACKGROUND	12
2.1 Grease trap	12
2.2 Grease trap waste (GTW)	14
2.3 GTW utilisation alternatives	17
2.3.1 Utilisation for industrial applications	17
2.3.2 Anaerobic digestion (biogas production).....	19
2.3.3 Composting.....	21
2.3.4 End-of-Waste status	21
2.3.5 Incineration	21
2.3.6 Pyrolysis.....	22
2.3.7 Boiler fuel applications	24
2.3.8 Biodiesel production.....	24
2.3.9 Dewatering and storage in a geotube	26
2.4 Available separation technologies.....	27
2.4.1 Mechanical separation techniques.....	27
2.4.2 Acid hydrolysis	30
2.4.3 Use of organic solvents.....	30
2.4.4 Enzymatic hydrolysis	31
2.4.5 Saponification by alkali addition	31
2.4.6 De-emulsifying	32
3 TEST METHODS.....	33
3.1 Test arrangements for pretesting	33
3.2 Test arrangement for the pilot study.....	34
3.3 Restrictions during the pilot study	36

3.4	Financial feasibility analysis	37
4	RESULTS	39
4.1	Pretesting on laboratory scale.....	39
4.1.1	Heating and settling	39
4.1.2	Heating and acid hydrolysis	40
4.1.3	Saponification prior acid hydrolysis.....	43
4.2	Pilot plant results.....	43
4.2.1	Dewatering of GTW	44
4.2.2	Fractionation of the dewatered mass (FOG recovery)	47
4.2.3	Financial feasibility	49
4.2.4	Evaluation of the project management (pilot plant)	54
5	CONCLUSIONS	55
6	RECOMMENDATIONS FOR FUTURE WORK	56
	LIST OF REFERENCES.....	58

ABBREVIATIONS AND TERMS

Brown grease	Dewatered organic fraction from grease trap waste
BTU	British thermal unit. The amount of heat required to raise the temperature of 1 pound (0.454 kg) of liquid water by 1 °F at a constant pressure of one atmosphere.
C:D	Lipid number, where C is the number of carbon atoms in the fatty acid and D is the number of double bonds in the fatty acid.
DAF	Dissolved air flotation
DSR	Design science research
EoW	End of Waste
EWC	European Waste Codes / European Waste Catalogue
FAM	Fatty acid mixture
FFA	Free fatty acid
FOG	Fats, oils and grease
GTW	Grease trap waste (also trap effluent)
IH ²	Integrated Hydropyrolysis plus Hydroconversion
Lipid	Any of a group of organic compounds that is soluble in nonpolar organic solvents and general insolubility in water, such as fats, oils, waxes, sterols, triglycerides
NREL	National Renewable Energy Laboratory of the U.S. Department of Energy
ppm	Parts per million (as mg/kg)
rag	Intermediate layer formed during separation of oil and water
RSD	Research science design
TOC	Total organic carbon content (in %)
TOG	Total oil and grease content (in ppm or %)
TOFA	Tall oil fatty acid
VS	Volatile solid
WtE	Waste-to-energy
WWTP	Waste water treatment plant
%(V/V)	Volume concentration, the volume of a constituent relative to the volume of the total solution (in %)
%(w/w)	Mass fraction, ratio of the mass of a substance to the mass of the total mixture (in %)

1 INTRODUCTION

This chapter contains a description of the business case (the problem definition), the process design (the solution under study) and a short explanation on how the experimental study was made and the research method used.

1.1 Business case

Fats, oils and grease (FOG) are unwanted substances in sewages and wastewater treatment plants (WWTP). When a stream of warm greasy wastewater cools down in a sewer system, edible fats and greases solidify and form with other disposed solids blocks further up in the drain pipe. Fats may also cause growth of mycelia, a part of fungus, which demands oxygen while decomposing organic compounds. Grease containing sewage water affects therefore the availability of oxygen needed for biological treatment of wastewater, causing additional costs and energy demands at WWTPs. It also makes sewage sludge drying difficult. (Peltonen – Enström - Pääkkönen 2007.)

To protect sewages and WWTPs against inconveniences described above, FOGs are removed by grease traps before they enter the sewer, as well as before they enter the WWTP. In this study it was found that in 2008-2009 the annually collected amount of grease trap waste (GTW) from local grease traps and WWTPs was 6.6 kg per capita in the Finnish regions of Kainuu and Oulu (367 500 inhabitants in total). For Finland with a population of 5.4 million people this would roughly mean over 36 000 tonnes of collected GTW annually. This quantity most-likely increases in the near future, because of more strict regulations which increase the emptying intervals of grease traps built after 2007 (DI 2007). To compare, the total amount of sludge collected from services and households in 2009 was 271 000 tonnes, together with 15 000 tonnes from the food and beverages production industry (Waste statistics 2009). The share of GTW from the total amount of sludge would then be about 12%.

In a recent article in the newspaper Helsingin Sanomat (2013), Sami Sillstén, manager of the Helsinki Region Environmental Services Authority (HSY) reported that they remove nearly monthly about 40 tonnes of grease waste from the sewer from just one spot in the city centre of Helsinki. The grease is originated from restaurants located in that area. These restaurants should have well working grease traps serviced according the regulations, but the problem is that they

are not sufficiently inspected by the local authorities. Grease traps may be full, improperly working or traps are lacking completed. (Halminen & Rissanen 2013.) Based on this news, the Finnish Water Utilities Association (FIWA) started an investigation about grease deposits in sewer networks throughout Finland. FIWA wants to find out how often water service corporations need to remove grease left-over from the sewer system. They are also interested to know if other cities have been able to allocate sources of grease deposits. (Salomaa 2013.)

Collected GTW is classified as special waste, which makes its disposal expensive. Special waste should be transported by registered waste carriers and disposed in accordance with the current legislation. GTW is not hazardous, but needs nonetheless special transportation equipment and pre-treatment because of its high water content, which can be up to 99%. The Finnish Government Decision 1049/1999 does not allow discarding of liquid waste, i.e. waste containing more than 200 litres of free water, wastewater or a by viscosity water-like liquid, on landfills. GTW should therefore be dewatered prior landfill disposal. Dewatering of GTW is complicated, as fats are emulsified or soapified due to use of cleaning detergents and hot water, and mixed with solids.

In the near future, dewatering prior landfilling only is not sufficient. From January 2016, the Finnish regulation for landfills set by the Ministry of the Environment forbids disposal of waste containing more than 10% organic matter on landfills for non-hazardous waste (Valtioneuvoston asetus rajoittaa orgaanisen jätteen sijoittamista kaatopaikalle. 2013). Dry GTW contains 80% to 100% organic matter.

Landfill disposal plants and waste collections companies have an urgent need for a solution how to deal with their collected GTW. This study describes a technical solution how to maximize the dewatering of GTW in order to minimize the amount of waste. Additionally, alternatives are given how to utilize or dispose the organic dewatered fraction cost-effectively according the upcoming regulations.

The process solution presented in this work is confirm the European waste hierarchy of (1) prevention (dewatering reduces waste generation), (2) reuse and preparation for reuse (in industrial applications), (3) recycle (composting or biofuel production) or (4) recovery (e.g. incineration as a co-fuel). The less favourable option (5) disposal (landfilling, waste incineration, waste gasification and other finalist solutions) is minimized.

Besides its environmental improvement, the process may help to make the GTW collection system more effective and organized. By improving the waste collection logistics and reducing waste disposal costs by dewatering, the GTW collection service will become more cost-effective, which makes the service easier to extend. In other words, waste collection companies can use the dewatering process to expand their market share in GTW collection services.

1.2 Process design

The technology under study is a fairly simple process to decrease the amount of collected GTW by fractionating it into sewer eligible water, some solids, and an organic fraction for reuse or final disposal. The main effort of the process is that it can save up to 85% on special waste disposal costs.

The developed process is able to deal with the fluctuating grease and water contents in collected GTW. Low grease content in the raw material just extends the time needed to collect a full batch of dewatered mass. The capacity of the pilot plant described in this study is 23 m³, which practically means that the plant can deal with three collected batches of GTW (3 - 7 m³ per suction vehicle) at the time. The take-in of raw material as well as the dewatering process is automated, which makes the process suitable for continuous use, though the procedure it serves is a batch process. The dewatering process works at atmospheric pressure and relatively low temperatures (65 - 70 °C). The process does not use expensive catalysts or enzymes, only regular chemicals as sulphuric acid and sodium hydroxide are used.

The mass of the aqueous GTW (up to 99% of water) is weighed at the waste collection terminal, where after it is pumped straight from the suction vehicle into the receiving tank. GTW is preheated (40°C) and stirred slowly to homogenize before pumping it to the reactor. In the reactor, the mass is acidified (pH <3) to speed up the separation of grease and water, and settled for 2 to 4 hours at 65 - 70°C. Water is decanted off at the bottom of the reactor. The pH of the discard water is raised (pH >8) by sodium hydroxide, before pumping the discard water into a flotation unit (DAF). This will remove remaining grease contents and lighter solids by forming foam (soap) which is scraped off the water phase. Remaining heavier solids settle in the following settling tank, which is installed before release to the sewer. The in the reactor remaining greasy top layer is dewatered further in a separation column where after the dirty water fraction is

pumped back to the receiving tank. The dewatered organic fraction is transferred to the product collection tank for reuse purposes.

The product of the process, about 15% to 45% of the raw material, is a partly dewatered organic fraction of mainly fatty acids. The final water content of the dewatered mass depends on the way the product will be reused or disposed; optimal dewatering is in many cases not needed. Even when the process product still has high water content (in this study up to 60%), it can for example be used as a feedstock for low-grade soap used for road cleaning, for energy production (e.g. biogas, boiler fuel), or be composted. The less favourable option for disposal would be incineration or gasification as a waste. Landfill disposal is not an option for the near future due to the upcoming regulations in 2016.

1.3 Research study

The research study was initiated by a business partner in the waste disposal business. They needed a solution on how to deal with collected GTW according to the upcoming legislation. As little information was available about the annually collected quantity of GTW, an inventory was made at two municipal waste centres in Northern and Central Finland during one year. Wastewater treatment plants and a biogas installation were visited in Central Europe and a literature review on known separation technologies for GTW was initiated. Potential separation processes for GTW were pretested on laboratory scale, optimized and scaled up.

Based on the obtained empirical results, a so called α -process design for a pilot plant was prepared in close cooperation with a waste collection company. The goal was to gather as much information and experiences as possible, in order to improve the process products' quality and the overall process efficiency for the next production design. The purpose of the pilot plant was to provide quantitative proof that the designed process has technical potential to succeed on full scale basis. It was not built with production or operating efficiency in mind. Nevertheless, once the prototype design proves the concept, these considerations can be added to the prototype design as part of a formal production design, or as a component of a regular operations implementation project.

Before the building of the pilot plant could be initiated, an environmental permit had to be applied from the State Regional Administrative Agency (AVI) in Finland. The environmental permit

describes the specific operations of individual facilities, the way of data analysis and how the results are reported to the supervisory authorities. It also incorporates limits defined for emissions, and formulates emission monitoring methods and test schedules. Monitored substances include also raw materials, chemicals, process water or other substances used in the operation. Prior testing, a risk assessment was submitted to the regional Centre for Economic Development, Transport and the Environment (ELY Centre). ELY centres are monitoring activities covered by environmental permits throughout their life cycles in cooperation with municipal environmental protection officials. (Ympäristö.fi 2013.)

Reports were written from every test run. In this way it was possible to check afterwards the quantities of received GTW, the amount of fed chemicals as well as the amount and quality of the waste water and solids removed. By allowing a method of trial-and-error during testing, blind spots in the chemical process, technical design problems, and gaps in safety issues were instantly observed and resolved in order of importance. All observed special and exceptional situations and problems with their possible solutions were listed and prioritized. This resulted in a clearly scheduled *action plan*, which was constantly updated during the testing process. Throughout the process of learning from mistakes and problem solving, the process design improved and developed continuously. At the same time an instruction manual was drafted for future use. After the testing phase the process was evaluated as a whole. Recommendations were made about to how to proceed with further process development and how to initiate the use of the already built pilot plant.

This report is summary of knowledge obtained from the literature review, the laboratory experiments and the pilot study. It also contains an estimation of the cost-effectiveness of the process.

1.4 Design science research

The research method used in this study is design science research (DSR). The DSR process involves the search for a relevant real-world business problem, the design and construction of an artefact (a construct, model, method, or instantiation), and its' ex ante (based on prior assumptions) and ex post (measures of past performance) evaluation. DSR is a general research approach with a set of defining characteristics and can be used in combination with different research methods (Gregory & Wayne 2010, 5).

Design science research is conducted most frequently within a positivistic epistemological perspective (Gregory & Wayne 2010, 6). Positivism is a philosophy of science which is based on knowledge gained from 'positive' verification of observable experience rather than, for example, introspection or intuition. Scientific methods or experimental testing are the best way of achieving this knowledge. Epistemology is referred to as "theory of knowledge". It questions what knowledge is and how it can be acquired, and the extent to which knowledge pertinent to any given subject or entity can be acquired.

The goal of DSR is to develop knowledge to describe, explain and predict a problem domain (Van Aken 2005). Knowledge and understanding of the problem domain and its solution are achieved in the building and application of the designed artefact. The outcome is mostly an individual or local technology-based solution, which can be used to design solutions for specific field problems. The results cannot be readily generalized to other settings. (Gregory & Wayne 2010, 6. Hevner 2004.)

The utility, quality, and efficacy of a design artefact must be rigorously demonstrated via well-executed evaluation methods. Effective DSR must provide clear and verifiable contributions in the areas of the design artefact, design foundations, and/or design methodologies. DSR must be presented effectively both to technology-oriented as well as management-oriented audiences. (Järvinen 2005, 111.)

2 THEORETICAL BACKGROUND

This chapter is a summary of the findings from the literature study. The first subchapters describe the working principals of a grease trap and explain the composition of grease trap waste. The third subchapter contains a summary of currently used utilisation and disposal options for GTW. The last chapter is an overview of different techniques which can be applied for the fractionating of GTW.

2.1 Grease trap

Grease traps, or gravity interceptors, are plumbing devices installed to prevent formation of blockages by grease and solids in drain pipes. They also avoid impeding of wastewater treatment. Grease traps appear especially in the food industry, such as professional kitchens, grillrooms, caterings, slaughterhouses and the meat and fish processing industry. WWTPs may require the installation of grease traps on other locations as well (DI 2007, 54), such as at vegetable oil refineries, laboratories, laundries, hospitals, storages and parking places. To protect WWTPs from passed through greases and fats, traps are also installed as a pre-treatment step before the sewage water reaches the biological part of the WWTP process. Air bubbles may be introduced causing the grease to float to the surface of the water where it may then be removed by a skimming device.

