

# Integrated Master Degree in Chemical Engineering

## *Antifoam in Food Industrial Application*

*Understanding of the Mechanism and Product Development*

### Master Thesis

by

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Developed within the course unit of Dissertation

Performed in

GOVI N.V.



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February, 2013



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## **Acknowledgments**

I would like to thank all the people at GOVI that in one way or another contributed to the accomplishment of my work, especially to my supervisor Ilse Hostens for her patience, availability, constant incentive and support. For their empathy given to the foreign students and the possibility given by the company to make this internship possible.

To my fellow colleagues, Luísa and Mafalda, that lived this experience with me, for their companionship, support, incentive and unconditional help through time.

To Professor José Luís Figueiredo, my supervisor at FEUP, for his advices, availability, time and support.

To Professor Luís Miguel Madeira, for his availability and for giving me this opportunity.

To my beloved friends who accompanied me during my academic journey, helping me when I needed it, especially Raquel, Luísa, Sara, Sofia and João.

And finally a big thank you to my parents, sister and Nuno for their love, unconditional support and incentive in every moment of my life.

## **Resumo**

O principal objetivo do presente trabalho incidiu sobre o desenvolvimento de um produto anti-espuma, baseado no que a empresa retém actualmente no mercado, que fosse de acordo com as imposições colocadas pelo consumidor. Foi dado a entender, por um consumidor, que o produto comercializado possuía falhas substanciais após um curto período de compra/fabrico. Com recurso a várias análises que visavam a resolução do problema, os requisitos impostos foram cumpridos e chegou-se à composição ideal de um produto anti-espuma bastante melhorado e com bons resultados.

Ao longo do trabalho vários testes, baseados em análise bibliográfica, foram utilizados como forma de distinção entre os vários produtos formulados. Os valores ótimos primordiais como os valores de pH, da temperatura da água, a percentagem ideal de sílica na solução, entre outros, foram encontrados pela assimilação dos vários resultados obtidos e a diferenciação dos vários tipos de produtos anti-espuma formulados assim conseguida.

O novo produto cumpre então todas as condições impostas pelo consumidor tendo alcançado resultados melhores do que os obtidos com o produto comercializado, o que leva a uma possível reposição de mercado.

**Palavras-chave:** Antifoaming, Foam film, Defoaming, Foaming.

## **Abstract**

The main purpose of the present work focused on the development of an antifoaming product, based on the one that the company has currently on the market, in an attempt to fulfill the requirements defined by the consumer. It was noticed by a consumer that the commercialized product had substantial flaws after a short period of usage. After profound research and experimentation the requirements have been met and the ideal product composition with good results was reached.

After intensive study, several tests were selected and applied to differentiate the various formulated products. The optimum values such as the pH value, the water temperature, the ideal silica percentage on the solution, among others, were found by the assimilation of the various results obtained, allowing a clear distinction of the formulated antifoam products.

The new product meets all the specifications defined by the consumer and since it has accomplished better results than the commercialized one it may lead to a possible market replacement.

**Keywords:** Antifoaming, Foam film, Defoaming, Foaming.

## **Declaração**

Declara, sob compromisso de honra, que este trabalho é original e que todas as contribuições não originais foram devidamente referenciadas com identificação da fonte.

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Catarina Sousa

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## NOTATION AND ACRONYMS

$B$  - Bridging coefficient;

$E$  - Entering coefficient;

$FV_e$  - Ending foam volume;

$FV_i$  - Initial foam volume;

$F_r$  - Foam Reduction;

$H_F$  - Foam height;

$H_{RES}$  - Residual foam height;

$P_c$  - Capillary pressure;

$S$  - Spreading coefficient;

$V_F$  - Foam volume;

$\gamma_{O/A}$  - Surface tension at the oil/air interface;

$\gamma_{W/A}$  - Surface tension at the water/air interface;

$\gamma_{W/O}$  - Surface tension at the water/oil interface;

AF - Antifoaming;

FA - Foaming Agent;

PBs - Plateau Borders;

RAP - External Reference Antifoaming Product;

# CHAPTER 1

## INTRODUCTION

This introductory chapter aims at describing the context involving the present work, including the relevant literature review, and also regards the thesis organization in its 6 Chapters.

### 1.1 CONTEXT

The prevention of the foaming dates back to the beginning of 20<sup>th</sup> century using mechanical devices to suppress it. These ways were expensive due to the energy that they required for their applications, and there so, to reduce the costs, chemical methods were preferred to destroy foam. Since the late 1940s the antifoaming agents were commercialized and found many applications in the pharmaceutical industry, medicine, food industry, chemical industry, etc. From that time, the lab research on the foam inhibitors begun and several works have been published. <sup>[1]</sup>

This work's aim is to improve one of the company's antifoaming agents that has been commercialized and has a bad feedback from the customer. This product is applied on a food industry department, and it was proposed a development of a new product formulation in order to satisfy the customer needs.

### 1.2 GOVI N.V. - COMPANY PRESENTATION

GOVI N.V. is a family owned company that was created in 1910. It is dedicated to the manufacturer of engineered process-chemicals that are supplied to a variety of industries and the main production sites are located in Belgium, Italy, Serbia and Russia. The company's focus is to improve the existing self-made products and to develop new products that are better performing and economically competitive. <sup>[2]</sup>



Figure 1 - Company's commercial logo.

### 1.3 STATE OF ART

This work is based on two types of antifoaming agents, the oil based and the silicone oil based antifoams. It was implemented four different test methods for effectiveness of those antifoaming agents using different components on their formulations. Therefore, this section addresses the relevant state of the art concerning the antifoaming agents and the theoretical part that they follow.

#### 1.3.1 Brief history

In general, the formation of stable foam causes problems in most industrial processes, affecting directly the quality of the final product, reducing the carrying capacity of containers or causing pumping problems, among others. That is why there is a constant need of antifoaming agents to reduce or completely eliminate the volume of undesired foam. <sup>[3],[4]</sup>

#### 1.3.2 Foam systems

Foam is a highly non-equilibrium system, that consists in a cellular structure in which cells that contain gas are surrounded by liquid films (Figure 2). The stable property of foams is secured by the presence of surfactants, a substance that reduce the surface tension of water by adsorbing at the liquid-gas interface, by the surface elasticity, surface viscosity, steric and electrostatic interactions in the foam films.

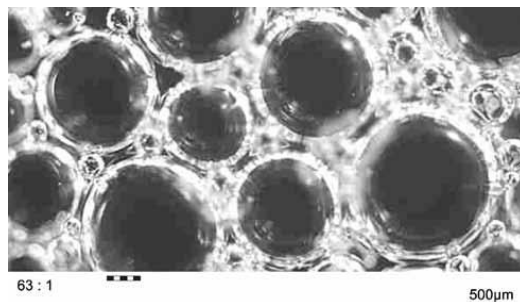


Figure 2 - Photomicrograph of detergent foam. <sup>[3]</sup>

Foam becomes a problem when it is metastable, that is, when it fails to decompose immediately. Their destabilization involves processes based on pressure differences between different sizes foam bubbles, drainage and rupture of the foam films. The last principle is the base of every foam control agents. <sup>[3]</sup>



### *1.3.3 Types of Antifoams*

An efficient foam control agent uses appropriate hydrophobic solid particles, oil drops or oil-solid compounds, depending on the specific foaming agent (surfactants, proteins or soluble polymers). When all the antifoaming entities are dispersed in the solution, they are called as “heterogeneous” antifoams. On the other hand, in some specific cases, the foaming agent can also work as a suppression agent. In that case, the antifoam agent is called “homogeneous”, less efficient antifoam compared with the first one but with various advantages:

- Low costs;
- No residual stains on the final product;
- Food compatibility;
- Etc.

One of the biggest disadvantages of this type of antifoam is the fact that it is very system-dependent, and the system conditions are difficult to predict and maintain as optimum. <sup>[5]</sup>

### *1.3.4 Fast and Slow antifoams*

The antifoams classification is divided into two large groups, which differ on the location where the antifoam enters the air/water surface and begin the foam destruction process, and consequently in their time scales.

Fast antifoams involve a foam film rupture mechanism that usually leads to complete foam destruction within seconds and reduces the foaminess of the surfactant solutions. Therefore these antifoams are preferred when the complete foam suppressing is needed.

On the other hand, slow antifoams, that implicate foam destruction through compression of the antifoam globules in the *Plateau Borders* (PBs)<sup>1</sup>, usually require

---

<sup>1</sup> Plateau Borders (PBs) - Known as the junction of the interconnecting channels of the bubbles. Due to the interfacial curvature between the foam films and the PBs, the pressure is lower on that area, creating a capillary suction effect on the liquid from the center of the film assisting the flow between the neighboring cells.<sup>[7]</sup>

many minutes or hours, due to the slow water drainage from the foam and residual long-standing foam that remains on the last stage of the foam decay. [4],[5]

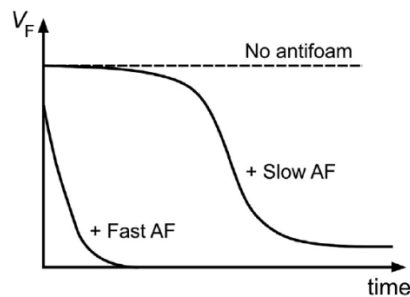


Figure 3 - Foam evolution after foaming, comparison of the fast and slow antifoams. [5]

The Figure 3 illustrates the difference referred before.

### 1.3.5 Antifoaming/Defoaming mechanism

#### - Slow Antifoams:

The presence of slow antifoams defines four distinctive stages in the foam evolution as seen in Figure 4.