A traditional grease trap or interceptor is based on separation by gravity. Solids heavier than water sink to the bottom, while the lighter fats and oils float on the water surface. The water fraction in the middle continues through the outlet pipe to the drain (see figure 1). In the traps, greases are normally subjected to a natural enzymatic hydrolysis which breaks fats into free fatty acids and glycerol. The water-soluble glycerol is washed out with the wastewater, while the liberated free fatty acids float on the water surface.

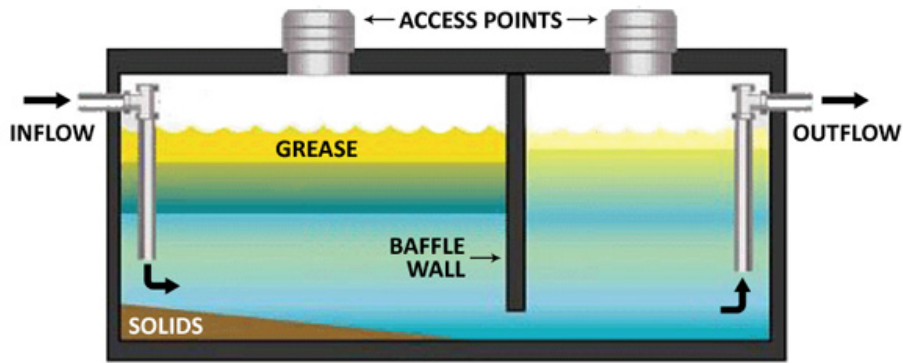


FIGURE 1. Principal of a grease trap (Central Restaurant Products 2013)

The separation effectiveness of a grease trap depends on the fed water temperature and the possible detergents used, as hot water and detergents dissolve fats into the water stream. This is one of the reasons that it is not allowed to discard huge amounts of hot water (over 40 °C in Finland) into the sewer. Before entering the sewer, wastewater should also have a pH between 6.0 and 11. (Vesihuollon yleiset toimitusehdot, 21. 2002.)

Nowadays, the regulations for water and sewer facilities set by the Finnish Ministry of the Environment prescribe the minimal design dimensions of the compartments for sludge, grease and separated water. The sizes are based on the rated flow, which is calculated by multiplying the wastewater flow with given correction coefficients for the inlet temperature (below or above 60°C), grease density and harmfulness of the wastewater to the sewer. Harmfulness is rated high when detergents are used, or when grease traps are installed in establishments with a high hygiene level such as in hospitals. (DI 2007, 56-58.)

The interval for emptying and cleaning of grease traps is determined by the usage frequency, wastewater volumes and the size of the separator. When the grease collection space fills up, the grease interface touches the water outlet level and grease discards with the wastewater flow. Some waste collection companies advise to empty grease traps at least twice a year (Lassila & Tikanoja 2013). The Finnish standard method SFS-EN 1825-2 (2002) recommends to empty and clean even ones or twice a month. Since 2007, new grease traps have to be equipped with a filling alarm (DI 2007, 24), which helps to determine the right time for emptying.

Grease traps are usually emptied completely, as the grease is partly emulsified throughout the water phase and mixed with sediment. In some cases only the surface layer is removed. During cleaning, hot water and high pressure may be used to remove the remaining fat stacked on the walls. After emptying and cleaning, the collected GTW can contain up to 99% water.

2.2 Grease trap waste (GTW)

Grease trap waste (GTW), or trap effluent, is a combination of rotted food solids with partly hydrolyzed cooking or frying oils, fats and greases, as well as detergents and a high amount of water (up to 99%). At room temperature, GTW forms a non-homogenous emulsion, with a strong unpleasant odour caused by acetic and/or butyric fermentation (Garro – Lemieux – Jollez - Cadoret 2007, 5-6). The term “trap grease” technically refers to drained kitchen waste. However, it is sometimes cross-contaminated with septic grease. GTW can be sludge or liquid collected from grease traps installed at meat, fish or other animal origin food preparation and processing sites (coded in the European Union as EWC 02 02 04), edible grease and oil mixtures collected at WTPPs (EWC 19 08 09), or separately collected municipal liquid waste or sludge of edible oils and fats is coded as EWC 20 01 25.

So called *brown grease* is the recoverable organic fraction from GTW, sometimes also mentioned as lipid layer or FOG (fat, oils and grease) fraction. The melting point of brown grease is somewhere between 35°C and 45°C, so it is usually solid at room temperature. It contains a high content of free fatty acids (15 – 100% FFA), sulphur (often 300 - 500 ppm), a high peroxide value (over 30 meq/kg), metals and various other impurities. A high peroxide value implies that the oil or grease is rancid. (Haas 2010; Austic 2010, 3; Blackgold Biofuels 2013.)

The high free fatty acid content stabilizes the emulsion, which makes it hard to separate. Difficult separation is also caused by a viscous interface, a so called rag layer, formed between the oil and water phase. This intermediate region of light mineral solids, water droplets and adhering organic compounds is known to be extremely stable and disruptive to efficient dewatering (Kupai – Harbottle – Xu – Masliyah 2012). The density of the rag layer is less than that of the free water phase and therefore floating on the water surface. In some cases, the rag layer splits and part of it settles to the bottom of the free water layer. Apparently, drainage of oil from the mineral solids can make the floc denser than water. (Hirasaki – Miller - Jiang - Moran - Fleury 2006.)

The total amount of oils and grease (TOG) in GTW can vary much due to different feed-stock and washing operations and the way GTW is collected. According to the study of Austic (2010), a realistic estimation of the share of usable brown grease in GTW would be 2%. This estimation is based on interviews with experienced plant operators and experts in the GTW dewatering field in the US. Tyson, former worker at the National Renewable Energy Laboratory of the U.S. Department of Energy (NREL) suggested that the percent of usable brown grease in GTW ranges from less than 1% to 7% (Austic 2010). This correlates with the results obtained during this research study (0.2% to 7% of TOG). Salonen and Salminen 2003 determined TOG contents of 2.4% to 56%; Haas (2010) reported lipid layers of 0.2% to 58%. Garro et al. (2007) measured 2.8% in their experiments.

The quantity of collected GTW on yearly basis was estimated by monitoring the amounts of received GTW during one year at two waste centres in Central and Northern Finland, which are the official central municipal collection points for the areas. The waste centres are located in Kajaani (Municipal Waste Authority of Kainuu, Ekokymppi) and Oulu (Rusko Waste centre). In 2008, Ekokymppi collected 873 tonnes of GTW from about 83 000 inhabitants. This amount included GTW received from two municipal WWTPs (19.7%) in the Kainuu area. The waste centre in Oulu received in 2009 1285 tonnes from about 284 500 inhabitants. GTW was mainly derived from restaurants and school kitchens (36%) and the food production industry (16%). Other sources were caterings at industrial and retail stores and private properties. In the Oulu area, no GTW was received from the local municipal WWTP, as they mixed trapped grease waste onsite with dewatered sludge for composting. Based on these results, 6.6 kg GTW per capita was annually collected including the share of WWTPs (estimated as 20% of the total) or 5.4 kg per capita without the share of WWTPs.

The quantity of annually collected GTW in this study (6.6 kg GTW per capita) correlates with the estimation from the Urban Waste Grease Resource Assessment (UWGRA) made in 1998 by NREL. They estimated that the averagely collected amount in the US would be 6.1 kg (13.4 lbs) of GTW per capita per year, including GTW from WWTPs. Austic (2010) estimated for the Wake County area US a 10 times higher amount, about 70 litres of hauled trap effluent per person per year. Austics' estimation is based on an average collected amount of 1.8 m³ (480 gallons) of GTW per month per grease trap. At least for Finland, this amount would be an overestimation, as grease traps are rarely emptied monthly. The average emptying interval for the Oulu region was 1 to 2 times per year (3.5 m³ per grease trap), and for the Kainuu area 3 to 4 times per year (3.2

m³ per grease trap). The collected quantities will most-likely increase in the future, as waste and wastewater regulations become stricter and the monitoring improves. This will increase the intervals of emptying and the amount of grease traps installed. Collected amounts can be locally bigger when the GTW collection area includes also industrial wastes from the preparation and processing of meat, fish and other foods as potato chips factories.

Fats consist of a wide group of compounds that are generally soluble in organic solvents and insoluble in water. A fat, chemically known as triglyceride, is formed from one molecule of glycerol and three fatty acids as shown in figure 2.

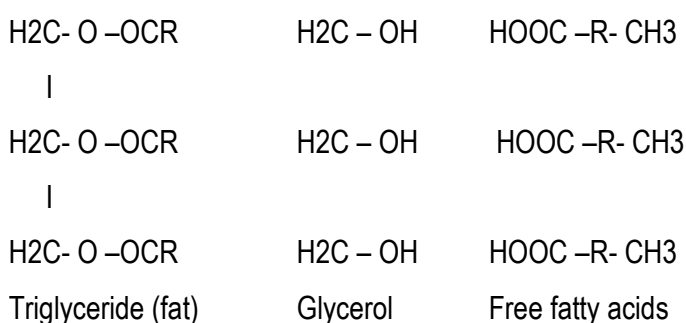


FIGURE 2. Molecular structures of triglyceride, fatty acid and glycerol

When each carbon atom in the chain is saturated with hydrogen, a fat is called "saturated". Saturated fats can stack themselves in a closely packed arrangement, so they can freeze easily and are typically solid at room temperature. Melting points are up to 70 °C. Animal fats contain mainly saturated fatty acids, such as palmitic (C16:0) and stearic acids (C18:0).

Unsaturated fats contain double bonds within the carbon chain, and are oily with melting points well below room temperature. Cooking oil mainly consists of monounsaturated fatty acids (as C18:1), and fish oil polysaturated fatty acids (eg. C18:3, C20:3). As unsaturated fats contain fewer carbon-hydrogen bonds than saturated fats with the same number of carbon atoms, unsaturated fats will yield slightly less energy during metabolism than saturated fats with the same number of carbon atoms.

The fatty acid distribution of GTW was studied in 2010. Samples were taken from a restaurant, a wastewater treatment plant and the food industry. Unsaturated oleic acid (C18:1 cis) and saturated palmitic acid (C16:0) were the most common fatty acids (see table 1). Similar results of

fatty acid composition of brown grease were for example found in a study of Canakci and Van Gerpen (2001), 42.4% and 22.8% respectively.

TABLE 1. Fatty acid composition of GTW from different sources (Eurofins Scientific Finland Oy. Analysis date: 4.2.2010)

C:D *	Common name	Restaurant GTW %	WWTP GTW %	Industrial GTW %	Melting point °C	Boiling point °C
C14:0	myristic acid	3,6	3,8	0,6	59	250
C16:0	palmitic acid	27,3	28,3	6,0	64	352
C16:1 cis	palmitoleic acid	2,4	2,4	12,0	-0,1	210
C18:0	stearic acid	7,4	6,9	4,5	70	383
C18:1 trans	vaccenic acid	2,1	2,1	15,1		
C18:1 cis	oleic acid	36,3	34,2	30,3	13	360
C18:2 cisΩ6	linoleic acid	9,6	9,2	9,6		
C18:3 trans	gamma-linolenic acid	0,4	0,4	10,4		
C18:3 cisΩ3	alpha-linolenic acid	2,5	4,5	2,5	-11	232
	<i>other fatty acids <1%</i>	8,4	8,2	9,0		

* C:D means lipid number, where C is the number of carbon atoms in the fatty acid and D is the number of double bonds in the fatty acid.

2.3 GTW utilisation alternatives

Waste treatment is going through a powerful period of change. In 2011, 22% more waste was incinerated than in the year before, amounting to over ten million tonnes. The amount of recycled waste rose by 18%. In contrast, 19% less waste was placed at landfill sites or long-term deposits than in the year before. (Waste statistics 2011.) The trend for GTW utilisation will be even faster in the near future, due to the tighten legislation for disposal of organic wastes from the year 2016. The following subchapters give an overview of alternatives for GTW utilisation, which may be applied to crude or dewatered GTW.

2.3.1 Utilisation for industrial applications

Saturated fatty acids, such as tallow fatty acids, are used for the production of soaps and lubricants. Unsaturated fatty acids, e.g. tall oil fatty acids (TOFA) utilized from pine oil, are commonly used in the chemical industry for the production of alkyd paints, oil-based varnishes,

adhesives, inks, lubricants, polymers and soaps. Mixtures of fatty acids (FAM) can be used as feedstock for oil mixtures with cold-resistance or anti-oxidant properties.

Fats, oils and greases (FOG) derived from GTW consists of both unsaturated fatty acids (i.e. oleic acid) and saturated acids (i.e. palmitic acid), which somehow limits its reuse potentials. FOG can be used for preparations of esters (i.e. FOG methyl esters) or be sold as a co-fuel for incineration.

Haas (2010) studied the fractionation of the lipid phase of GTW into unsaturated fatty acids (UFA) and saturated fatty acids (SFA) to obtain oleic acid and stearin, respectively. The equipment, energy, chemicals and numerous process steps needed for dewatering and purification make processing expensive. The fluctuating product quality would also be an issue for sales to the fatty acid market.

In October 2013, the contract prices of fractionated fatty acids in Europe were 850-1200 €/t according ICIS pricing as shown in table 2. The market price for FAM depends on its purity, water content and availability. A realistic estimation for FAM with less than 2% of water will be about 300 €/t (Forchem 2013).

TABLE 2. Contract prices of fractionated fatty acids in Europe at 30th October 2013 (ICIS 2013)

		Price Range at 30.10.2013	Price Range 4 weeks earlier
C18 Distilled standard tallow oleic	€/t	1100-1200	1050-1200
C18 Fully hydrogenated tallow stearic	€/t	900-1000	850-950
C18 Triple pressed palm stearic	€/t	850-950	850-950
C18 Standard palm oleic	€/t	1100-1200	1100-1200

The actual amount of valuable fatty acids in GTW (about 2 to 3%) and the relatively small amounts of annually collected GTW in Finland makes the fraction hardly interested for the fatty acid industry. Cross-border collection would be needed, but that would be logistically challenging.

2.3.2 Anaerobic digestion (biogas production)

An anaerobic digester process breaks biodegradable material down to release energy in the form of biogas (50-70% methane), which can be used for the production of heat, electricity or renewable vehicle fuels. It also produces a nutrient-rich matter, digestate that can be used as a soil conditioner.