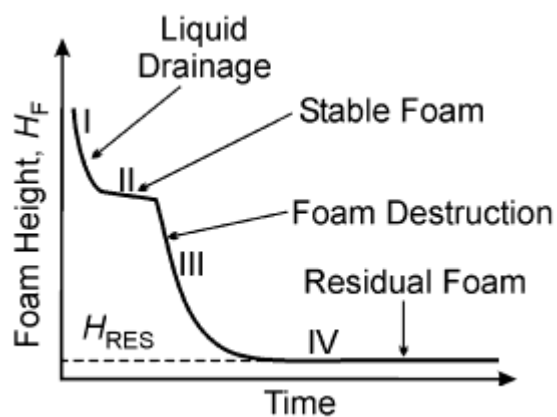


Figure 4 - Stages of foam evolution in the presence of slow antifoam [Foam height as a function of time,  $H_f(t)$ ] [4]

During the stage I, the foam film thins down due to the narrowing of the PBs and nodes (where the PBs met). The smallest bubbles disappeared owing to the air diffusion across the foam films.

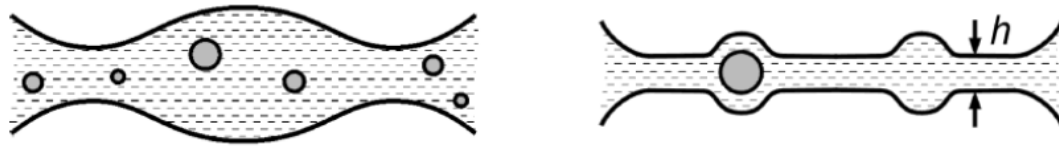


Figure 5 - Narrowing of the PBs and nodes through stage I. [4]

On the stage II, as the small bubbles disappear, the density of the PBs and nodes decrease several times, letting to the oil drops accumulation in their walls. This fact leads to the reduction of the radius of curvature of the PBs walls and therefore it increases the capillary pressure ( $P_c$ ).



Figure 6 - Stretching of the nodes and transport of the antifoam droplets to the PBs. [4]

Once the critical value of the compressing  $P_c$  is reached, the foam destruction begins. The rupture of the bubbles has a constant rate during the most time of the stage III as seen in the Figure 4. This rate then gradually decreases and when the foam volume remains almost constant the stage IV is reached, persisting for hours in some cases. It is notice that in the end of the test the solution still has long-standing residual foam and its thickness depends on the surfactant/antifoam pair. [4]

- *Fast Antifoams:*

The fast antifoams can operate through various mechanisms: *Bridging-Dewetting*, *Bridging-Stretching*, and several mechanisms related to oil spreading. In all of these mechanisms the oil drop connects the foam film surfaces, making a “bridge” between them. The *Bridging-Dewetting* mechanism has four sub processes that are illustrated on the Figure 7.

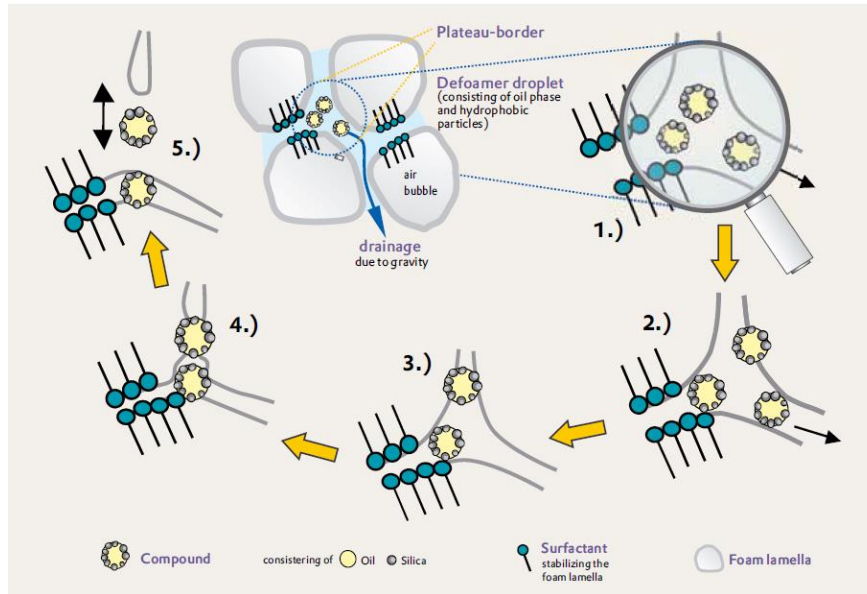


Figure 7 - Illustration of the antifoaming/defoaming mechanism of silica/oil droplets in an aqueous foam: 1.) Draining foam; 2.) Entry of the defoamer droplet into the foam interface and spreading; 3.) Bridging between adjacent foam films; 4.) Dewetting; 5.) Rupture of the foam film. [6]

In the first step, *Draining foam*, the oil droplets from the oil phase are enriched in the water/air interface (foam lamella). This process is described in terms of the entering coefficient,  $E$ , which respects the following condition:

$$E = \gamma_{W/A} + \gamma_{W/O} - \gamma_{O/A} \quad (1)$$

Where,

$\gamma_{W/A}$  - Surface tension at the water/air interface

$\gamma_{W/O}$  - Surface tension at the water/oil interface

$\gamma_{O/A}$  - Surface tension at the oil/air interface

The antifoam drop can only enter the foam lamella if  $E$  is greater than zero. But this condition is not enough for this to happen due to the fact that practical systems are rarely at equilibrium.

During the second step, the liquid flows around the oil droplet resulting on the spreading of the antifoams droplet on the foam lamella and developing a thinner foam film. The spreading coefficient,  $S$ , is given by:

$$S = \gamma_{W/A} - \gamma_{W/O} - \gamma_{O/A} \quad (2)$$

If the spreading coefficient is positive, the “spreading” occurs, forming a thin oil layer at the air/water surface, inducing the beginning of the foam lamella rupture.

On the third and fourth step starts the “bridging” between two different foam bubbles through the oil droplet. This process is given by the bridging coefficient,  $B$ :

$$B = \gamma^2_{W/A} + \gamma^2_{W/O} - \gamma^2_{O/A} \quad (3)$$

This coefficient must be greater than zero for bridging to occur, implying also that the oil/water contact angle must exceed 90 degrees. After that, if every condition is as it was mentioned, the foam film ruptures.

Alternatively, in the *Bridging-Stretching* mechanism, the bridge has time to deform, extending in radial direction until the rupture of the bridge center.

The Figure 8 is a schematic presentation of the different behaviors of the oil droplets with different coefficient's values.

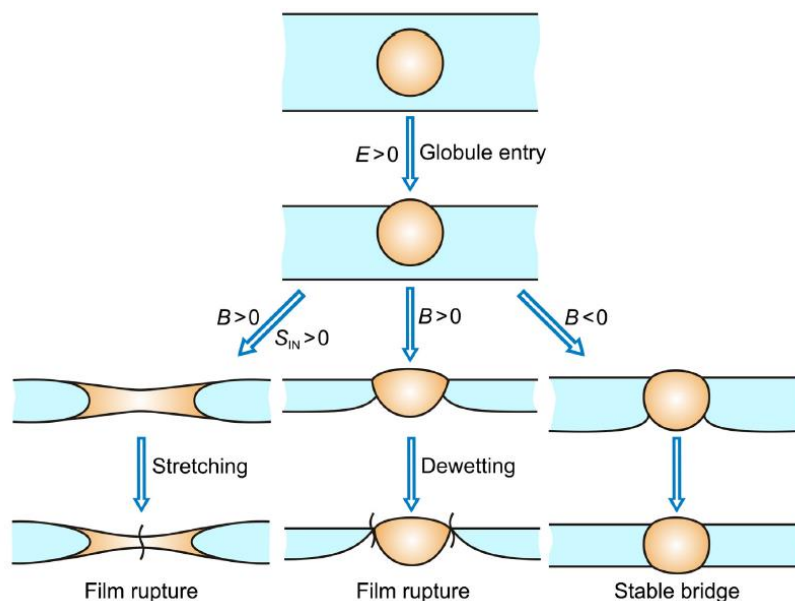


Figure 8 - Schematic presentation of the entry of an oily globule at the foam film surface. [5]

Note that the above coefficients cannot describe the rate at which oil entering and spreading occurs they only determine whether the occurrence of oil entering and spreading is thermodynamically feasible for a specific oil/surfactant system. The scheme presented on Figure 8 shows that it is possible that the foam remains stable

even when the entering coefficient is positive. It happens when the bridging coefficient is negative, and therefore the film rupture does not occur. <sup>[3],[5],[8],[9]</sup>

#### **1.4 THESIS PURPOSE**

This work aim is to improve the current GOVI antifoaming on the market satisfying the existing consumer's needs.

In order to achieve the improved formulation various test were made and the optimum conditions were found, solving the problem of the product's efficiency lost through time.

#### **1.5 THESIS ARRANGEMENT**

This thesis is organized in 6 chapters. After this introductory Chapter presenting the context involving the thesis, in Chapter 2 is described the implemented methods in this work used to analyze the performance of the tested antifoaming agents. Chapter 3 provides the results and their individual discussion that allowed to conclude which were the best conditions and product formulation that truly satisfied the customer needs. Chapter 4 refers to the major conclusions of this work and future improvements. Chapter 5 and 6 regard the references and the appendix, respectively.