The addition of fat increases the yield of biogas, as it degrades easily (Amon 1998, 409). Partly dewatered or even crude GTW is therefore an excellent source for biogas production when mixed with other animal and plant-based feeds such as separately collected biowaste, slaughterhouse waste and energy crops. Wang (2012) studied the anaerobic co-digestion of thickened waste activated sludge with grease trap waste. The highest GTW loading rate achieved without digester failure was 20 % (V/V), or 65.5 % (VS). The substantial enhancement in methane yield was likely due to the stepwise increase of co-substrate addition as it provided longer time for microbial acclimation and reduced the inhibitory effect of GTW. (Wang 2012.)

Anaerobic digesters with an operation temperature of 30 to 45 °C are referred to as mesophilic systems. So called thermophilic systems are operating at 50 to 60 °C. Due to lower temperatures, the mesophilic process consumes less energy. Mesophilic systems are considered to be more stable than thermophilic digestion systems. An explanation is that at higher temperatures and pH values, the concentration of free ammonia in the reactor increases, which may inhibit bacterial activity and thus reduces biogas production. Benefits of thermophilic digestion systems are a higher methane production in a shorter reaction time, due to increased temperatures. This allows a higher organic load, as well as a smaller reactor size compared to the mesophilic process. Operation at higher temperatures also facilitates hygienisation of the end digestate, which meets the regulations in the European Union (EY 1774/2002). In mesophilic processes hygienisation has to be done before or afterwards, by heating chopped feedstock of less than 12 mm particle size at minimal 70°C for at least one hour. (Jääskeläinen – Juovinen 2010.)

The water content of the feed material should be at least 50% in order to cleave long chain molecules by hydrolysis and acid forming bacteria. The optimal water content for a mesophilic digestion process is usually over 90%. A thermophilic process has an optimal water content of

70%. (Lampinen 2004.) The need for high water content makes crude or partly dewatered GTW suitable for biogas production.

Anaerobic digestion has in recent years received increased attention among governments in a number of European countries, among these the United Kingdom, Germany and Denmark. Biogas production is common practice in Central Europe, but in a wide-spread country as Finland biogas installations are not yet available at all locations, especially in North of Finland. In 2011, 37 biogas installations were in operation and 33 plants planned or under construction (Huttunen - Kuittinen 2012) as shown in figure 3. Most of the plants are mesophilic processes.

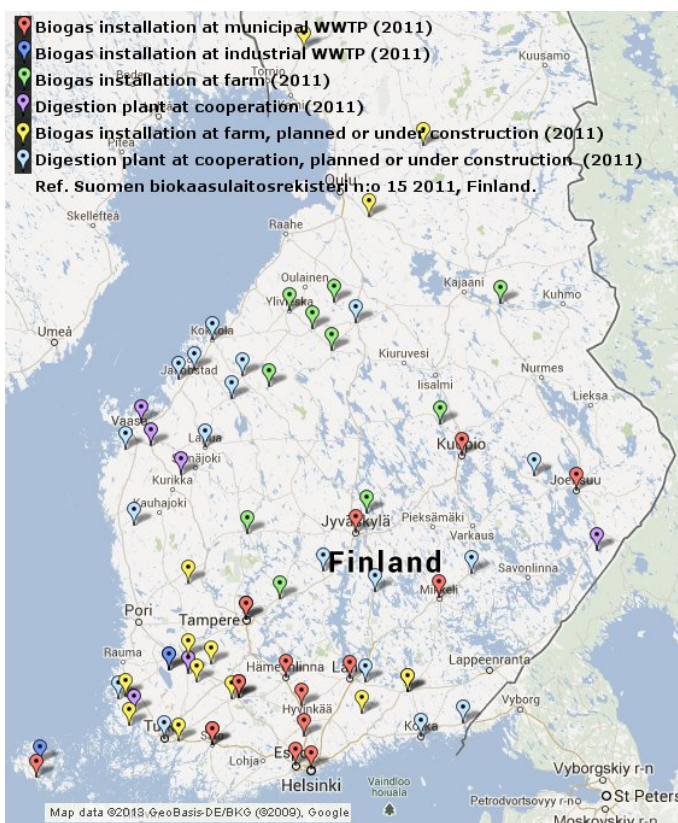


FIGURE 3. Installed and planned biogas installations in Finland in 2011 (Huttunen - Kuittinen 2012)

The first biogas installation in North Finland will be installed at the waste centre of Oulu. The start up will be in the end of 2014. The mesophilic anaerobic digester will produce 15 000 MWh of energy of 20 000 tonnes of organic biowaste on yearly basis. Oulu Waste centre will contribute 10 000 to 14 000 of biowaste; the rest will be WWTP sludge as well as collected GTW. (Oulu waste centre 2013.) Also stored collected GTW in geotubes (§2.3.9) will most likely be used as a feedstock. At the same time, a private company (Viherengas Järvenpää Oy) is planning an even

bigger biogas installation in the same area. If the plant will be built, it will increase the competitiveness in the area, lowering disposal cost prices.

2.3.3 Composting

A fairly simple way for GTW utilisation would be windrow or reactor composting after impregnation of GTW into an organic support material. Disadvantages of composting are foul odour caused by poor hygienisation, restrictions for use as a fertiliser, significant losses of nutrients, a high energy consumption needed for aeration and mixing, and wasting of the materials energy content. (Lampinen 2004.) In case of windrow composting, also cold weather conditions restrict biological composting processes.

Despite these disadvantages, in the Wake County US significant quantities of trap effluent are mixed with other solid compostable waste (like wood chips) to create a saleable compost product which is sold primarily for landscaping purposes. (Austic 2010.) In Finland, GTW is often mixed with municipal WTP sludge prior composting.

2.3.4 End-of-Waste status

An interesting sight of view in the EU is the so called End of Waste (EoW) status stated in the Waste Framework Directive (2008/98/EC). The EoW status is given to waste that has been processed to meet specific quality criteria and can be classified as a marketable product or a secondary raw material. Currently, scrap metal and glass are the only two materials that have been completed and have regulations. The European Commission is currently studying inclusion of biowaste. (Waste Framework Directive 2012.) If biowaste gets an End of Waste status in the future, this could make highly biodegradable GTW valuable after anaerobic digestion or composting.

2.3.5 Incineration

Incineration with energy recovery (through heat) is a waste-to-energy (WtE) technology such as gasification, pyrolysis and anaerobic digestion. Incineration requires oxygen and high temperatures (about 850 °C or higher). Incinerators reduce the mass of the original solid waste by

80–85%, depending on feedstock composition and the degree of recovery of materials such as metals from the ash for recycling.

The fuel value of crude GTW is low, because of its high water content. Therefore, at least part of the water should be removed beforehand. Like recovered fuels (REF), thermal utilisation of partly dewatered GTW can be conducted in fluidized bed combustion processes alongside a conventional fuel with more calorific fuel. This can be coal, plant biomass or segregated municipal waste. In 2011, eight power plants in Finland used REF as part of the energy production (Finnish Solid Waste Association 2011). Waste incineration deals with strict environmental regulations as the Incineration Directive 2000/76/EY and VNa 151/2013. For example high sulphur, sodium or potassium contents may restrict straight burning. (Forsell, 2011.)

2.3.6 Pyrolysis

Pyrolysis is the decomposition of organic material in the absence of oxygen at typically of 450 to 550 °C. The process, originally used for the production of charcoal, has been adopted for feedstock as fuel wood, timber waste, packaging materials, food industry by-products, field biomasses and different manures. Process products are coal, tar, distillates (so called bio oil or pyrolysis oi) and gases as methane, hydrogen, carbon monoxide and carbon dioxide. (Kujala 2012.) During so called *slow pyrolysis* the increase in temperature lasts from minutes to a few hours, while in *fast* or *flash pyrolysis* heating happens in less than 2 seconds. The benefit of slow heating is that it increases the carbon content in the distillate, which makes the bio oil fraction more valuable. The PAHs formed in the pyrolysis process are enriched in tars and gases, which has to be considered in their handling and utilization (VTT 2012).

The tar-free distillate including acetic acid and furfural was found to be a promising and marketable product for various purposes, involving pesticides, biocides, repellents, wood preservatives and metal coatings (VTT 2012; Kujala 2012.) Bio oil contains valuable bio-chemicals that can be used as food additives or pharmaceuticals. Bio oil has been successfully tested in engines, turbines and boilers, and been upgraded to high quality hydrocarbon fuels although at a presently unacceptable energetic and financial cost (US Department of Energy 2005.) The greatest challenges in the use of pyrolysis oil as fuel are a lower heating value, greater acidity and higher solid content compared to fossil fuel oils (Starck 2011).

Starck (2011) studied the commercial profitability of a bio oil production facility in Savonlinna region in Finland. The results of the calculations indicate that bigger 400 BDMTPD (Bone Dry Metric Ton per Day) facilities were profitable as long as the cost of biomass is reasonable and the gross investment remain fair. Smaller facilities were not profitable investments.

Produced bio-coal can be burned like charcoal, but may for example also be used as a soil conditioner to improve crop yields as it improves the soil texture, increasing its ability to retain fertilizers and release them slowly. Gas can be burned to drive turbines or steam generators to produce electricity and steam. (VTT 2012; Kujala 2012.)

Pyrolysis has also been applied to the decomposition of organic material in the presence of superheated water or steam (hydrous pyrolysis), for example, in the steam cracking of oil. The Gas Technology Institute (GTI) has developed a catalytic process called *Integrated Hydrolysis and Hydroconversion* (IH2) that turns biomass feedstock, ranging from wood to algae, directly into high quality hydrocarbon gasoline and diesel blending components. Biomass is converted into carbon oxides, water and charcoal in the presence of hydrogen in a fluid-bed hydrolysis stage, under high pressure (14–35 bar) and intense heat (300–700 °C). The char is removed by a cyclone, and the vapour from this stage is directed to a second stage hydroconversion unit which further removes oxygen and produces gasoline and diesel products with less than 1% oxygen. The liquid is condensed and the gas from the process is sent to an integrated steam reformer. The char can be used as a renewable boiler fuel and burned to make steam or electricity. The process is still under development and additional work is needed to commercialize the IH2 technology. (Marker – Roberts – Linck – Felix - Ortiz-Toral – Wangerow – McLeod - Del Paggio – Gephart – Starr – Hahn 2013.) As the process uses water, GTW could be used as a possible feedstock for this technique.

Biomass gasification is a pyrolysis process that uses high pressure and high temperatures (1000 °C) to convert organic material into a synthetic gas of carbon monoxide, hydrogen, carbon dioxide, and small contents of methane. This so called syngas can be burned directly or used as a starting point to manufacture fertilizers, pure hydrogen, methane or liquid transportation fuels. The use of syngas is subject to strict limits. The costs for purifying syngas can be high, up to 50% of the total production costs (Lassi – Wikman 2011, 71). In 2013, a 140 MW biomass gasification plant was started up at Vaskiluodon Voima Oy in Vaasa (Finland). The bio-gasification plant was constructed as part of the existing coal-fired power plant, and the produced gas will be

combusted along with coal in the existing coal boiler. (World's largest biomass gasification plant inaugurated in Vaasa. 2013.) Affordable Bio Feedstock US is planning to generate electricity from dewatered GTW and from the separated solids, either through gasification or as boiler fuel for a steam generator (Austic 2010).

2.3.7 Boiler fuel applications

Processed GTW (brown grease) is successfully used in the US as a fuel for steam boiler applications. The product has a BTU value (heating value) of 70 000 BTUs per pound, which is just about 2 000 BTUs underneath a diesel product. The high heating value makes the oil suitable to power heat boilers. Affordable Bio Feedstock US uses brown grease to power the boiler that creates the heat to extract brown grease from GTW. Moreover the extracted oil is used to warm four other buildings, saving the firm \$30 000 annually in heating costs. (Mekeel 2009.)

Boiler applications require hardly pre-treatment of the feedstock, and are able to deal with high contents of moisture, soap, insoluble and unsaponifiable substances, and other contaminants. The boiler modifications required to convert a typical diesel or multi-fuel boiler are very similar to those performed in order to run fatty acid esters (biodiesel). The fuel holding tank and lines to the boiler should be heated and the fuel should be recirculated to prevent settling of the material. Any copper and brass which can get in contact with the fuel should be replaced, as both will degrade in the presence of vegetable oil. Furthermore, the wetted parts of the fuel pump should be checked for their compatibility with both vegetable oil and acidic environments. The oil should be preheated to at least 77 °C (170 °F) before passing through the spray nozzle. The spray nozzle may need to be changed, as ash can build up around the nozzle. (Austic 2010.)

2.3.8 Biodiesel production

Biodiesel has attracted considerable interest as an alternative fuel for combustion in compression-ignition (diesel) engines (Moser 2009). In Central Europe, the use of animal fats as raw material for biodiesel has increased. One significant environmental advantage of animal fat biodiesel is that it generally has lower nitrogen oxide (NO_x) emissions than biodiesel made of other sources (Mäihäniemi 2008).

The chemical composition of biodiesel is dependent upon the feedstock from which it is produced. GTW is a variable mixture of vegetable oils and animal fats of differing origin and dissimilar fatty acid compositions (Moser 2009), and so will the produced biodiesel quality be inconsistent. Biodiesel prepared of feedstock containing animal fats has poor low-temperature properties, compared with biodiesel produced from vegetable oils with lower melting points, such as soybean or canola. Biodiesel of GTW could therefore be considered in warm areas, but for Finnish cold weather conditions it is less useful.

A primary problem with the use of GTW for biodiesel production is its high sulphur content (300 – 500 ppm). Reducing sulphur is very difficult, only a few companies have developed processes to achieve this. Chakrabarti found that distillation plus activated carbon was sufficient to break the 15 ppm threshold required for on-road fuel sale in the US. The process is expensive and probably not feasible on a commercial scale. (Austic 2010.)

In addition, since only the FOG component of GTW is refinable for biodiesel production, converting FOG waste into biodiesel leaves the remaining waste disposal of food residuals and wastewater a challenge (Wang 2012). Processing also comes with the generation of liquid and solid waste by-products as new waste streams.

Biodiesel prepared of dewatered GTW has a very high gel point, and requires extensive physical filtration. In addition, it often fails the distillation temperature and carbon residue tests which must be passed to sell biodiesel as an on-road fuel. (Austic 2010.) Biodiesel production of GTW is further on challenging, due to its high content of free fatty acids (15-100% FFA), solidification at room temperature and water contamination. With conventional technology, so called alkali-catalyzed transesterification, high levels of FFAs require multiple costly processes and real-time system adjustments. FFAs namely react with the alkali catalyst to form soap and water. When the FFA level is above 3%, the soap inhibits separation of the glycerol from the methyl esters and contributes to emulsion formation during the water wash. (Van Gerpen 2005; Moser 2009.)