## CHAPTER 2

### TEST METHODS FOR ANTIFOAMING/DEFOAMING EFFECTIVENESS

With the aim to determine the effectiveness of antifoaming products four different methods were implemented in this work.

Generally, an antifoaming effectiveness test method comprehends two different phases, the incorporation of the antifoaming product in an aqueous solution and the addition of the foaming agent in the previous solution aiming to a comparison of the formed foam in the presence of various antifoam agents. This chapter presents the four different tests methods used in this work to compare the antifoams efficiency.

#### 2.1 ANTIFOAMING CYLINDER TEST (INTERNAL TEST METHOD)

##### 2.1.1 Summary

This method describes a qualitative method to evaluate the capacity of antifoaming agents in aqueous surfactant solutions.

A certain mass percentage of an anionic tensioactive surfactant, a Foaming Agent (FA), is added to a water/antifoam agent solution. The foam is generated with 20 handshakes of a 250 ml cylinder, and after 30 minutes of rest, any remaining foam is measured.<sup>[10]</sup>

##### 2.1.2 Significance and use

This test is designed to determine the ability of a material to eliminate undesirable foam that can be generated in many food and chemical industries. It could be used to determine the relative effectiveness of one antifoaming agent versus another.<sup>[10]</sup>

##### 2.1.3 Material

- *Apparatus:*
  - Cylinder - The cylinder should have a 250 ml volume;
  - Cylinder plastic caps;
  - Disposable polyethylene pipet (7 ml volume);

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### *Chapter 2 - Test methods for antifoaming/defoaming effectiveness*

- Electronic balance accurate to 0,01 g;
- Beaker - The volume is adjusted by the number of samples;
- pH/temperature measure equipment;
- Stopwatch.

- *Reagents:*

- Antifoaming agent - material to be tested;
- Hard/Tap water stock - The water must be at 25 Celsius degrees, with a hardness no higher than 400 ppm, and a pH between 6 and 8;
- Surfactant solution - Use a 28% [% (m/m)] FA solution.

#### *2.1.4 Procedure*

- *Part I:*

- Put the cylinder on the electronic balance and with the help of a disposable pipet measure 0,16 g of the antifoaming agent (approximately 5 drops);
- Tare the scale and add to the cylinder 80 g of the tap water (note the respective volume);
- Close the cylinder with a plastic cap, and shake it up 20 times and register the respective volume.

- *Part II:*

- Add to the cylinder one drop of the surfactant solution, using a disposable pipet, close again and shake it up 20 more times;
- Once you stop shaking, start the stopwatch and note the volume of the higher layer of foam (*initial top layer foam volume*);
- Observe the foam, and after 30 minutes note the top (*ending top layer foam volume*) and the bottom volume of the foam film (if the foam disappears at the center of the foam film, record the time of the stop watch and each top and bottom volume of the foam film).
- Add one more drop of surfactant solution to the cylinder and repeat the steps above. Do it one more time, until you have a total of 3 drops of FA solution on the cylinder.



- Dispose of the used surfactant/antifoaming agent solutions and thoroughly clean the test apparatus to prevent their carryover to the next determination.<sup>[10]</sup>

## **2.2 STANDARD TEST METHOD (ASTM - E2407, 2009)**

### *2.2.1 Summary*

This method describes a qualitative method to evaluate the capacity of defoaming agents in aqueous surfactant solutions.

A dilute surfactant solution is placed on a high-speed blender, and its mixture generates a certain quantity of foam. After the addition of the defoaming agent and one minute of gentle agitation, the remaining foam is compared with the previous volume. The ability of the defoaming agent to reduce foam is expressed as a percent foam reduction.<sup>[10]</sup>

### *2.2.2 Significance and use*

A defoaming agent is a material that eliminates or suppresses foam that already has been formatted. For the present work this defoaming test was applied on the formulated antifoam products. This allowed the comparison of the defoaming capacity of different antifoaming agents.<sup>[10]</sup>

### *2.2.3 Material*

- *Apparatus:*

- Blender - With a 1,2 l glass cup, at least;
- Disposable polyethylene pipet (7 ml volume);
- Electronic balance accurate to 0,01 g;
- Beaker;
- pH/temperature measure equipment;
- Stopwatch.

- *Reagents:*

- Antifoaming/defoaming agent - material to be tested;
- Surfactant Solution - Tap water solution with a concentration of 1 gram of foaming agent per liter of solution; The water must be at

25 Celsius degrees, with a hardness no higher than 400 ppm, and a pH between 6 and 8; <sup>[10]</sup>

#### **2.2.4 Procedure**

- *Part I:*

- Add to the blender cup 250 ml of the surfactant solution, start the blender on the maximum power and turn it off after 30 seconds. Let it rest for 3 minutes allowing the separation of the foam and the liquid, recording the volume of the created foam (*Initial foam volume*);

- *Part II:*

- Add 200 ppm of the defoaming/antifoaming agent (approximately 2 drops), start the blender on the lower power capacity and turn it off after 1 minute. Let it rest for 6 minutes and record the final/remaining foam volume (*Ending foam volume*);
- Dispose of the used surfactant/defoaming agent solutions and thoroughly clean the test apparatus to prevent their carryover to the next determination. <sup>[10]</sup>

#### **2.2.5 Calculations**

The equation below describes the percentage of foam reduction due to the addition of a defoamer/antifoam product to a solution that produces undesirable foam. <sup>[10]</sup>

$$F_r \% = 100 \times \frac{FV_i - FV_e}{FV_i} \quad (4)$$

Where,

$F_r$  / % - Foam reduction;

$FV_i$  / ml - Initial foam volume;

$FV_e$  / ml - Ending foam volume;

## **2.3 ANTIFOAMING/DEFOAMING ULTRA-TURRAX T50 TEST (INTERNAL TEST METHOD)**

### *2.3.1 Summary*

This method, as the first one, describes a qualitative method to evaluate the capacity of antifoaming/defoaming agents in aqueous surfactant solutions.

- *Application in antifoaming agents:*

A certain mass percentage of FA is added to a water and antifoam agent solution. The foam is generated with a high speed industrial blender, and after 6 minutes of rest, any remaining foam is measured.

- *Application in defoaming agents:*

A dilute surfactant solution is placed on an industrial high-speed blender, and its mixture generates a certain quantity of foam. After the addition of the defoaming agent and one minute of gentle agitation, the remaining foam is compared with the previous volume. The ability of the defoaming agent to reduce foam is expressed as a percent foam reduction.

### *2.3.2 Significance and use*

As the first method, this test is designed to determine the ability of a material to eliminate undesirable foam and it could be used to determine the relative effectiveness of one antifoaming/defoaming agent versus another.

### *2.3.3 Material*

- *Apparatus:*

- Plastic beaker - Volume of 1800 ml;
- Industrial blender Ultra-turrax T50;
- Disposable polyethylene pipet (7 ml volume);
- Electronic balance accurate to 0,01 g;
- pH/temperature measure equipment;
- Stopwatch.

- *Reagents:*
  - Application in antifoaming agents:
    - Antifoaming agent - material to be tested;
    - Hard/Tap water stock - The water must be at 25 Celsius degrees, with a hardness no higher than 400 ppm, and a pH between 6 and 8;
    - Surfactant solution - Use a 28% [% (m/m)] FA solution.
  - Application in defoaming agents:
    - Defoaming agent - material to be tested;
    - Surfactant Solution - Tap water solution with a concentration of 1 gram of foaming agent per liter of solution; The water must be at 25 Celsius degrees, with a hardness no higher than 400 ppm, and a pH between 6 and 8;

#### *2.3.4 Procedure*

##### *Application in antifoaming agents:*

- *Part I:*
  - Put the plastic beaker on the electronic balance and with the help of a disposable pipet measure 1,8 g of the antifoaming agent;
  - Tare the scale and add to the beaker 900 g of the tap water;
  - Mix, with the ultra-turrax T50, the previous mixture for 1 minute with the equipment on its minimum power capacity (not higher than 4000 rpm), and let it rest for 3 minutes recording the respective volume.
- *Part II:*
  - Add to the beaker 11 drops of FA solution, using a disposable pipet, mix it for 30 seconds (power of the equipment not lower than 6000 rpm) and let it rest for 6 minutes recording the final foam volume.
  - Dispose of the used surfactant/antifoaming agent solutions and thoroughly clean the test apparatus to prevent their carryover to the next determination.

*Application in defoaming agents:*

- *Part I:*
  - Add to the plastic beaker 900 ml of the surfactant solution, mixing it with the industrial blender at 6000 rpm and turn it off after 30 seconds. Let it rest for 3 minutes allowing the separation of the foam and the liquid, recording the volume of the created foam;
  
- *Part II:*
  - Add 200 ppm of the defoaming/antifoaming agent (approximately 6 drops), start the industrial blender on the lower power capacity (not higher than 4000 rpm) and turn it off after 1 minute. Let it rest for 6 minutes and record the final/remaining foam volume;
  - Dispose of the used surfactant/defoaming agent solutions and thoroughly clean the test apparatus to prevent their carryover to the next determination. <sup>[10]</sup>

### *2.3.5 Calculations*

This method's calculations follow the same equation given in the standard test method, Equation 4 of the present work. This is only applied on the defoaming effectiveness measure part. <sup>[10]</sup>

## **2.4 ASSIMILATING PUMP TEST (INTERNAL TEST METHOD)**

### *2.4.1 Summary*

This test method describes qualitatively the effectiveness of a certain antifoam agent diluted on a fresh starch solution that it is pumped under high velocity.