During the last decade, different technologies have been developed to convert low-quality plant and animal based FOG to biodiesel. Pretreatment processes using strong acid catalysts have been shown to provide good conversion yields and high quality final products (Van Gerpen 2005) without soap formations. Despite that, the corrosive nature of acid, slow reaction rate and higher temperature conditions limit the use of the technology for esterification reactions. Other potential

strategies for the production of biodiesel from feedstocks with high FFA content include feedstock purification such as refining, bleaching, and deodorization to remove FFA content and other undesirable materials.

Enzymatic transesterification by lipase seems a feasible method for biodiesel production. Enzymes do not form soaps and can esterify both FFA and triglycerides in one step without the need for subsequent washing step. Enzymes have shown good tolerance for the FFA level of the feedstock but the enzymes are expensive and unable to provide the degree of reaction completion required to meet the ASTM fuel specification (Van Gerpen 2005).

Proved commercial technologies are still few and their feasibility is uncertain. A US company BlackGold Biofuels (2013) claims that it can handle high concentrations and highly variable amounts of FFAs in a single process, without adjustments, without producing soap. They have developed fuel purification technologies, including desulfurization, and are currently looking to commercialize their process. However, it is very capital intensive, and probably does not make sense for the relatively small quantities (Austic 2010). The method developed by Pacific Biodiesel Technologies (2013) allows use of up to 50% FFA feedstock without loss of yield. Another example is RPM Sustainable Technologies (2013) which is using a proprietary acid catalyzed esterification pre-processor in conjunction with a base catalyzed trans-esterification, to produce ASTM/EN quality biodiesel fuel from dewatered brown grease. The company projects that an estimated \$1 million investment for the equipment could pay for itself in three years, if it handles 750 m³ of brown grease per year. (Dowling 2012.) Examples of patents for biodiesel production of GTW or other high FFA feedstock are US2004/0254387, US2007/0232817, WO2004/048311, US2007/0033863 and US2007/0277429.

2.3.9 Dewatering and storage in a geotube

The waste centres of Oulu, Ylivieskä and Lahti (Finland) are using a container of geotextile (TenCate Geotube®), to (temporary) store and simultaneously dewater collected GTW. Before pumping GTW into the tube, polymers are added to bind fats and solids and separate the water. Effluent water drains through the small pores in the textile into the sewer. After the final cycle of filling and dewatering, the sludge continues to densify due to desiccation as residual water vapour escapes through the fabric. Disadvantages of this technique for GTW are poor dewatering (40-

50% water remains) and that the grease is still mixed with the solids. Because of the high water content, landfill disposal of the content is not allowed, and waste incineration would be uneconomically. At the moment, full geotubes remain yet on-site without waste reuse.

2.4 Available separation technologies

This subchapter contains a summary of available techniques, including heating, settling, centrifugation, flotation, as well as chemical (acid, base, solvents, demulsifiers) and enzymatic treatments, which are used to separate free water and FOG from GTW.

2.4.1 Mechanical separation techniques

US patent 7,161,017 B2 (2007) consists of a method to separate the waste into three layers (floating, liquid and sludge). The first step is settling the GTW at room temperature. The typical mass balance is a floating top layer of 8 % (w/w), a middle liquid layer of 58 % (w/w) and 34 % (w/w) of bottom sludge (figure 4).

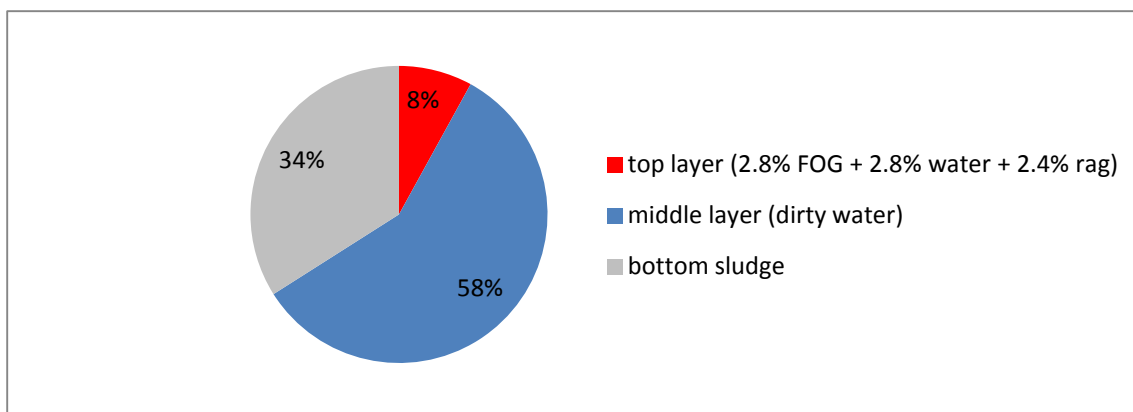


FIGURE 4. Typical mass balance of GTW after settling at room temperature (US7161017B2 2007)

The bottom sludge is split into two phases by centrifugation, to remove 2/3 of the water off (Garro, et al. 2007). Centrifugation is a process that involves the use of the centrifugal force to speed up the sedimentation of mixtures. The process is used to separate components with different densities (e.g. oil, water and sediments) or two immiscible liquids (e.g. water and oil). The rate of centrifugation is specified by the angular velocity measured in revolutions per minute (RPM), or acceleration expressed as times gravity (x g). The conversion factor between RPM and x g

depends on the radius of the sample in the centrifuge rotor. The higher the centrifugal speed, the better the separation. In an experiment of Saadatmand, Yarranton and Moran (2009) about rag layers in oil sand froths it was found that the settling process was essentially over at a centrifugal speed of 3000 rpm meaning 1000 times gravity (figure 5).

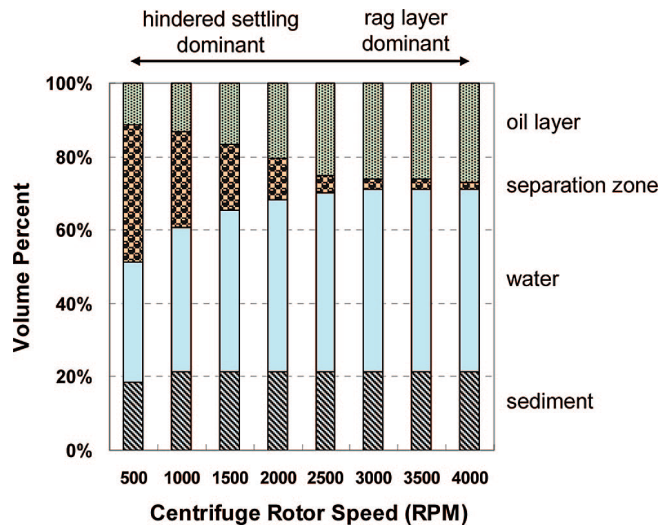


FIGURE 5. The gradual change of the separation zone (rag layer) at different centrifugal speeds (Saadatmand, et. al. 2009, 8830.)

The middle layer as well as the removed water from the bottom sludge (in total about 80% of the starting material) is treated in a dissolved air flotation (DAF) unit before being disposed in the municipal sewage network (Garro, et al. 2007).

The top layer (8 %) is heated (60 to 95 °C) and filtered with a rotary screen filter to remove particles greater than 500 μm, followed by a three-phase centrifugation (Garro, et al. 2007). Other patents use similar filtration technologies at 45 to 65 °C with a filter size of 254 μm (US2007/0277429 2007) or 35 to 52 °C with a filter size of 150 μm, followed by removal of smaller solids (>40-50 μm) by liquid-solid separation and a decanter centrifuge (US2007/0033863 2007). Water can be removed from the top layer by evaporation, e.g. at 65-90 °C. In this temperature range glycerol is less viscous, but still stable (Biodiesel-Glycerol Evaporation and Refining. 2012).

A general mass balance after filtration and centrifugation of the top layer is 35 %(w/w) of FOG at the top, 35 %(w/w) of rag (oil, water and light solids) and 30% of bottom sludge (water and heavier solids). The rag-layer and bottom sludge are disposed.

The FOG layer (2.8% of the starting material) is essentially free of water and is constituted of a mixture of free fatty acids, tri, di, and monoglycerides, trimer and dimer acids, oxidized monomers (light fraction), unsaponifiables and other coloured long chain oxidized products (poly-glycerides, polymers and their products of oxidative decomposition). The FOG can be used for preparations of esters (i.e. FOG methyl esters) or it can be further fractionated into unsaturated fatty acids (UFA) and saturated fatty acids (SFA) to obtain oleic acid and stearin, respectively. (US2007/ 7161017B2 2007). (Garro, et al. 2007.)

BWI Equipments Inc. (<http://www.bwiequipment.net/>) has developed a proprietary filtration/gravity separation system that uses steam to pre-heat GTW as it comes off the truck. The process has an initial screening to remove larger particulates. The material is transferred to a preheated settling tank where the brown grease is removed at the top. The remaining waste water is decanted off at the bottom. The process can treat up to 189 m³ (50 000 gallons) of GTW in an 8-hour shift. Affordable Bio Feedstock (US) combined the separation unit with a water treatment unit to reduce the BOD of the discharged water fraction, which can be 5 000 – 7 000 ppm. The complete system sold by ABF would cost \$1.2 million for a 5 day a week plant, running 150 m³/d (40 000 gallons), or \$1.6 million for 380 m³/d (100 000 gallons). (Austic 2010.)

In 2008, the system of BWI was installed at Kline's Services in PA US. The company gathers waste from grease traps and deep fryers at restaurants, supermarkets and food-production plants, as well as from municipal WWTPs, where much kitchen waste ends up. Another plant was installed in Fort Lauderdale (FL, US). The process reduced the amount of FOG influent into the WWTP, which was driving the costs down for companies that collect GTW for disposal. This again reduced the amount of illegal discharges into the ocean and sewer locations.

The plant discharged the separated water fraction (79% of GTW) directly into the sewer at a discharge cost of \$0.005/gal (1.3 \$/m³). Disposal of the particulates (5.7% of GTW) added an additional \$0.005/gal to the processing costs. To compare, discharge of unprocessed GTW to the same WWTP would have cost \$0.10/gal (26 \$/m³), so a saving of 95% on GTW disposal costs. (Austic 2010.) To compare, in 2013 the disposal costs for special waste in Oulu (Finland) were 74 €/t (excl. ALV) and the costs for wastewater disposal to the sewer about 1.5 €/m³. The separated brown grease (12% of GTW) at \$0.10/lb (220 \$/t) was sold for boiler fuel applications (Austic 2010).

2.4.2 Acid hydrolysis

Most fat splitting technologies hydrolyze fats at temperatures between 100 °C and 260°C, with or without pressure and use of catalysts. Used catalysts are alkyl-aryl acid or cyclo-aliphatic sulphonic acid with sulphuric acid (0.75-1.25 %(w/w)) or other mineral acids. The higher the temperature or pressure the shorter the reaction time. Under catalytic conditions, atmospheric pressure and moderate heating (100 – 105°C) a reaction time of 12 to 48 hours is sufficient (Twitchell process). The batch autoclave operation uses injection live steam to hydrolyze fat. Due to venting the desired agitation and operating pressure is maintained. (Garro, et al. 2007.)

After settling a formation of an aqueous and a fatty acid phase appear. The fatty acid phase is treated with mineral acid, where after it is washed with water to remove traces of the mineral acid. The fatty acid phase is reacted under catalytic conditions for a period of 5 to 19 hours at 150-175°C, or 2 to 4 hours at 240°C without a catalyst for similar yields. The Colgate-Emery process uses also high temperature (250-260°C) and pressure (50 bars) with a reaction time of 2 to 3 hours. (Garro, et al. 2007.)

Difficulties for fat hydrolyzing processes are high labour costs, for moderate conditions the long reaction time, and in some cases the catalyst handling and high equipment costs. The Colgate-Emery process is also restricted as it needs a relative clean start up material. (Garro, et al. 2007.)

2.4.3 Use of organic solvents

Another separation method (US2008/7338602) is to remove organics from dewatered GTW by mixing with a solvent, followed by mechanical separation of the FOG fraction. Solids are washed and dried to remove traces of FOGs and water. The inert solids can be safely disposed according EPA regulations. The solvent is separated from the FOG and reused in the same process. The recuperated grease fractions are pure and homogenous, constitute an excellent raw material for further oleo-chemical processes and applications. The patent does not specify the solvent used, neither yield percentages nor examples.

2.4.4 Enzymatic hydrolysis

Lipase is an enzyme that catalyzes the hydrolysis of fats. Enzymatic operations by lipase from *Candida rugosa*, *Aspergillus niger*, and *Rhizopus arrhizus*, has been studied at temperatures of 26 to 46 °C, for periods of 48 to 72 hours. The inconvenient of this process is that it works well for specific substrates under specific conditions, but in case of GTW the method is less selective. (Garro, et al. 2007.) The impact of an enzymatic treatment may show significant effect in batch reactors, but probably has no considerable effect on long time use in continuous reactors. In this case, enzyme regeneration is needed. (Izah - Ohimain 2013.) Also long reaction times and great volumes required to obtain the optimal concentrate are also current problems involved in this kind of procedure. (Garro, et al. 2007.)

2.4.5 Saponification by alkali addition

A chemical method to optimize the separation of GTW would be to first break the ester bonds under alkaline conditions prior acid hydrolysis. The reaction known as saponification converts fats first into soap. Saponification is a process by which triglycerides (fats) are reacted with sodium or potassium hydroxide to produce glycerol and a fatty acid (figure 6).

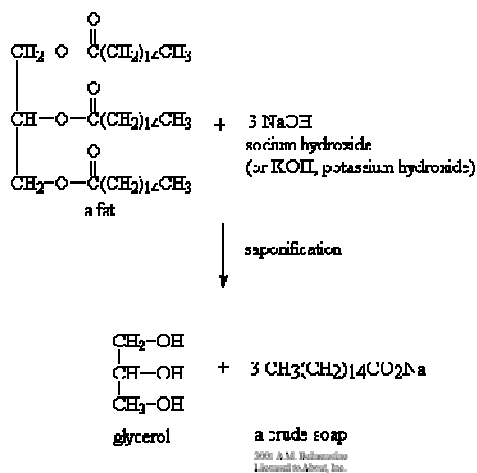


FIGURE 6. The chemical reaction of saponification (Helmenstine 2001)

Next, acid hydrolysis dissociates formed fatty acid soap (sodium salts of free fatty acids) into fatty acids and sodium. The method is also used for the separation of tall oil (obtained from pinewood) from tall oil soap. When tall oil soap is acidulated, fatty acid soaps and the alkali resinates are converted into their acidic forms, namely free fatty acids, resin acids and inorganic salts. The

acidulation step is carried out in various types of reactor vessels at elevated temperatures, where the tall oil soap is combined under intensive agitation with a concentrated acid which reduces the pH to about 3. Thereafter, the obtained mixture is allowed to separate into an oil phase (tall oil) and a brine aqueous solution, typically in a High Density Separator (HDS) unit. The separation of the oil and aqueous phases is often hindered by the presence of a so called rag layer as explained in §2.2. (Stigsson - Naydenov 2011.)