A certain quantity of a fresh starch/antifoaming solution is pumped within the same recipient and after 10 minutes the remaining foam on the surface is compared with the foam formed while using the blank solution, a fresh starch solution without the addition of antifoaming agent, and other antifoaming agent's solutions.

### *2.4.2 Significance and use*

This method was applied due to the fact that one major consumer of the company's antifoaming was not completely satisfied. This client uses the antifoam agent to

avoid the formation of undesirable foam through a high pressure water potato cutting process. The water used to cut the potatoes is recirculated on the facilities for one day before its replacement by a fresh one. The durability of the antifoaming effectiveness was questioned since after a few days it did not meet its requirements.

### *2.4.3 Material*

- *Apparatus:*

- 250 ml glass beakers;
- Pump - Flow rate of  $0,26 \text{ l}\cdot\text{min}^{-1}$ ;
- Retort stand clamps;
- 60 ml syringe;
- Heating plate.

- *Reagents:*

- Fresh starch solution - Peel and chop 1,5 kg of potatoes and add 2500 ml of tap water, mix it and let it rest for 24 hours;
- Antifoaming agent - material to be tested;
- Tap/Hard water;
- Distillated water.

### *2.4.4 Procedure*

- Previously wash the pump interior and its tubes with hot and cold distillated water letting each one circulated for at least ten minutes, avoiding any remaining contaminators;
- Make a fresh starch/antifoaming solution with a concentration of  $2 \text{ g}\cdot\text{l}^{-1}$ ;
- Replace the water in the tubes with the previous solution with the help of a syringe;
- Start the pump, avoiding cavitation, and close the let out clamp so the exit fluid gets more velocity;
- Let the solution circulate for 10 minutes and take a picture to compare with the final results of other antifoam samples.

# CHAPTER 3

## RESULTS AND DISCUSSION

This chapter includes the paramount results obtained, which have led to the fulfillment of the main objective of this work. Some of the results mentioned were unsatisfying and not essential to this work conclusion, thus they were not represented to avoid the accumulation of information on one graph that could lead to a difficult interpretation.

### 3.1 REFERENCE ANTIFOAMING AGENT

The Reference Antifoaming Product (RAP) is the base for every antifoaming agent and samples produced at GOVI during this work. It was tested via the antifoaming cylinder test, and Figure 9 represents the behavior of a RAP product (fresh, with four weeks and one year) after the addition of one drop of FA to the antifoaming/tap water solution. The graph represents the variation of the created foam's volume during time, and the "Water Volume" line represents the water/foam interface. Therefore, if the foam volume coincides with the "Water Volume" line, the antifoaming works as a fast antifoaming agent since it has no residual foam.

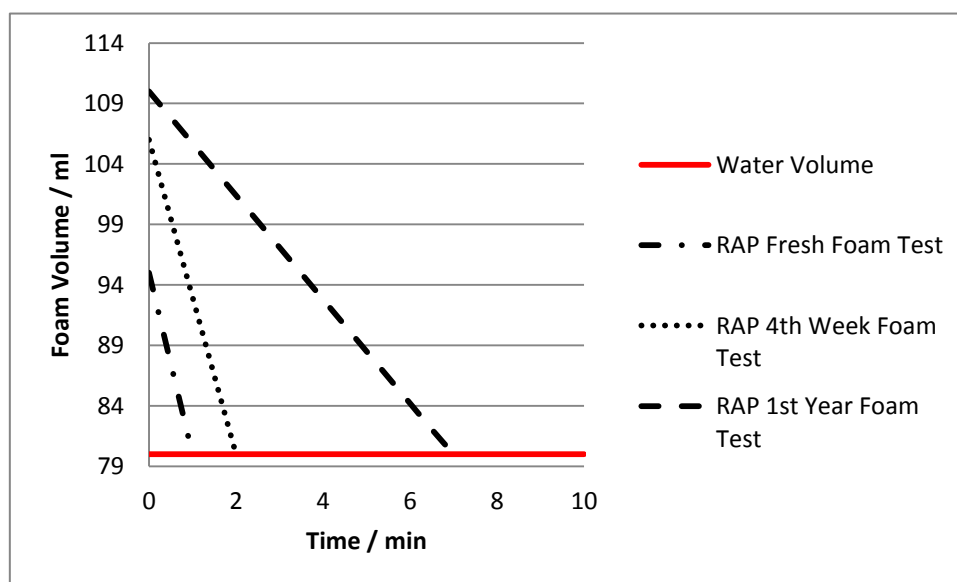


Figure 9 - Foam volume as a function of time for RAP on the first and fourth week.

As seen in Figure 9, RAP behaves as a fast antifoaming agent since it has no residual final foam, and its action takes less than 10 minutes. The difference between tests is explained by the degradation that normally all antifoaming products have through time, usually 6 months after its formulation.

### 3.2 GOVI'S OIL BASED ANTIFOAMING AGENT

The main antifoaming product produced in GOVI, the 0120178.53, is an oil based antifoam that contains a tensioactive and silica powder as the hydrophobic component. This product required alterations in its composition in order to improve its antifoam capacity.

#### 3.2.1 Optimum storage temperature

This sample was formulated on the 20<sup>th</sup> September and divided into three subsequent samples with different storage temperatures, room temperature, 4 and 40 degrees Celsius. Figures 10, 11 and 12 represent the results of the sample .53 through the antifoaming cylinder test on the first and fourth week with the addition of one drop of FA.

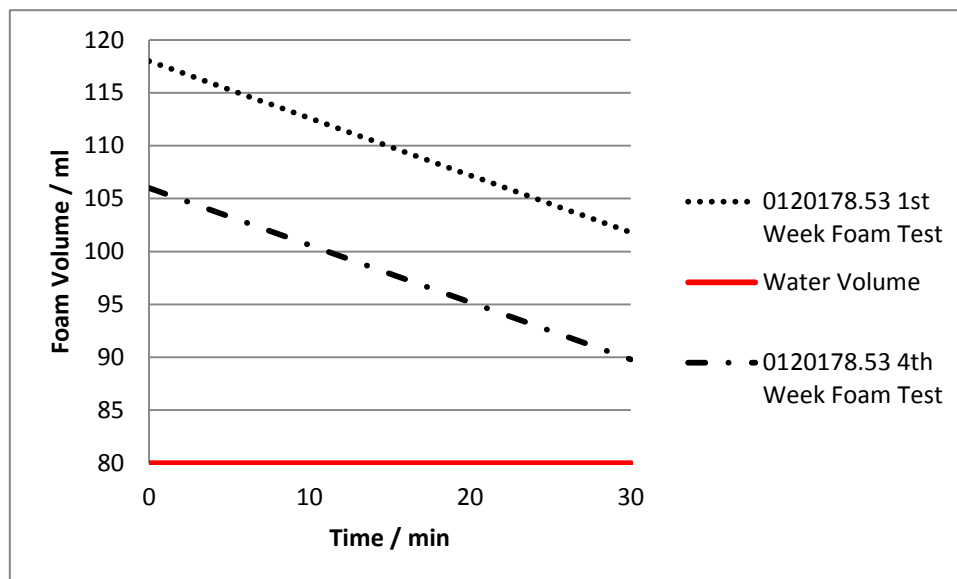


Figure 10 - Foam volume as a function of time for the sample 0120178.53 on the first and fourth week (storage at room temperature).



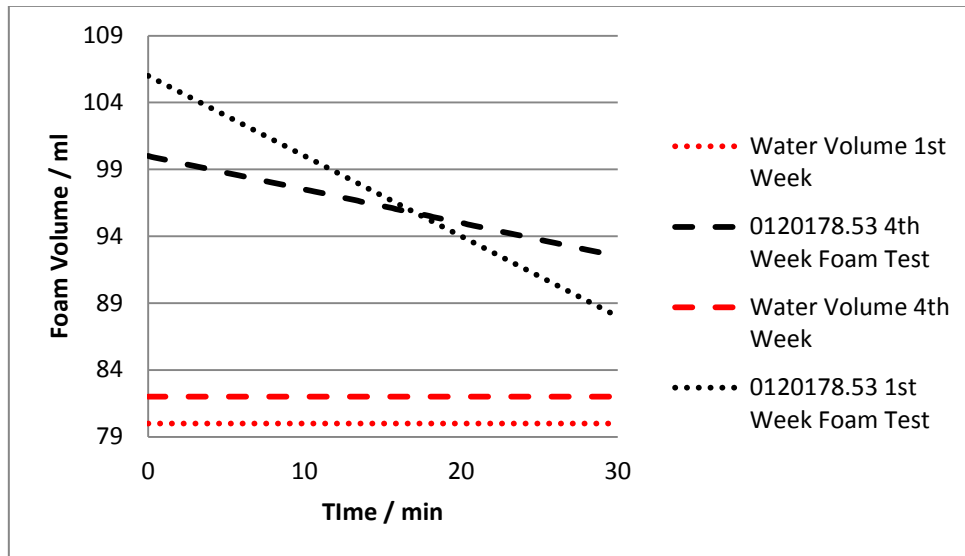


Figure 11 - Foam volume as a function of time for the sample 0120178.53 on the first and fourth week (storage at 4 degrees Celsius).