2.4.6 De-emulsifying

Hydrophobic solids, glycerol and a high ion concentration (e.g. sodium) in the water phase of GTW produces an emulsion which is difficult to separate. Destabilizing or breaking an emulsion is the process in which an emulsion is separated into its component phases. Demulsification mechanisms for breaking oil/water emulsions include aggregation/flocculation, sedimentation, and coalescence. (Urrutia 2006, 12.)

The factors favouring emulsion breakdown are temperature, time, the addition of demulsifiers, and reducing shear or agitation. Increasing temperature decreases emulsion viscosity and increases the Brownian motion of droplets less than 2 μm in diameter and hence accelerates the rate of particle collisions. Increasing residence time allows the different emulsion breakdown mechanisms to take place. Adding demulsifiers promotes flocculation or replaces the stabilizing film at the interface with a weak film. Reducing shear or agitation during emulsification contributes to an increase in droplet size and as a consequence to an increase in the frequency of collisions, aggregation, settling and coalescence. (Urrutia 2006, 16.) A macroporous resin could be a good choice to remove sodium ions from glycerol/water solutions with a high salt concentration (Society of Chemical Industry 2009).

3 TEST METHODS

This chapter describes the test methods used during the research study. The first subchapter describes the experimental test methods used during pretesting. The next subchapter describes how the pilot plant study was made. In the third chapter restrictions for the pilot plant study are listed. The last chapter describes how the calculations for the financial feasibility analysis were made.

3.1 Test arrangements for pretesting

Pretesting started with small samples (50 ml – 1 litre) of GTW. Heating was done by placing the measuring cylinder or beaker with sample material in a temperature controlled water-bath. Fractionation by settling only (1 x g) was tested by settling the mass in one litre measure cylinders and obtaining the volumes of different fractions during specific time intervals. Fractionation with an industrial decanter centrifuge was simulated by centrifuging at 1000 xg (3000 rpm) for 1 minute.

The laboratory testing was scaled up to a 30 litre batch installation (used in §4.2.2) made of PE (tube height 1135 mm, diameter 190 mm). Water and bottom sludge could be removed from the bottom. Heating happened by circulating hot water (up to 80°C) through a spiral copper tube placed inside the tube. Due to the transparent material of the column, separation could be easily visually obtained.

The batch test was ones more scaled up to a 5 m³ installation. The process was operated manually, and with the same principal as the smaller batch plant. The installation was tested onsite at a waste centre in Kajaani. Practical knowledge obtained was used for the design of the actual pilot plant.

Water and dry solids contents were observed by centrifuging the sample (about 40 ml) at maximal speed with a bench laboratory centrifuge (6000 rpm, 10 min) to separate the emulsion into an organic top layer, free water and bottom sludge. The fractions were separately weighed (\pm 0.1 mg) and dry solid contents were analysed by drying the fractions at 105 °C.

The pH was measured with a laboratory pH meter and glass electrodes, daily calibrated at pH 4, 7 and 10.

A solution of concentrated sulphuric acid in deionised water (1:1) was used for acid hydrolysis. Sodium hydroxide was used for saponification reactions.

3.2 Test arrangement for the pilot study

The pilot plant was built onsite at a waste collection centre. Needed equipments, chemicals, space and resources were financed by the customer according a pre-agreed budget plan. The customer took also care of delivering the test batches of GTW and removing of the separated fractions. Planning and installation of process' automation was outsourced, as well as all plumbing and electricity works. The pilot plant was managed by a technical engineer, working in close cooperation with a chemical researcher, who took of the development of the chemical process and samples analysis.

The pilot process was controlled by continues measurements of the pH, temperature, electrical conductivity, and surface levels detection (by pressure) in the tanks. Electrical conductivity electrodes and a dielectric rod were installed to distinguish phase changes between water and grease. Continues measurements were used for automatic operation processes and control, alarms are given when a measure exceeds the norm. Results were stored by a data-logger. Data-logging included also data about time, pump rates for chemical addition (sodium hydroxide and sulphuric acid) and feed pumps, and the working of automatic valves.

Laboratory samples were taken from the GTW as received, from the separated water phase before disposal to the sewer (effluent), and from the separated top layer. From each batch parameters as pH, temperature, total oil and grease (TOG) and water content were analysed. TOG was measured by WILKS InfraCal IR and the water content visually after centrifugation (6000 rpm, 10 min). Ammonium (photometrical by Macherey-Nagel Visocolor) was measured to check whether the GTW was contaminated with septic tank sludge. Environmental limits were set for the discard water prior disposal to the sewer. The pH of the effluent should be over 6 and the TOG less than 50 ppm.

Truck drivers were able to empty their collected GTW straight into the receiving tank without additional supervision. If the collection tank was full, a magnetic valve would automatically close the intake line and a red sign was shown to the truck driver. A technical operator was needed to initiate the dewatering process. Figure 7 shows the operation diagram of the pilot plant.

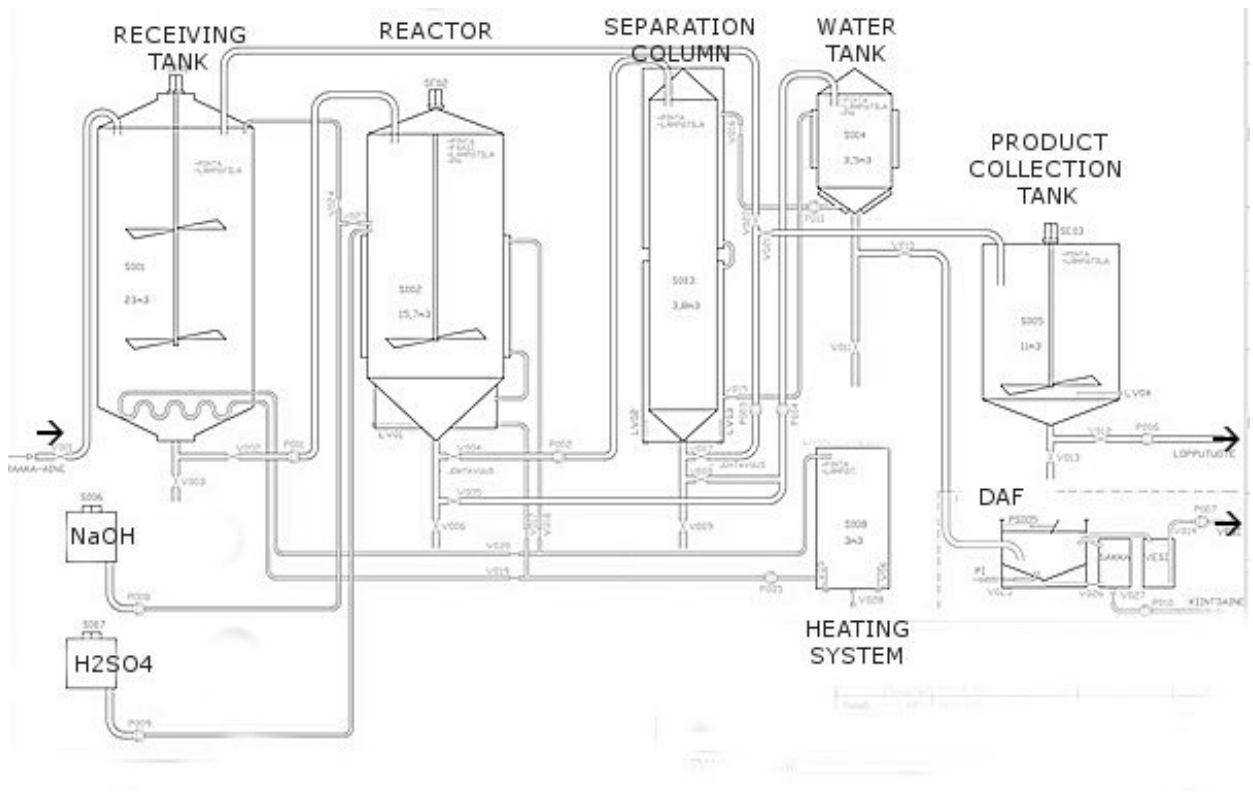


FIGURE 7. Operation diagram of the pilot plant

Collected GTW (3 - 7 m³/batch, pH 3 - 5) enters the 23 m³ receiving tank. The waste is heated up to 40°C, to speed up water separation. The waste is settled overnight, where after the first fraction of separated water and bottom sludge (50 – 70 %) is pumped into the reactor of 15 m³. The level of the grease-water interface should have been automatically controlled by measuring change in electrical conductivity. Due to malfunctioning of the measurement system (fouling or wrong scaling), controlling was done visually by taking samples to obtain the border of the water phase and the floating greasy layer.

The raw material is heated to 60 -75°C, with an optimum of 65 -70°C. The pH of the dirty water phase in the reactor is lowered below 3 by sulphuric acid, to improve the separation. Acid separates emulsified grease from the water and settles heavy solids to the bottom. After settling of 2 to 4 hours, bottom solids and free water are pumped in batches of 3 m³ into the water tank

(3.5 m³) and from there to the dissolved air flotation (DAF) tank. The grease-water interface was obtained visually by taking samples. In the future this will be done by measuring change in electrical conductivity.

In the first compartment of the DAF unit the pH is raised to 8 – 10 by a solution of sodium hydroxide (NaOH). NaOH reacts with leftover fats in the water phase to form soap. Due to the addition of air bubbles foam appears. The foam is scraped from the water surface and collected into the sludge tank of the DAF unit, which contains a small amount of acid to break the foam. Water overflowing to the clean water tank where remaining solids settle to the bottom. After settling the water is clean enough to discharge into the sewer (TOG <50 mg/kg, pH >6).

The upper fraction which remained in the reactor (brown grease) is pumped to the separation column (3.8 m³). During pilot testing it was found that the volume of the dewatered mass was too small to be treated in the reactor as planned, for the reason that the grease content (TOG) of the collected GTW was about 10 times smaller than expected (0.2 - 0.5%). Dewatered fractions from different batches were therefore collected to be treated off-side.

A small preliminary reactor was built to test additional fractionation of the dewatered layer. The purpose was to separate valuable fatty acids (FAM) from the rag layer, the organic interface of light solids, microbial mass and water. This was done by a process of soapification, acidification and mechanical separation at elevated temperatures. The material was moderately heated (40 °C) and NaOH was added to change the fats (triglycerides) into soap, where after the mixture was neutralized with sulphuric acid to a pH of 3 - 4 to dissociate soap into fatty acids and glycerol. After additional heating (70 °C) and settling the reactor was emptied in fractions of bottom sludge, brown water (containing organics as glycerol), a rag layer of hydrophobic light solids and finally the remaining mixture of saturated and unsaturated fatty acids.

3.3 Restrictions during the pilot study

Test runs could be made only during the late spring until the end of October, because the pilot plant was built in a cold hall. Water lines would freeze below 0 °C and sodium hydroxide solution solidifies already below 10 °C. Isolation of the space, by building a wall inside the hall was too expensive during the pilot testing. For future use though this will be anyhow crucial. Only in this way the installation can be used through the whole year.

The test hall did not contain floor drains and was not connected to the sewer as planned. Effluent water had to be collected in separated tanks and emptied manually. For this reason the test had to be made batch-like and continuous operation (water removal and treatment before disposal to the sewer) could not be tested during the pilot study.

The pilot testing period included the summer holiday period, which practically meant that the installation was out of use for almost 2 months because of a lack of human resources.

The literature study to annually collected GTW amounts was limited to the Kainuu and Oulu regions (Finland) and may not reflect the total annual amount collected in Finland.

3.4 Financial feasibility analysis

The final part of this study addresses the financial feasibility of the business idea. A sales forecast was prepared for the case that a private company in the waste collection business would buy the dewatering installation under study.

By fluctuating the dewatering efficiency of the installation and the economic value for the dewatered organic fraction (negative in case of disposal or positive in case of sales), different profit and loss accounts were prepared. A profit and loss account is a financial statement that shows the net profit or loss incurred over a specific accounting period, typically over a fiscal quarter or like in this case a year.

Operating margins (1) were calculated from the created profit and loss accounts to measure the profitability ratio.

$$\text{Operating margin (\%)} = \text{operating income} / \text{sales revenue} \times 100 \quad (1)$$

Operating income (2) is the difference between operating revenues and operating expenses (variable and fixed expenses), excluding income tax expenses, dividends to shareholders, and interest on debt.

$$\text{Operating income} = \text{revenue} - \text{operating expenses} \quad (2)$$

The profitability of the process design was estimated by looking at the change in operating margin over time (in this case 10 years). The operating margin gives an idea of how much profit (before interest and taxes) the dewatering plant makes on each euro of sales. By comparing these figures to each other and to other competitive solutions, such as GTW collection and transshipment for disposal only, the quality of the process design can be determined. If the margin is increasing, it is earning more per euro of sales. A good operating margin is needed to be able to pay for costs such as loan interest and taxes. A higher operating margin means that the company has less financial risk.

4 RESULTS

This chapter describes the main results obtained during development process, starting from step-wise pre-testing and up scaling in the laboratory up to the testing of the dewatering by the pilot plant and further fractionation of the dewatered mass for FOG recovery. At the end of the study a financial feasibility analysis was made and the project management was evaluated.

4.1 Pretesting on laboratory scale

This subchapter contains a summary of the laboratory testing part of the research study. It describes combinations of different methods to separate GTW by heating, settling, acid hydrolysis and saponification.

4.1.1 Heating and settling

Laboratory pretesting started in the March 2008. The first samples of collected GTW were taken at the waste centre in Oulu from two different batches. Samples were emulsions with a water content of 93 and 92 %, a total oil and grease content (TOG) of 7% and dry solids (0.2 and 1%), respectively.