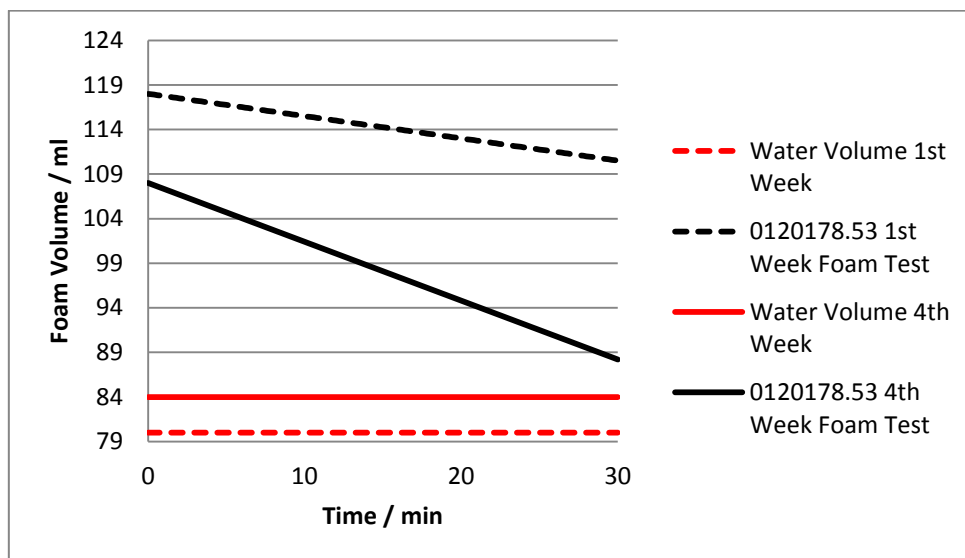


Figure 12 - Foam volume as a function of time for the sample 0120178.53 on the first and fourth week (storage at 40 degrees Celsius).

Figures 10, 11 and 12 show that product 0120178.53 is a slow antifoaming agent due to its final residual foam. Although the improvement on its performance through time, justified by the gradual dissolution of the silica particles on the oil solution, with the exception of the 4 degrees Celsius case, foam is still detected after 30 minutes of rest. Considering the final foam volume after 30 minutes (Figure 12), it is possible to conclude that the optimum sample storage temperature is 40 degrees Celsius. Since most part of the consumers are not equipped with industrial ovens that permit this range of temperatures, the sample must be stored at room temperature, and therefore, the final improved product must have good results

under these conditions. The sample stored at 4 degrees Celsius presented silica agglomerations two months after formulation. This is not an acceptable aspect and for that reason the product must be stored at a temperature above 5 degrees Celsius.

### *3.2.2 Optimum water pH value and temperature*

Since test conditions are key to secure good results, it was tested which pH value and tap water temperature set the ideal conditions.

- *Optimum water pH value:*

To reach the optimum pH value, the GOVI's lab test method was repeated with tap water with a pH value range of 4 to 10. This was made using Sodium Hydroxide (NaOH) and Citric Acid ( $C_6H_8O_7$ ) as an increasing and decreasing product, respectively, of the pH value. The best results were obtained with a pH of 6 and 7, and therefore, another test was made to compare the results using tap water with a pH value of 6 and with tap water without an adjusted pH value. With the results presented on Table 1, it is possible to conclude that the optimum pH value is the one that has not been modified. For that reason, since the tap water pH is between 6 and 8, it is not necessary to use base/acid products in order to achieve better results.

- *Optimum water temperature:*

To discover what the optimal tap water temperature is, several tests were performed, with increasing temperatures of 5 degrees Celsius from 5 to 35 degrees. The results are showed on Table 2, and it is evident that the optimum temperature is 25 degrees Celsius since, at the end, it has no residual foam.

Table 1 - Results of the antifoaming cylinder test with acid and neutral tap water.

Product		Part 1					Part 2					
										Volume of the foam (ml)		
0120178.53 made on 25/09/2013		LES Drops	Product weight (g)	Water weight (g)	1st Volume (ml)	2nd Volume (ml)	3rd Volume (ml)	4th Volume (ml)	Time	Top	Bottom	Dif
Water pH	7,96	1	0,17	80,30	84	84	110	90	30'	90	80	10
		2					110	94	30'	94	80	14
		3					114	98	30'	98	80	18
	6,04	1	0,18	80,62	84	84	110	100	30'	100	80	20

Table 2 - Results of the antifoaming cylinder test with different tap water temperatures.

Product		Part 1					Part 2					
										Volume of the foam (ml)		
0120178.53	Date of Manufacture	Product weight (g)	Water weight (g)	1st height (ml)	2nd height (ml)	4th Height (ml)	5th Height (ml)	Time	Top	Bottom	Dif	
5°C	25/09/2013	0,16	80,09	84	84	110	98	30'	98	80	18	
10 °C		0,16	80,04	84	84	114	100	30'	100	80	20	
15 °C		0,17	80,01	86	84	110	90	30'	90	82	8	
20 °C		0,16	80,00	84	82	110	94	30'	94	80	14	
<b>25 °C</b>		<b>0,18</b>	<b>80,04</b>	<b>84</b>	<b>84</b>	<b>96</b>	<b>84</b>	<b>25'</b>	<b>84</b>	<b>84</b>	<b>0</b>	
30 °C		0,17	80,08	84	84	116	94	30'	94	82	12	
35 °C		0,16	80	84	84	100	90	30'	90	80	10	

### 3.2.3 Optimum silica type and mass percentage

Generally, the antifoaming industry uses two different types of synthetic amorphous silica: the precipitated and the fumed silica. Both types can show a hydrophilic and a hydrophobic characteristic, but it is known that it is important for the antifoaming effectiveness that the silica particles have hydrophobic surfaces. This characteristic allows a higher depth and rate of penetration of the antifoam droplets in the foam lamella.<sup>[3]</sup>

To conclude on which type of silica is best as an antifoaming agent, numerous tests were performed comparing different types of silica and silica grain size. The best results were obtained with sample 0120178.80, which is composed of hydrophobic fumed silica with a smaller grain size than the hydrophobic precipitated silica contained on sample 0120178.53. The sample 0120178.53 used for the comparison was formulated on the 25<sup>th</sup> September, the same day as the other mentioned sample.

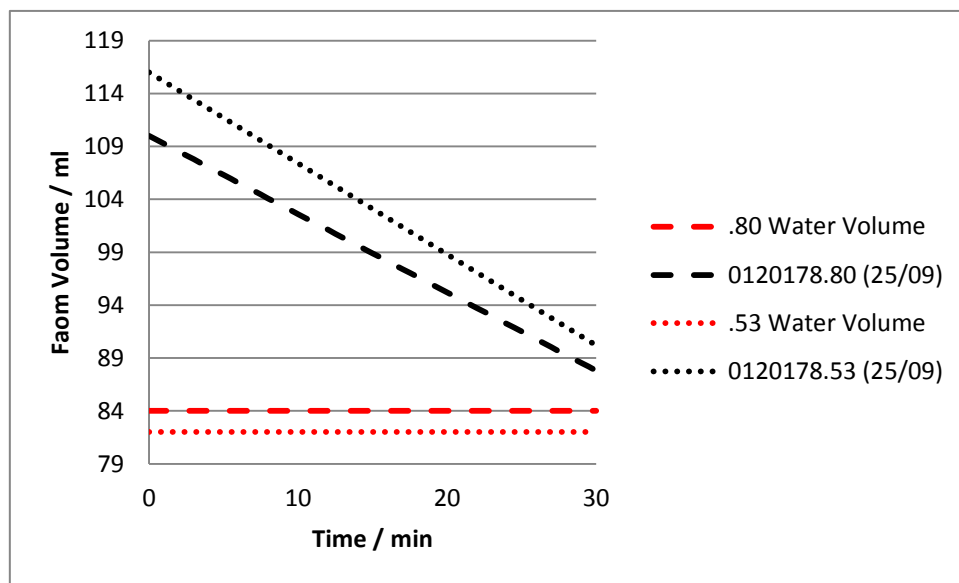


Figure 13 - Comparison of the foam volume as a function of time for the samples 0120178.53 and 0120178.80 four weeks after their formulation.

Comparing the previous results showed on Figure 13, it is clear that the most efficient antifoam agent is 0120178.80. Consequently, it is possible to conclude that the silica particles smaller grain size of the sample .80 allows it to have a faster entry in the foam film and thus a faster film rupture, making this a more effective antifoaming agent.

To understand if the antifoaming effectiveness had some connection with the silica percentage on its composition, tests were performed with the same silica type as the sample .53 but with different silica mass percentage, from 1 to 7 percent.

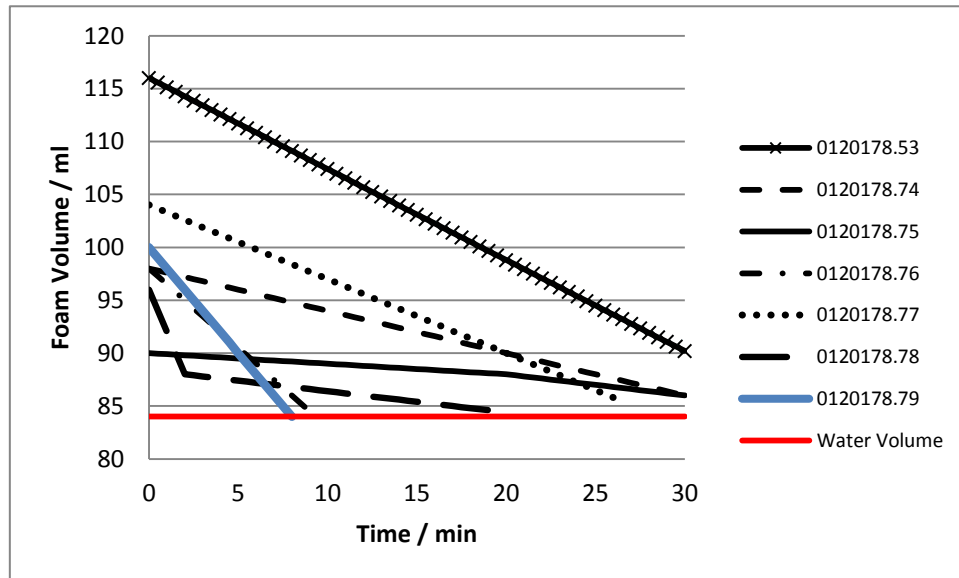


Figure 14 - Comparison of the foam volume as a function of time for samples .53, .74, .75, .76, .77, .78 and .79 to compare the connection between the silica mass percentage and its effectiveness.