The separation of water, grease and sediments was first obtained by settling only at room temperature. Up to 40% of free water separated for the sample with the lowest dry solid content (figure 8 at 20°C). The sample was heated and settled at 60°C and 75 °C. After a settling time of half an hour, the separation efficiency was 60 % for both cases (figure 8) In other words, separation improved at elevated temperatures. For the sample with a higher solid content (1 %) separation at room temperature was less obvious, even after 17 hours of settlement there was no clear free water phase. From these results it can be concluded that the more solids GTW contains the more difficult it becomes to separate.

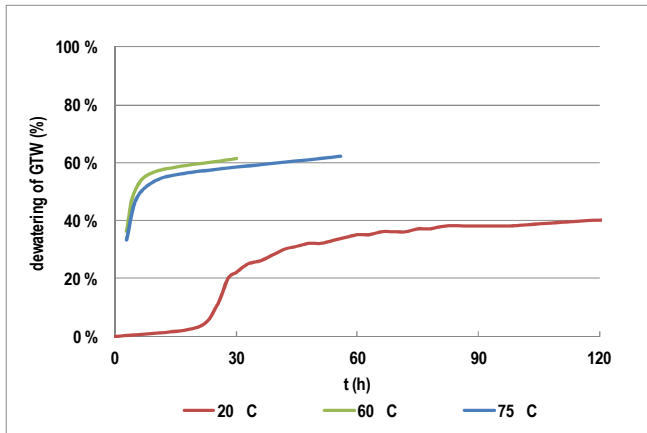


FIGURE 8. Effect of temperature (at 20, 60 and 75 °C) on dewatering of GTW with low dry solid content (0.2%) and 7% TOG.

A laboratory centrifuge was used to simulate the working of a decanter centrifuge. The sample with 1% of dry solids and a total of 92.4% of water was heated up to 65°C and centrifuged for 1 minute at 1000 g. Centrifuging separated the sample into a floating layer (15% of the starting material), and 85% of turbid water (0.1% dry solids) with bottom sludge (1.3% dry solids). The top layer contained still 60% water. The remaining lipid layer was 6.2% of the starting material (GTW).

Compared to settling at 60-70 °C (1 g for 30 min), centrifugation (1000 g for 1 min) improved the dewatering from 60% to 85% and speeded up the settling time. The investment costs of a two- or three-phase decanter centrifuge are tough high and the relative small amount of sediments (< 2%) makes optimal separation of solids difficult with one decanter centrifuge only. Also the sticky grease can give problems when cooling down. Therefore it was tried to find other solutions to optimize separation.

4.1.2 Heating and acid hydrolysis

The laboratory test was scaled up to a 30 litre batch installation. GTW sample material was taken from the waste centre in Oulu in June 2008. Concentrated sulphuric acid was added (0.4%) to lower the pH of the GTW from 5.3 to 1.7 and hydrolyse triglycerides into fatty acids. The pH was kept below 2 by continues addition of sulphuric acid (1:1).

At room temperature, 75% of water separated, but the water phase was still a dirty grey emulsion of fats and solids, which is not allowed to drain off into the sewer. Part of the solids was mixed with grease floating on the water phase, so called rag layer (explained in § 2.2). At the surface floated some white soapy particles.

After 4 hours at 70°C, the floating phase split in two layers; on top a dark oily phase (FAM), and below a rag-layer consisting of grease, light solids and water, similar to the findings of in US patent 7,161,017 B2 (2007). The FAM layer grew in time; after 15 hours at 70°C the top layer was 12% of the total mass, below that a rag-layer of about 6%, followed by 79% of reddish-brown fairly clear water and about 3% of solids on the bottom. The FAM layer solidified below 60°C (yellow lighter colour). The pH of the top layer of free fatty acids was lower (pH 1.2) than the pH of the water phase (pH 1.6 - 2.3). The change of the separation efficiency for the different fractions is shown in figure 9.

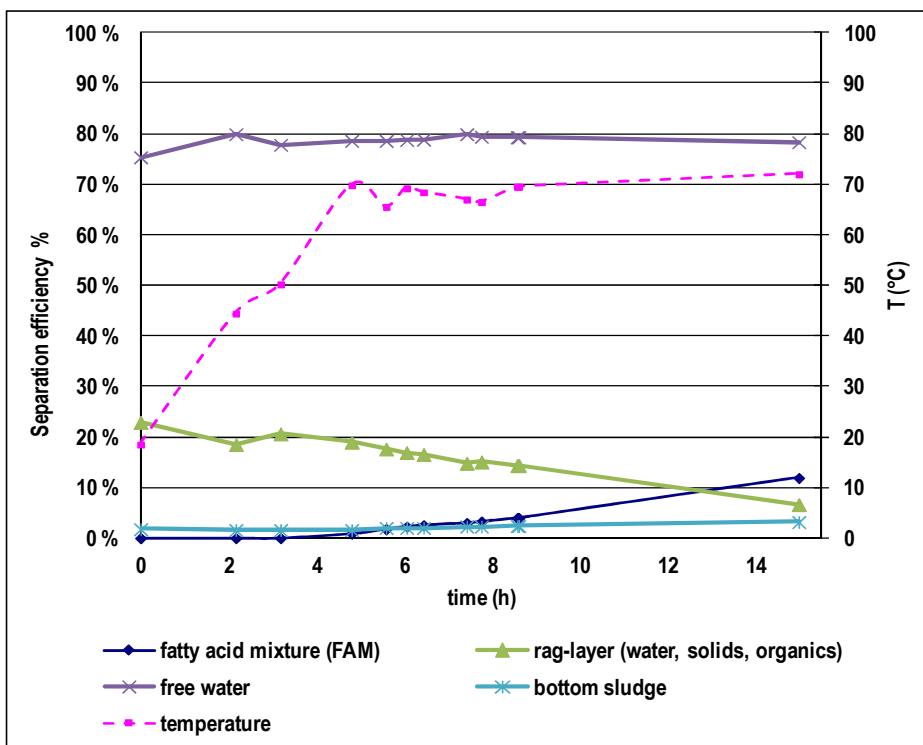


FIGURE 9. Separation efficiency of GTW by heating only.

Laboratory experiments were continued in 2010 with GTW from the waste centre in Oulu. Samples were taken from three different sources, namely GTW from the local fish industry, stored GTW in a Geotube (mainly restaurant GTW) and freshly collected GTW from restaurants. Particles over 4 mm were removed prior testing. The water phase of the fish GTW was white and

cloudy. The Geotube material had been stored outside, in cold conditions (as low as -25 °C). It contained 19 to 22% of free water which was removed prior testing. The fresh taken GTW had a clearly higher solid content than the Geotube material and did not contain free water.

Samples were mixed and transferred into a 1 litre measuring cylinder and heated up in a water bath (65 - 72°C). Sulphuric acid was added and the separation of the layers obtained as %(V/V) (see table 3).

TABLE 3. Results of GTW separation tests by acid hydrolysis (T = 65 – 72 °C)

Test ID	GTW source	H ₂ SO ₄ - addition (%) %(V/V)	free water fraction %(V/V)	rag-layer (solids, water, organics) %(V/V)	oily top layer (fatty acid mixture) %(V/V)
1	fish	0,0%	31%	69%	0%
		9,6%	75%	24%	1%
2	fish	0,0%	24%	76%	0%
		5,0%	64%	36%	0%
3	restaurant (stored)	0,0%	0%*	100%	0%
		0,9%	7%	67%	26%
4	restaurant (stored)	0,0%	0%*	98%	2%
		0,5%	18%	64%	19%
		0,7%	0%	75%	25%
		1,9%	0%	60%	40%
5-1	restaurant (fresh)	0,0%	0%	66%	34%
		0,5%	0%	48%	52%
		0,8%	0%	74%	26%
5-2	restaurant (fresh)	0,0%	0%	83%	17%
		0,2%	0%	47%	53%

* Free water was removed prior acid addition (19 – 22%).

GTW from the fish industry was clearly different than GTW from restaurants. The rag-layer of fish waste needs to be treated separately, as the layer can still include fatty acids. The white cloudy water fraction (high BOD) should be treated prior disposal to the sewage. During acid hydrolysis free water separated (64-75%), but an oily FAM layer was not formed.

In case of restaurant GTW, it appeared to be important to remove water as well as prior as during the acid hydrolysis. After addition of sulphuric acid (0.5%) free water separated, but after a short while the free water phase mixed again with the upper layer. The optimal acid addition for

dewatered restaurant GTW was 0.2 % to 0.5%. An overdose of acid (0.8%) destroyed the FAM layer. Crude GTW needed much more sulphuric acid (2%) than dewatered GTW.

A problem with testing on small scale (up to 30 litres) was that the inhomogeneous samples were not representing the whole waste fraction, resulting in false positive or negative conclusions; a bigger testing installation (5 m³) was designed to test the acid hydrolysis process. In the period 2010-2011, the installation was tested on-site at a waste collection centre in Kajaani. The process was operated manually. GTW (2 – 3 m³) was heated up to 65°C and sulphuric acid was added to endorse separation of water and solids. It was found that the highly variable yield of fatty acids was depending on how well fats were broken up prior to acid hydrolysis.

4.1.3 Saponification prior acid hydrolysis

To improve the breaking up of fats into fatty acids, a saponification step was introduced to the process prior to acid hydrolysis. By raising the pH to 11 – 12 by addition of 1.0 - 1.2% sodium hydroxide, fat changed into a dark thick homogenous mixture of fatty acid salts (sodium soap) and glycerol. Saponification was tested at different process temperatures (40 to 90°C). It was concluded that 90°C and a reaction time of at least 12 hours was sufficient for getting the reaction to an end.

Acid hydrolysis seemed most effective when the soap was first neutralized to pH 7 by concentrated sulphuric acid (addition of 0.5 – 0.9%) and then slowly to pH 3 (another 0.6 – 0.9%). The acid hydrolysis took about 6 hours. At the top a dark lipid layer (10-15% of the starting amount) was formed. On the bottom appeared a brown fuzzy water phase (75 - 85%), which still contained some fats and about 5 - 10% of solids. This phase was difficult to separate by settling. The loss of organic matter to the water phase (as glycerol) is not favourable for utilisation purposes. Dewatering prior saponification, would have decreased the amount of dirty water.

4.2 Pilot plant results

Pilot testing of the process was started in spring 2012 and finished in the end of October. During the period of 23.5.2012 and 21.9.2012, 65 tonnes of GTW was treated, delivered in 7 batches and treated during 5 test runs (table 4).

TABLE 4. Overview of received GTW during the pilot study

Test run Nro.	Batch Nro.	Date received	Amount (t)	pH	TOG (%)
1	1	23.5.2012	11.6	3.6	n.a.
2	2	4.6.2012	9.5	4.1	0.3
3	3	28.8.2012	7.9	4.1	0.4
4	4	18.9.2012	2.2	4.9 *	0.3
	5	19.9.2012	11.3	4.5	0.2
5	6	20.9.2012	11	4.6	0.2
	7	21.9.2012	11		

* Included septic tank waste (110 mg/l NH₄-N and 40% of dark bottom sludge)

4.2.1 Dewatering of GTW

The first batch of GTW (11.6 t) was received at 23.5.2012. The batch was not dewatered, but sodium hydroxide was added straight to the receiving tank to raise the pH from 3.6 to 11.6 at a temperature of maximal 53 °C. Soap was formed. After five days of settlement the pH was 11.3. Sulphuric acid (50%) was added to break the soap into fatty acids; first to pH 7 and then slowly to pH 5. The temperature during acidification was 60 °C. The whole mass was pumped via the separation column to the collection tank. Samples were taken from the surface, the sampling tap (lower intermediate layer) and from the bottom. The surface consisted of a dark floating layer, most likely fatty acids. A sample taken of the top layer contained 10% of TOG, with a pH of 5.8. The intermediate sample was light brown *milky*, with a pH of 5.7 and a TOG of 0.3%. The bottom sample was a dark liquid containing some solids and on top of it some foam, from where can be concluded that part of the fatty acids were still in saponified form (pH 5.0). TOG was 0.6%. As the process build did not have a return line from the collection tank to the reactor (or receiving tank), the batch could not be treated further and was taken away from the installation by a suction vehicle for waste disposal. Because of problems with incorrectly built chemical lines (backpressure valves missing, slope of pipes, position of dosing pump) the chemical somehow addition failed, exact chemical additions were not known.

A second batch (9.5 t) was received at 5.6.2012. The low grease content (0.25% TOG) of the received mass was too small to process in the reactor, if it would have been dewatered first. Therefore, it was decided to saponify the whole batch by addition of sodium hydroxide (pH 9.4) under slow stirring and a temperature of 50°C. Concentrated sulphuric acid was automatically

added by the acid hydrolysis program (neutralisation to pH 7, followed by slow addition to pH 3). Water separation appeared at 78°C, no separation appeared below 50°C. The bottom sludge (1.2 m³) was pumped to water tank for waste disposal (40 % TOG). The water fraction was pumped back to the receiving tank, and the top layer (less than 5% of the starting material) was transferred to the separation column for further examination offside. By centrifugation it was measured that the top layer contained as an average 53% fats or fatty acids, 45% of water and 2-3% of solids (figure 10). The light brown water fraction was pumped to the reactor to raise its pH to 6.3. Straight after pH raising the TOG was below the limit for sewer disposal (27 ppm).

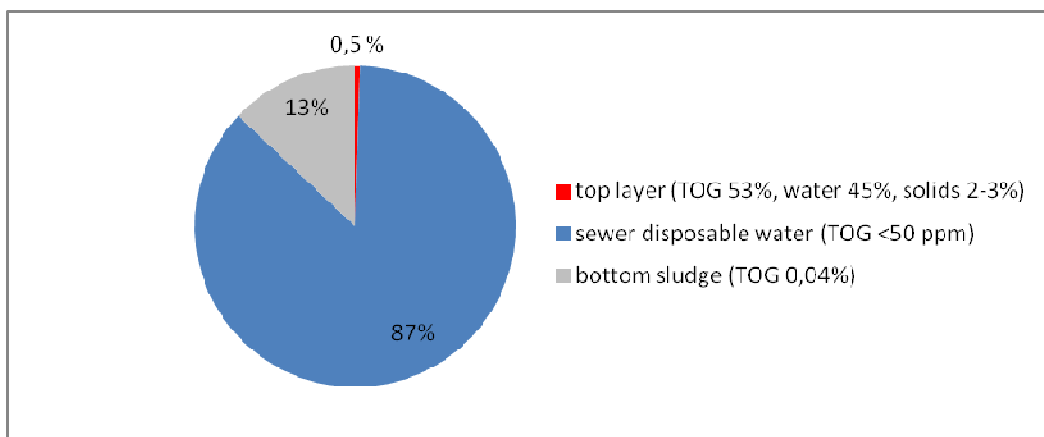


FIGURE 10. Pilot test results of received batch from test run 2 with 0.5% top layer, 87% of free water and 13% of bottom sludge.

In the end of august the third test run was made (7.9 t). The mass (TOG 0.4%) was gently heated up (40°C) and settled overnight. After settling, the water with the bottom sludge contained 0.04% of TOG. The settled fraction was removed (about 50%) and treated in the DAF-installation at pH 8 - 10 to decrease the emulsified grease content in the water phase prior to disposal. The leftover fatty waste was stored in the collection tank for further treatment offsite.

The fourth batch (2.2 m³) contained some septic tank sludge. This was concluded after visual obtaining (40% of bottom sludge, black colour) and too high ammonium content (110 mg/l NH₄-N). The measurement of ammonium was found to be a good quality measure to check if the GTW contains septic tank sludge. As the batch was relatively small it was mixed with batch 5 (11.3 m³). The TOG of batch 5 was low, 0.2%. The mass was gently heated up (40°C) and settled overnight. The next day 3 m³ of water with bottom sludge (TOG 0.03%) was pumped to the water

tank for further DAF treatment at pH 8 - 10. The leftover was returned to the collection tank (about 10 m³) to be mixed with the batch 6 and 7.

The fifth test run consisted of batch 6 and batch 7 each 11 m³, with a TOG of 0.2% - 0.3% and pH 4.6. After settling at 40°C the bottom sludge contained 0.7% TOG, the separated water phase about 70 ppm TOG. The water phase was pumped to the reactor and its pH decreased to 3 to improve the separation of grease and water. After settlement the TOG decreased to 16 – 49 ppm, which acceptable for sewer disposal (limit of 50 ppm). The water phase was transferred to the DAF installation. By raising the pH over 8 and bubbling air, the last part of TOG was removed by the DAF installation. Foam, containing fatty acids salts (soap), was formed and scraped of the water surface.

As foreseen, the pilot plant had continues need for process alterations and repairs. The planned budget was not enough to cover the costs, and repairs delayed the process testing. Delays in the time planning caused e.g. GTW delivery problems during the summer holidays and so on further delays on pilot testing.

The installed dielectric rod to obtain separated phases of water and grease did not work as expected, due to fouling. Similar problems appeared with the electrical conductivity electrodes installed after the reactor and before the DAF tank. Because of fouling and possibly wrong scaling the values were implausible and could not be used as control parameters to automate the process. Due to these restrictions, the process automation could not be further developed.

The collection tank (11 m³) for the separated grease did not have a return to the reactor, meaning that the dewatered fraction could not be processed further after collection.

Technical problems appeared with the chemical line for the addition of concentrated sulphuric acid. Even when used piping and valves were made of acid resistant material, it appeared that 50%-concentrated acid was too strong. The acid oxidized the lines, leaks appeared and valves were blocked. As a solution it was decided to use less concentrated acid (20%). Due to summer holidays the fixing of the lines took several weeks, delaying the test runs badly.

The separated water was treated in a small second-hand DAF-unit which was rebuilt for automatic use in the pilot process. In the end of the pilot testing the process was still operated by

hand, as the automation was not yet ready. A problem with the open unit has also been its unpleasant smell. A replacement of the DAF-unit with a bigger capacity and one that can be closed would be necessary for future use.

Extended dewatering of the top layer for utilisation purposes is possible, but needs an additional smaller reactor and separation column, or preferably a decanter centrifuge and/or disk stack separator for optimal separation depending on the quality needs of the fraction. The pilot plant under study did not include this process step, as the existing reactor (used for dewatering) was too big for this purpose. However former laboratory experiments and literature study have shown that fractionation works out well after sufficient dewatering.

4.2.2 Fractionation of the dewatered mass (FOG recovery)

As the dewatered mass could not be further treated in the pilot plant, the dewatered fractions of batches 5, 6 and 7 were collected in the product collection tank (see figure 7 in chapter 3.2) and treated offsite in a smaller reactor. The amount was about 200 litres, which is 0.6% of the original GTW. The average TOG of this fraction was 27%, which shows that the fraction still contained much water.

A prototype reactor of a steel column with an inside diameter of 157 mm and 1500 mm height was used for further fractionation. The mass was mixed manually by a long stirring rod. The reactor was heated electrically in the lower part of the column. Heating was a problem as the mass cooled down in the upper part during settling. Better isolation of the reactor would have solved this problem.

Sodium hydroxide (addition of 6.5%) was added to increase the pH to 8 and left over the weekend at room temperature. After saponification, sulphuric acid was added to split the formed soap into fatty acids and glycerol. The pH was lowered to 7 by an addition of 0.3% H₂SO₄ at about 65 °C.

Free water (59 %) was removed via the bottom valve (figure 11). The dark brown but fairly clear water had a pH of 4.5. TOG was not measured, but assumed to be as high as 7%. The following fraction (13%) was an intermediate rag-layer of thick sludge with a TOG of 41%. The rag-layer was centrifuged in the laboratory and contained roughly about 50% water, 35% solids and 15% of

oily liquid (FAM) on top. The top layer (28%) solidified below a temperature of 36°C. The TOG content of the upper fraction was 61%, which means that it still contained additional water. Settling only was not effective enough to split off all water.

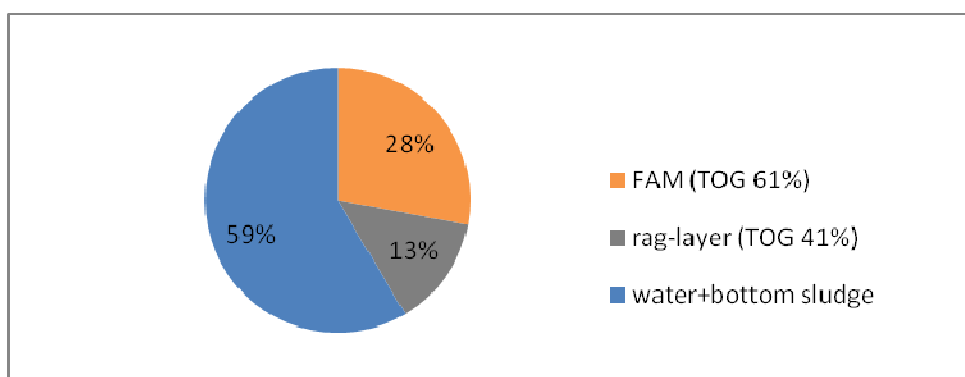


FIGURE 11. Separated fractions of dewatered GTW after saponification and acid hydrolysis in a small prototype reactor.

After separation of the dewatered GTW with a laboratory centrifuge (at 1000 g) three phases appeared; an upper dark oily layer of fatty acids (TOG 95 - 100%), a rag-layer of light solids, organics and water (TOG 36%), and dark brown water. As the FAM layer was nearly 100% of oil and/or grease, dewatering with a decanter centrifuge seemed more effective than settling.

The fatty acid composition of the top layer of the dewatered GTW after chemical treatment and settling was determined by gas chromatography according method ISO 5509:2000 (Jenni Pieti, student OAMK Oulu, 2012). The top layer consisted mainly of unsaturated oleic acid (18:1) and saturated fatty acids as palmitic acid (16:0), stearic acid (18:0) and myristic acid (14:0) (table 5), which was comparable with the fatty acid composition of GTW found in earlier studies (see table 1 in § 3.2).

TABLE 5. Fatty acid composition of dewatered top layer (Jenni Pieti, student OAMK Oulu, 2012)

Common name	C:D	chemical structure	SAMPLE 1 Area %	SAMPLE 2 Area %	SAMPLE 3 Area %	SAMPLE 4 Area %
Oleic acid	18:1	C ₁₈ H ₃₄ O ₂	28,18	29,72	30,27	30,93
Palmitic acid	16:0	C ₁₆ H ₃₂ O ₂	25,78	26,36	27,64	29,16
Stearic acid	18:0	C ₁₈ H ₃₆ O ₂	12,10	11,43	10,81	10,75
Myristic acid	14:0	C ₁₄ H ₂₈ O ₂	6,94	7,66	7,67	8,13
Gondoic acid	20:1	C ₂₀ H ₃₈ O ₂	1,45	2,25	2,00	2,17
Palmitoleic acid	16:1	C ₁₆ H ₃₀ O ₂	3,05	4,40	4,32	4,63
Lauric acid	12:0	C ₁₂ H ₂₄ O ₂	1,47	2,15	2,06	2,22
10-hydroxypalmitic acid	16:0	C ₁₆ H ₃₂ O ₃	9,53	9,89	n.a.	n.a.

4.2.3 Financial feasibility

The financial feasibility of the dewatering process under study is depending on many aspects and assumptions. It has been tried to use realistic costs. Some assets such as energy costs and costs for maintenance were still unknown in this early state and may be over or under estimated. Also the prospective costs for disposal of organic wastes may increase or decrease after the new landfill regulations in 2016 take effect. Waste disposal costs (or even profits in case of utilisation) are depending on the amount of available cost-effective utilisation alternatives for GTW or, in case of a monopoly, there will be no competition and prices will go up. These aspects will affect the profitability of the dewatering plant.

The quantity of the total collected GTW in the area (collected by several private companies), and the amount of GTW currently collected by private company X was used as a base for the forecast. It was assumed that the private company would be able to collect the total amount (estimated as 1285 t/a for the Oulu area, see §2.2) into three years, by providing cheaper GTW collection and disposal fees than its competitors in the surrounding. During the following years, it was supposed that the market would expand, due to stricter environmental regulations on grease traps and emptying intervals and the upcoming legislation in 2016 that prohibits disposal of organic waste on landfills. The maximum amount of GTW received to the plant was estimated to increase to 3000 t/a (15 m³/d), which is the maximum amount the current installation (pilot plant under study) can handle without extensions. If the company would decide not to invest in a dewatering installation it was estimated that there would be no market growth after the first three years. Figure 12 shows a picture of the estimated growth for the private company with and without the dewatering installation.



FIGURE 12. The estimated amounts of collected GTW for private company X with and without the dewatering installation under study.

Production costs include labour fees (at maximum plant capacity one full-time operator and one manager for 20% of the time are needed), monitoring costs, energy consumption, plant capacity (max. 3000 t/v), truck fees for loading and emptying of the plant, costs for repairs, insurances (1% of investment), leaseholds (1% of investment), the quantity and economic value of the organic fraction (as pure FOG or FAM) and the bottom sludge (negative when disposed or positive when sold), the quantity of the separated water and its costs for sewer disposal. Other aspects that have been taken in account include the costs and revenues for GTW collection (based on customers pricing in 2011) and transportation; investment costs of the installation, depreciations, and other fixed expenses.

Profit and loss accounts were prepared for different situations over a period of 10 years. An example for year 6 is shown in figure 13. The dewatering plant was estimated to run at full capacity (3000 t/v). The cost for disposal of bottom sludge and top layer fractions was fixed at 74 €/t, which is the current price for GTW disposal. Dewatering efficiencies were fluctuated.

PROFIT AND LOSS ACCOUNT				
(6th year, plant capacity 3000 t/a, dewatered fraction disposed as GTW (74€/t))				
		50% discharge water	75% discharge water	85% discharge water
	€/a	€/a	€/a	€/a
Revenues (without taxes)				
Service fee for emptying trap and transportation to waste treatment plant	318 258	318 258	318 258	318 258
Service fee for waste processing	214 014	214 014	214 014	214 014
Sales of dewatered fraction (0% of GTW)	0	0	0	0
TOTAL REVENUES	532 272	532 272	532 272	532 272
Variable expense				
Cost price for emptying trap and transportation to waste treatment plant	-143 299	-143 299	-143 299	-143 299
Materials and supplies (incl. energy consumption)	-32 654	-33 891	-34 386	-34 386
Personnel expenses	-79 688	-79 688	-79 688	-79 688
Maintenance costs, facility	-32 460	-32 460	-32 460	-32 460
Disposal fees & transport of produced waste and wastewater	-116 535	-60 743	-38 426	-38 426
Other variable costs (as quality control of sewer water and feedstock)	-44 929	-44 929	-44 929	-44 929
Other administrative and overhead costs (11.2% of total revenues)	-59 614	-59 614	-59 614	-59 614
TOTAL VARIABLE EXPENSES	-509 178	-454 623	-432 801	-432 801
GROSS MARGIN 1	23 094	77 649	99 471	99 471
Fixed expenses				
Laboratory analysis	-3 700	-3 700	-3 700	-3 700
Leasehold, 1%	-1 000	-1 000	-1 000	-1 000
Insurance, 1%	-3 048	-3 048	-3 048	-3 048
Taxes: property (on some items) 1%	-3 048	-3 048	-3 048	-3 048
Repairs and maintenance: buildings only. 1%	-3 048	-3 048	-3 048	-3 048
TOTAL FIXED EXPENSES	-13 844	-13 844	-13 844	-13 844
GROSS MARGIN 2	9 251	63 806	85 628	85 628
Depreciation (straight line, 8 years for the facility, other 20 years)	-32 281	-32 281	-32 281	-32 281
OPERATING PROFIT	-23 030	31 525	53 347	53 347
operating margin				
	-4 %	6 %	10 %	10 %
Loan interest after 6 years (10 years loan, 8% interest)	-13 473	-13 473	-13 473	-13 473
PROFIT BEFORE TAXES AND RESERVATIONS (PBT)	-36 504	18 051	39 873	39 873
VAT24%	0	-4 332	-9 570	-9 570
NET PROFIT	-36 504	13 719	30 304	30 304

FIGURE 13. Estimated profit and loss accounts for different dewatering efficiencies

Net profit and high operating margins are highly depending on the quantity of separated sewer eligible water. Operation margins were calculated for dewatering efficiencies of 50%, 75% and 85%. Figure 14 shows that a dewatering efficiency of 75% will make the plant profitable inside 4 years and at 85% after 3 years of operation (operating margin greater than 0%). The comparison shows also that if only 50% of GTW is dewatered, the plant will not become profitable, as the operating margin stays negative during the whole 10 years-period.