Figure 14 shows that increasing the silica percentage decrease the final foam volume and also the action time of the antifoaming agent in order to destroy the created foam. And so, the sample 0121078.79 (with 7 percent of the same silica type as the .53, the blue line on the previous graph) is a better choice as an antifoaming agent than the .53 that only has 1 percent of silica in its composition.

As seen on the results presented on Figure 13, the silica used on the sample .80 was proved to be the ideal silica type to use on an antifoaming agent. For that reason, a sample with 7 percent of the same fumed silica was formulated, tested and compared with the sample .79.

Past four weeks of their formulation the results are the ones presented on the Figure 15.

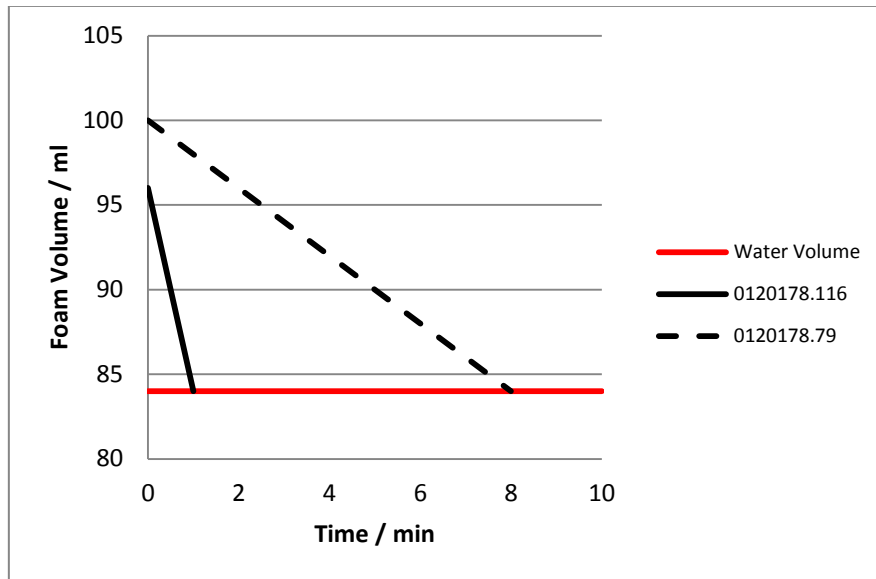


Figure 15 - Comparison of the foam volume as a function of time for the samples 0120178.79 and 0120178.116.

The results presented in Figure 15 show that the sample .116 has the best results, as it can eliminate any created foam almost instantaneously. However, this product has one big disadvantage, it has a pasty aspect, which is not admissible in the industry. On the other hand, the sample .79 remains liquid even in low temperature conditions, it has a good homogeneity and although the worse results, is still a better antifoaming agent than the one on the market, the .53.

#### 3.2.4 Optimum oil type

Since the type of oil also influences the rate of silica dissolution in the antifoaming sample, various samples with different oil types were formulated and tested in order to select which oil improves best the antifoaming property.

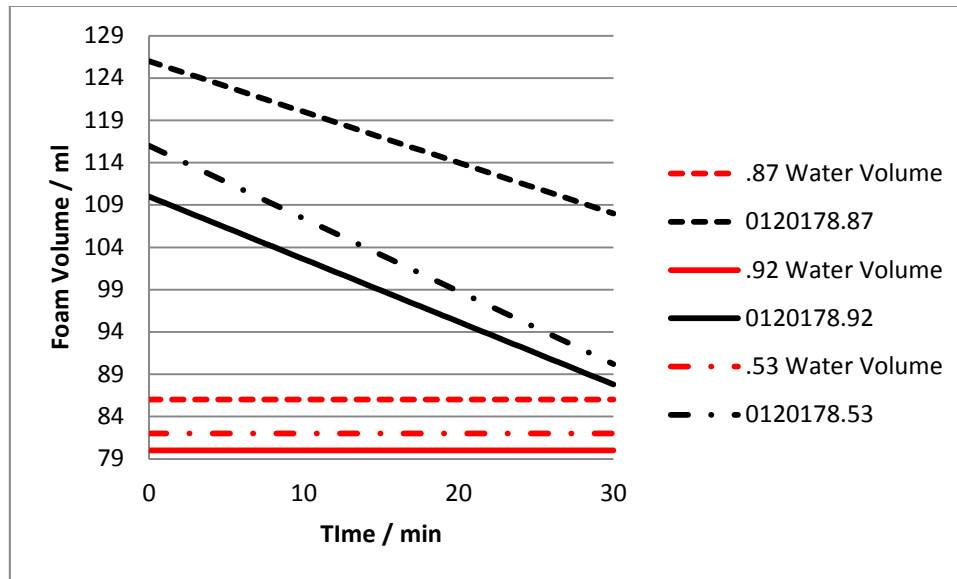


Figure 16 - Comparison of the foam volume as a function of time for samples .53, .87 and .92 in order to select the best oil type is.

Analyzing the previous graph, it is possible to see that the best results were obtained with the samples .53 and .92. Since the oil used on the sample .92 is more expensive, and the difference between their results is minimal, the best solution is to continue to use the same oil type, a vegetable oil. This oil was used on every sample mentioned until this section, so the sample .79 is still the main option for the replacement of the GOVI's actual antifoaming.

Note that other oil types were tested but since results were much poorer than the results obtained with the .53 sample, and as the accumulation of information on the graphs can be confusing, only the oil types with the best results were presented.

### 3.2.5 Optimum tensioactive

Tensioactives are chemical substances with a polar/non-polar structure that are usually located at the surface impeding the movement of molecules into the body of the fluid to attain a lower energy level and consequently reducing the surface tension.<sup>[11]</sup>

In order to remark if the used tensioactive type is the right one, new samples were made which combined anionic tensioactives with the nonionic one used in the GOVI antifoaming agent. The percentage with the best results was 30 percent of anionic and 70 percent of nonionic tensioactives. Figure 17 shows the test results of the

samples .53 and .79 compared with the results of the best sample, the 0120178.110, that does not contain silica in its composition, four weeks after its formulation.

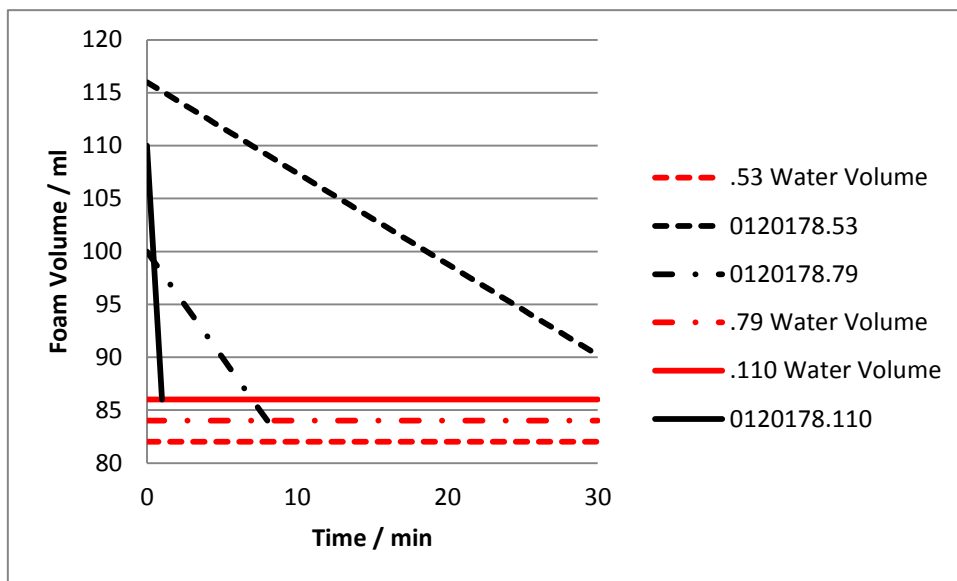


Figure 17 - Comparison of the foam volume as a function of time for the samples .53, .79 and .110 with the aim of discovering which the best tensioactive type is.

The results presented on Figure 17 show that the sample .110 can be entitled as the “perfect” antifoaming agent since foam destruction is almost instantaneous. However, it has the particularity of going against the consumer prerequisites, as it creates a layer of large particles on the surface of the antifoaming/water solution (Figure 18), thus it is not an option for the food industry, since the particles could get attached to the food surface, or cause problems on the facility line production.



Figure 18 - Solution’s surface aspect after adding the sample 0120178.110.



### 3.3 UPGRADE OF GOVI'S OIL BASED ANTIFOAMING AGENT

From results shown on the previous sub-chapter it is possible to choose the best replacement of the actual GOVI's antifoaming agent, always minding the limitations imposed by the client. It has to be a food approved and oil based product that has better results than the current one.

For that, the antifoaming 0120178.79 was the one selected. This section summarizes the results obtained in all the tests mentioned in this work that compare the GOVI's antifoaming agent and the replacement sample.

#### 3.3.1 Antifoaming cylinder method (Internal test method)

Figure 19 illustrates test results, under the same conditions, of samples .53 and .79.

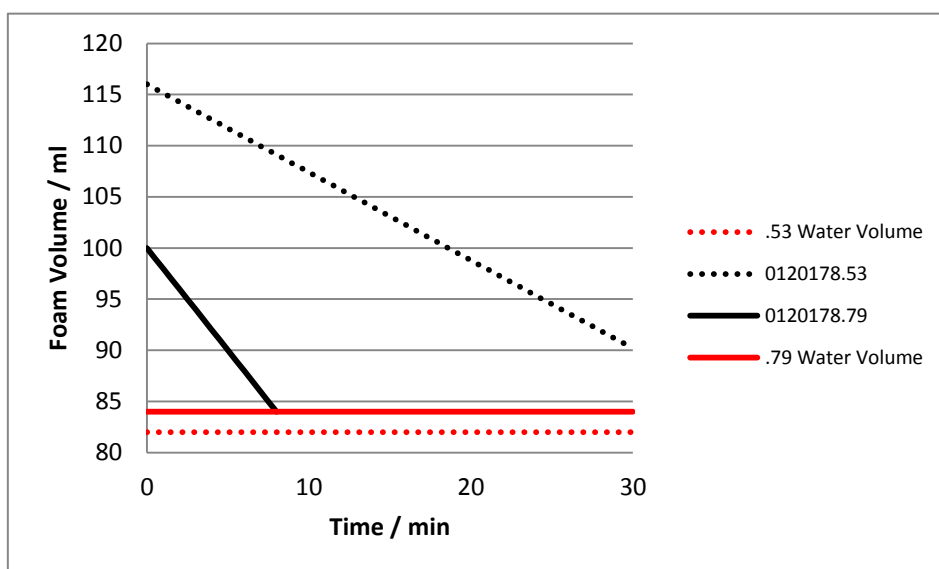


Figure 19 - Comparison of the foam volume as a function of time for samples 0120178.53 and 0120178.79 using the antifoaming cylinder test.

As predicted, the sample .79 has a better behavior, resulting in a final solution without any residual foam.

#### 3.3.2 Standard Test Method (ASTM - E2407, 2009)

The next table shows the results obtained with the samples 0120178.53 and 0120178.79 using the standard test method. The concentration of the surfactant solution is  $1 \text{ g}\cdot\text{l}^{-1}$ , as indicated previously in this report.

**Table 3 - Results of the samples 0120178.53 and 0120178.79 using the Standard Test Method (Blender Test Method).**

Product	Concentration (ppm)	Part 1			Part 2			
		Top	Bottom	Foam Volume (ml)	Top	Bottom	Foam Volume (ml)	Foam Reduction (%)
<b>0120178.53</b>	200	510	140	370	256	250	6	<b>98,4</b>
<b>0120178.79</b>		500	240	260	256	250	6	<b>97,7</b>

Analyzing the results it is safe to conclude that both antifoaming agents are good defoaming products, since this test is used to measure the defoaming effectiveness on an antifoaming/defoaming agent. It is important to mention that the graduation of the blender does not permit an accurate reading of the results.

### *3.3.3 Antifoaming/defoaming ultra-turrax T50 test (Internal test method)*

*- Measure of the antifoaming effectiveness:*

Table 4 shows the result attained with the antifoaming ultra-turrax T50 test, using the named industrial blender.

It is noticed that the sample 0120178.79 has better results and therefore is a best antifoaming agent than the sample 01201878.53, agreeing with the conclusions referred in the sub-chapter 3.3.1.

*- Measure of the defoaming effectiveness:*

Table 5 shows the results obtained also with the same method, but in this case using a different methodology aiming to see which is the best defoaming agent.

It is noticed that the sample 0120178.53 has better results and therefore it is a best defoaming agent than the sample 01201878.79. On sub-chapter 3.3.2 it was not evident which was the best defoaming agent, and with the previous results it is possible to conclude that it is the sample 0120178.53. But since the client needs an antifoaming agent, sample 0120178.79 is still the best option. The foam generated using this test method, is thicker and denser due to the high speed produced by the industrial blender, which explains the difference between the percentages on Table 3 and Table 5.

Table 4 - Results of the samples 0120178.53 and 0120178.79 using the antifoaming ultra-turrax T50 test.

Product	LES Drops	Part 1						Part 2					
		Date of Manufacture	Product weight (g)	Water weight (g)	1st height (ml)	2nd height (ml)	pH	3rd Height (ml)	4th Height (ml)	Time	Volume of the foam (ml)		
											Top	Bottom	Dif
0120178.53	11	25/09/2013	1,83	902,45	960	970	7,55	110	1050	6'	1050	950	<b>100</b>
0120178.79			1,80	902,07	950	960	7,65	1150	1000	6'	1000	950	<b>50</b>

Table 5 - Results of the samples 0120178.53 and 0120178.79 using the defoaming ultra-turrax T50 test.

Product	Defoaming Agent	Part 1						Part 2			Foam Reduction (%)
		LES weight (g)	Water weight (g)	pH	Top	Bottom	Foam Volume (ml)	Top	Bottom	Foam Volume (mL)	
0120178.53	200 ppm	3,25	901,16	7,53	1400	825	<b>575</b>	1350	850	<b>500</b>	<b>13,04</b>
0120178.79		3,25	900,13	7,51	1450	830	<b>620</b>	1500	890	<b>610</b>	<b>1,61</b>

### 3.4 SILICONE OIL BASED ANTIFOAMING AGENT

The silicone oil based antifoaming agents have a lower surface tension comparing to the other materials. For that reason, the thermodynamic requirements, the spreading, penetration and bridging coefficients are all positives in most cases. If they are present as antifoam emulsions they spread out particularly well and homogeneously in aqueous liquids. As a result, the silicone antifoaming can be used at a much lower dosage and the products can be used over a wide range of temperatures. Although silicone oil based antifoams cost more to produce than the oil based ones, they have more versatility and high efficiencies.<sup>[12]</sup>

After testing samples of different silicone oil based antifoam emulsions it became evident that one of the products was flawless in terms of foam destruction even with high concentrations of FA. Figures 20, 21 and 22 represent the different behaviors seen on samples 0120178.79 and 0120178.190, oil based and silicone oil based antifoams, respectively, with different FA quantities.

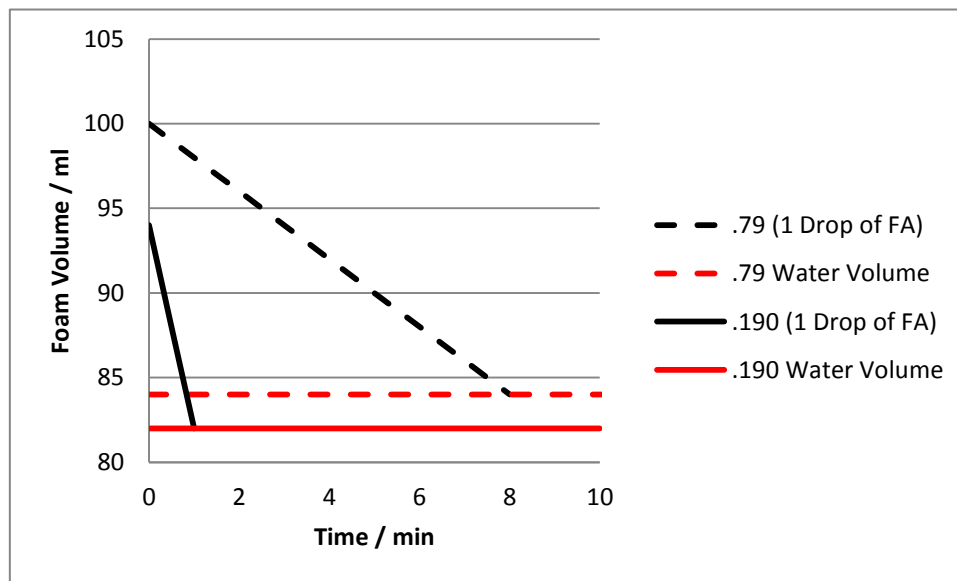


Figure 20 - Comparison of the foam volume as a function of time for samples 0120178.79 and 0120178.190 with one drop of FA.

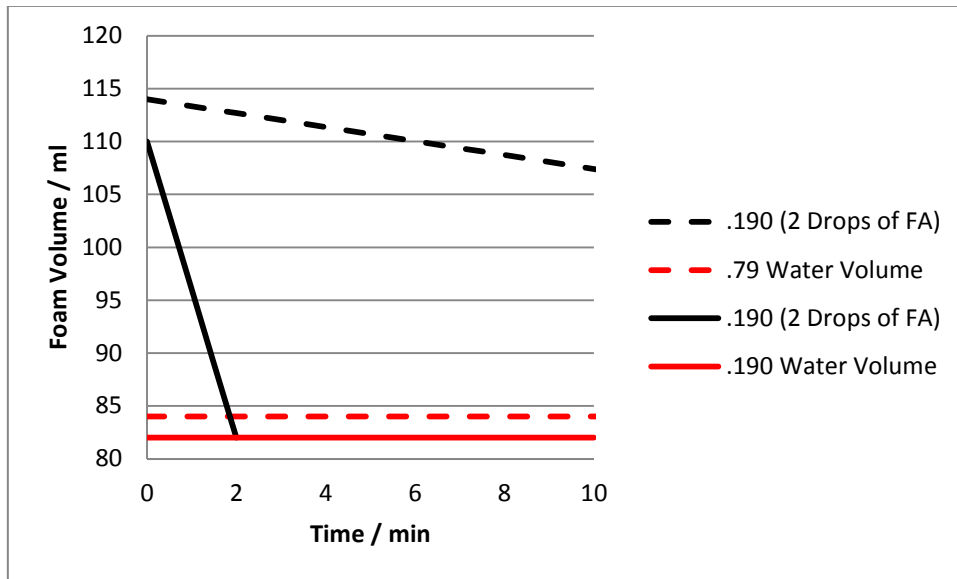


Figure 21 - Comparison of the foam volume as a function of time for samples 0120178.79 and 0120178.190 with two drops of FA.