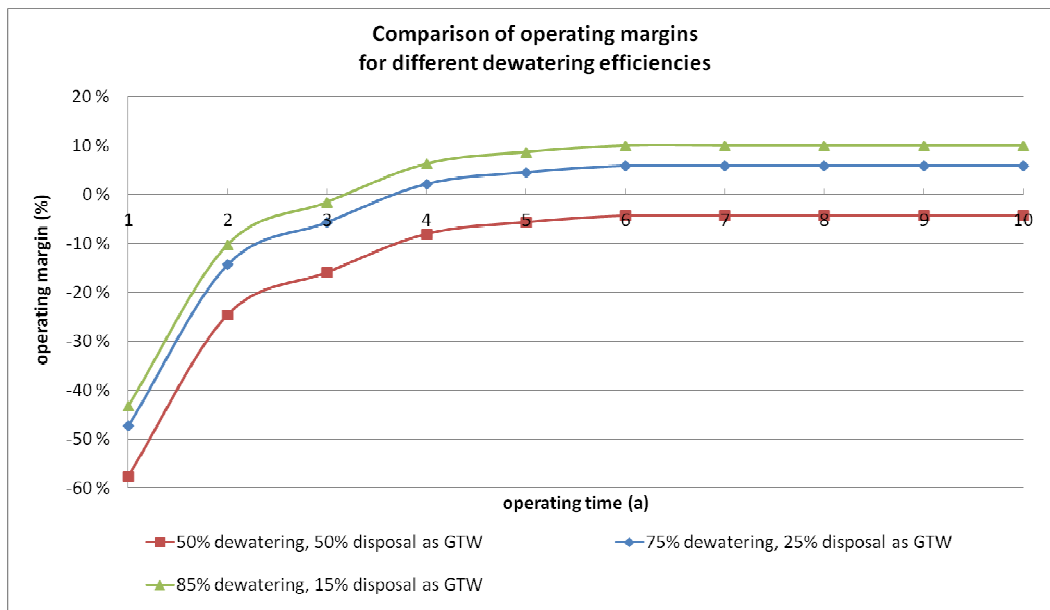


FIGURE 14. Comparison of operating margins for different dewatering efficiencies (50%, 75% and 85%) when the dewatered fraction is disposed as GTW to the local municipal waste centre.

Other examples (figure 15) show how the net profit changes if the organic top layer would be disposed or sold (2% or 7%), assuming that the plant runs at full capacity (3000 t/v after 6 years of operation) with a dewatering efficiency of 85%. The disposal cost was calculated as 74 €/t, according to the price list of Oulu Waste centre in 2013 for GTW. If the organic top layer would be sold as FAM or FOG, assumed that it would be in a pure form, its sales value was estimated to be 300 €/t.

The operating margin is 10% when the dewatered fraction is disposed, which is fairly low compared to collection and disposal only (without an installation). The estimated operating profit (in Euros) for dewatering prior disposal will be though 19% higher than in the case of disposal only. Net profit will be higher after 8 years of operation compared to no installation.

For the second and third case it was estimated that the organic top layer would be sold (2% or 7%). When 2% is sold the net profit increases with 35% compared to disposal only, and with 150% when 7% is sold. These estimations are purely theoretical and not established during the pilot testing.

PROFIT AND LOSS ACCOUNT				
(6th year, plant capacity 3000 t/a, dewatering efficiency 85%, unfractionated FAM/FOG sales at 300€/t)				
	0%	2%	7%	
	FAM/FOG	FAM/FOG	FAM/FOG	
	€/a	€/a	€/a	
Revenues (without taxes)				
Service fee for emptying trap and transportation to waste treatment plant	318 258	318 258	318 258	
Service fee for waste processing	214 014	214 014	214 014	
Sales of dewatered fraction (0% of GTW)	0	18 000	63 000	
TOTAL REVENUES	532 272	550 272	595 272	
Variable expense				
Cost price for emptying trap and transportation to waste treatment plant	-143 299	-143 299	-143 299	
Materials and supplies (incl. energy consumption)	-34 386	-34 386	-34 386	
Personnel expenses	-79 688	-79 688	-79 688	
Maintenance costs, facility	-32 460	-32 460	-32 460	
Disposal fees & transport of produced waste and wastewater	-38 426	-33 863	-22 457	
Other variable costs (as quality control of sewer water and feedstock)	-44 929	-44 929	-44 929	
Other administrative and overhead costs (11.2% of total revenues)	-59 614	-61 630	-66 670	
TOTAL VARIABLE EXPENSES	-432 801	-430 255	-423 889	
GROSS MARGIN 1	99 471	120 018	171 384	
Fixed expenses				
Laboratory analysis	-3 700	-3 700	-3 700	
Leasehold, 1%	-1 000	-1 000	-1 000	
Insurance, 1%	-3 048	-3 048	-3 048	
Taxes: property (on some items) 1%	-3 048	-3 048	-3 048	
Repairs and maintenance: buildings only. 1%	-3 048	-3 048	-3 048	
TOTAL FIXED EXPENSES	-13 844	-13 844	-13 844	
GROSS MARGIN 2	85 628	106 174	157 540	
Depreciation (straight line, 8 years for the facility, other 20 years)	-32 281	-32 281	-32 281	
OPERATING PROFIT	53 347	73 893	125 259	
operating margin	10 %	13 %	21 %	
Loan interest after 6 years (10 years loan, 8% interest)	-13 473	-13 473	-13 473	
PROFIT BEFORE TAXES AND RESERVATIONS (PBT)	39 873	60 420	111 786	
VAT24%	-9 570	-14 501	-26 829	
NET PROFIT	30 304	45 919	84 957	

FIGURE 15. Estimated profit and loss accounts when the FOG fraction is sold at 300 €/t.

The net profit depends for a great extent on the expected economic value of the organic fraction (negative when disposed or positive when sold). Estimations were made by fixing the cost prices for the disposal of discharge water (1.65 €/m³) and bottom sludge (74 €/t). The economic value of the dewatered organic top fraction was varied as disposed as GTW (-74 €/t), at a zero-charge (0 €/t) for example in case of energy production, or sold as product for industrial applications (240

€/t). The comparison of the change in operating margins during 10 years for these different scenarios is shown in figure 16.

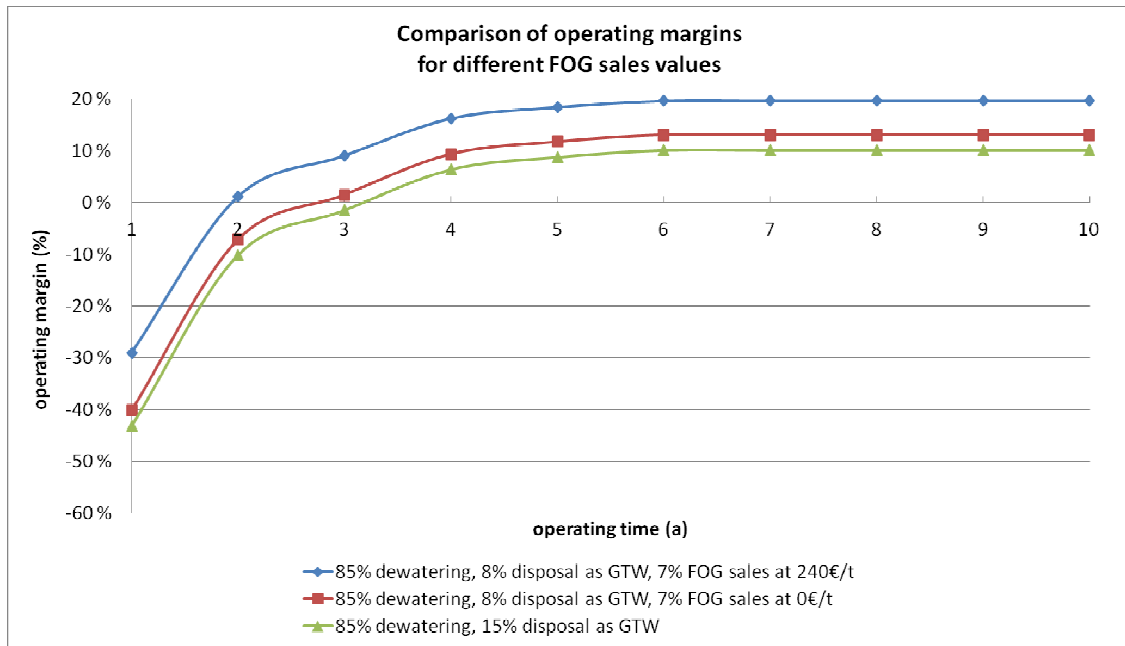


FIGURE 16. Comparison of operating margins for different economic values for the FOG fraction.

To compare, if no installation would be installed, meaning GTW collection and transportation for disposal only without additional investments, the operating margin would be 20%. An economic product value of 240 €/t would provide a similar operating margin, when the dewatering efficiency is 85% and 7% would be a saleable product (FOG or FAM). The net profit of the plant becomes equal to the case of no installation after 3 years of operation and doubles after 5 to 6 years of operation. When the utilised amount is 2%, the economic value of the product should be as high as 1120 €/t to reach a 20% operating margin. This would not be a realistic sales price as the market price for fractionated fatty acids has been 850 €/t in 2013 (ICIS 30.10.2013).

To make the dewatering process profitable, it is in the first place crucial to optimize the dewatering efficiency and cut the disposal costs, by finding cheaper disposal manners (biogas production, compost, co-incineration, pyrolysis). Better profitability will be obtained when the dewatered fraction is sold for industrial purposes that do not need too much of additional dewatering and purification steps such as steam boiler fuel or road soap. Even a zero-charge for the dewatered fractions would make the installation profitable in 2 to 3 years of operation, when the dewatering efficiency is 85% and the market share grows like estimated (figure 16).

4.2.4 Evaluation of the project management (pilot plant)

A practical problem, about 10 times lower grease content in the collected GTW than expected, noticed at the beginning of the pilot testing period required a change of the initial process scope; *FOG recovery of GTW* into *dewatering of GTW*. Since the project of building and testing of the pilot plant was already initiated, it was difficult to make such a huge adjustment. The viability of the whole project should have been re-assessed, as there was a risk that the forecasted result would not justify the original proposed investment in the project after changing the initial scope. Besides that, both aspects were important and depending on each other when it comes to the financial feasibility. Anyhow, an update of the original project plan, with new commitment of the steering group, would have resulted into new justified common primary and secondary goals. This would make it acceptable to focus first on optimizing the dewatering process. After this, suggestions could be made how to improve the separation and concentration of the top layer in a later stadium. The scope remained unchanged, which resulted into some uncertainty and doubt about the working of the process, even though the actual problem was the not the process under study, but the feedstock with not-foreseen extreme high water content (over 99%).

Research has shown that management of human resources, communications and scope defining are the most critical for chances on success or project failure (Jokinen, 2011). Projects dealing with such management problems are triggering inconveniences on time, budget and resource planning. Engineers typically focus only on technical risks, missing market, scope, supplier, resource and management risks that are actually more likely sources of business failure (Merritt & Smith, 2004). For future development projects these aspects should be taken in account, as it will improve the overall project and keeps up the motivation of all people involved in and around the project, which will eventually result in savings on time and money.

5 CONCLUSIONS

In this thesis, the feasibility of a designed process for GTW dewatering and FOG recovery was examined. For this purpose, the regional GTW availability and the potential alternatives for disposal or utilisation of dewatered GTW were evaluated. The technical and chemical part of the design was tested and evaluated by a pilot plant. The financial feasibility of pilot process was calculated for different scenarios by changing the dewatering efficiencies and economic values of the dewatered fraction as it would be utilised (positive value) or disposed (negative value).

It can be concluded that the designed process is technically suitable for dewatering purposes, but is not usable for extended dewatering of the FOG fraction, without extensional process units. To make the dewatering process profitable, it is important to optimize the dewatering efficiency to its maximum (close to 85%) and to cut the disposal costs, by finding cheaper disposal manners. Recovering of the dewatered fraction adds significantly to revenues when the fraction is utilised for such industrial purposes that do not need too much of additional dewatering and purification such as steam boiler fuel or road soap. A zero-charge for the dewatered fractions would make the installation profitable in 2 to 3 years of operation, when the dewatering efficiency is 85% and the installations runs on full capacity (3000 t/v).

Alternatives for disposal of the dewatered fraction may be biogas production, composting, co-incineration or in the upcoming future pyrolysis. Biogas production is favourable as long as the fee for waste processing is not too high. If the amounts of grease are low than composting may still be a reasonable option. Co-incineration is another alternative, but the high water content of the dewatered fraction makes it less cost-effective. Pyrolysis, especially hydrous pyrolysis and gasification, of biowaste into bio oil, syngas and tar is a technology which is still under development, but may become an interesting solution when plants are big enough to become cost-effective which will result in a lower more competitive feedstock price for biowaste.

Even though the profitability of dewatering and disposing the dewatered fraction as a waste is similar to collection and transshipment of GTW only, the process can give the business a competitive advantage, as it can be used for green care marketing purposes (waste reduction and reuse). This will help to increase the companies' market share in the GTW collection business.

The acid hydrolysis step in combination with heating and settling is effective to improve the separation of free water from GTW. As GTW is already acidic, the addition of sulphuric acid to lower the pH to 3 is relatively small. The discard water can be treated by raising the pH up to 8 (with sodium hydroxide) before it enters into the DAF unit. This will decrease TOG contents below the set sewer limit of 50 ppm.

Treating GTW by saponification was also tested by the pilot plant. Saponification is an option to fractionate fatty acids from GTW, but the loss of organic matter to the water phase should be taken in account as it lowers the energy value of the oily fraction and increases chemical and biological oxygen demands in the discard water.

6 RECOMMENDATIONS FOR FUTURE WORK

The test design needs still development and technical adjustment before it can be taken in use. The first improvement should be the building of a thermally insulated wall, to protect the space from cold weather conditions. Only after this the plant can be used the whole year around, which is crucial to make the plant profitable. Other reasons to build the wall are to protect the equipments from dust originated from other activities in the same working area, and to minimize odour passes to the surrounding. To make the process continuously, floor drains and a sewer line should be built, modifications made to the DAF installation, and the water collection tank should be equipped with automatic pH control and base addition. A return line from the reactor back to the collection tank should be built to make the process more effective. A new line for acid addition should be made of acid proof material (as glass fibre) to prevent against leakages. Less concentrated sulphuric acid may be used, e.g. 20 %(V/V). Monitoring of liquid levels in the reactor should be improved and technologies to obtain automatically water/grease interfaces should be reassessed. The automation programming should be updated and modified.

For FOG recovering of the dewatered GTW (brown grease) an own process unit is needed. Separation may be improved by the use of a decanter centrifuge. A minor is its high investments costs. Higher pressure, temperatures, or steam may be helpful to improve extended dewatering and purification of the partly dewatered fraction.

Destruction of the rag layer is complicated; further research is needed to find a solution how to destabilize this intermediate layer of organics, light solids and water. For the moment the fraction remains a non-valuable waste fraction.

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