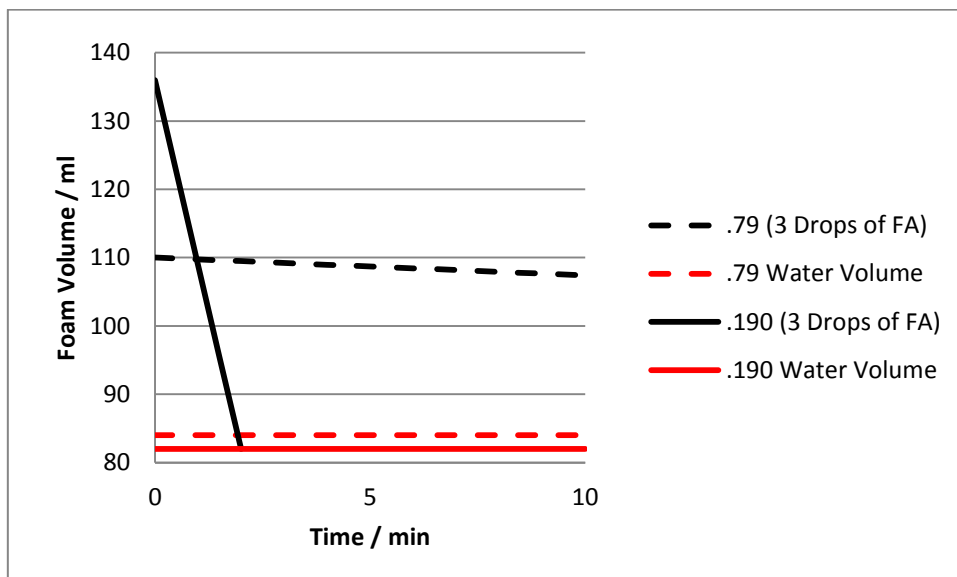


Figure 22 - Comparison of the foam volume as a function of time for samples 0120178.79 and 0120178.190 with three drops of FA.

It is evident that the silicone oil based sample is the one with the best results, eliminating foam in less than 5 minutes even with three drops of FA. Although the impeccable results, this type of product is not food approved and therefore is not an option for the current consumer. Nevertheless, it is a very efficient antifoaming agent, and it can be used in other industries. Even though emulsion breaks after a few weeks, this problem can be solved by altering the formulation/its composition.

### **3.5 SAMPLE BEHAVIOR ON ASSIMILATING PUMP TEST**

Since the main objective of this work is to replace the current antifoaming agent that GOVI has on the market, a test method was elaborated to compare the behavior of a fresh starch solution (blank solution) with and without the improved antifoaming agent. The test is called assimilating pump test which is described on chapter two of the present work.

Primarily, the previously made fresh starch solution was submitted to an antifoaming cylinder test in order to see how much foam volume was created due to the presence of starch on the solution.



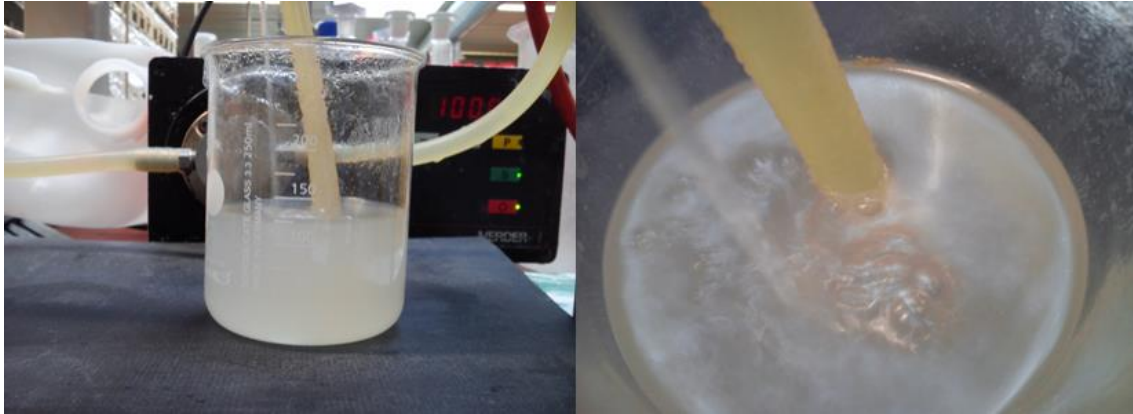
**Figure 23 - Residual foam volume of the blank solution after 30 minutes of rest.**

In this case, the solution with only fresh starch had residual long-standing foam with approximately 10 milliliters. Figure 24 shows the final result of the pump test made on the blank solution. It revealed that, under a certain circulating velocity, the starch is capable of creating foam that remains after a long time, which is prejudicial to the consumer facilities.



**Figure 24 - Results obtained with the blank solution on the assimilating pump test method.**

On the other hand, the results obtained with the addition of the improved antifoaming agent (0120178.79) are much more satisfying and no foam is created during the test (Figure 25).



**Figure 25 - Results obtained with the fresh starch/antifoaming solution on the assimilating pump test method.**

So it is safe to conclude that the addition of the improved antifoaming agent is the best option to avoid the formation of undesirable foam on the consumer facilities. This shows that the sample 0120178.79 is a good antifoaming oil based sample, with satisfying results long after being formulated, which could solve client complaints.

The results obtained using samples .110, .116 and .190 are presented on the appendix. Samples .110 and .116 formed big particles that could damage the potato quality as well as the customer facilities. Sample .190 appears to have the same fulfilling results as the .79 sample, but unfortunately it is not approved in the food industry.

## CHAPTER 4

### CONCLUSIONS AND FUTURE WORK

This work was aimed to develop an antifoaming product, based on the one that the company has currently on the market, in an attempt to improve the latest and fulfill the customer upcoming needs. This new version had to be able to combine durability, efficiency, be food approved and still present a homogeneous appearance at room temperature storage.

In order to deliver an antifoaming with such characteristics range, several test methods were implemented with the purpose of determining which were the optimum conditions that could lead to an ideal antifoaming product with better results than the one currently on the market. After this investigation, one of the tested formulas was found to be the best replacement since it fulfills all the requirements of the unsatisfied customer. This sample, the 0120178.79, is an oil based antifoam, and for it, food approved, that has a stable and homogeneous aspect through time at a room temperature storage. After one month of tests the results were still satisfying, unlike the sample of the commercialized product, and therefore the product can possibly be a good replacement that could improve client satisfaction.

The results obtained showed which were the optimum conditions to achieve ideal antifoaming properties. The optimum temperature storage for the product is 40 degrees Celsius. Since it is a difficult temperature mark to the majority of companies, a room storage temperature is not an option to exclude, as the results showed that although performance is worse, the difference is not that significant. The optimum pH values are between a range of 6 and 8. As tap water fits this range, all of the tests were made using tap water without previous pH regulation. The ideal water temperature is 25 degrees Celsius, and the optimum silica percentage is 7 percent. Since the best silica type, hydrophobic fumed silica, generates a sample with a non-acceptable viscosity, the replacement product has in its formulation the same silica of the one that is commercialized, hydrophobic precipitated silica. Regarding the optimum oil type and keeping in mind that it has to be food approved, the one with best results was a vegetable oil. The optimum tensioactive type, 30 percent of anionic and 70 percent of nonionic tensioactives, had good results but unfortunately it has the disadvantage of creating big particles on the water/air



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### *Chapter 4 - Conclusions and future work*

interface, which is a non-desirable effect in the food industry. Therefore, the tensioactive used on the developed product is on its whole nonionic.

Concerning about other industry types, some silicone oil based antifoams were tested and it turns out to be clear that this type of antifoams were much better foam destructors than the silica oil type. Since this type of antifoam is typically an emulsion, it can easily break, resulting on a non-usable product. As a suggestion for future work, solving this problem seems to be a good chance to achieve even faster and effective antifoams.

One of the main limitations of this work was the inability to reproduce an actual high pressure pump. The pressure of the water used to cut the potatoes on customer facilities is one of the main causes for foam formation, and consequently it determines if the antifoaming formulated is a good replacement or not. Another limitation was the product storage temperature. Since it must be above 5 degrees Celsius, it requires a specific isolated storage place. In Belgium, where the client's facilities are located, the temperatures at night can be below zero degrees Celsius which can damage the product.

This investigation work was successful since the main goal was accomplished based on the results obtained with the implemented tests, and it was a good opportunity to acquire new personal and professional skills.

## CHAPTER 5

### WORK REFERENCES

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## CHAPTER 6

### APPENDIX

This chapter contains the results obtained with the samples .110, .116 and .190 on the assimilating pump test method.

- 0120178.190:

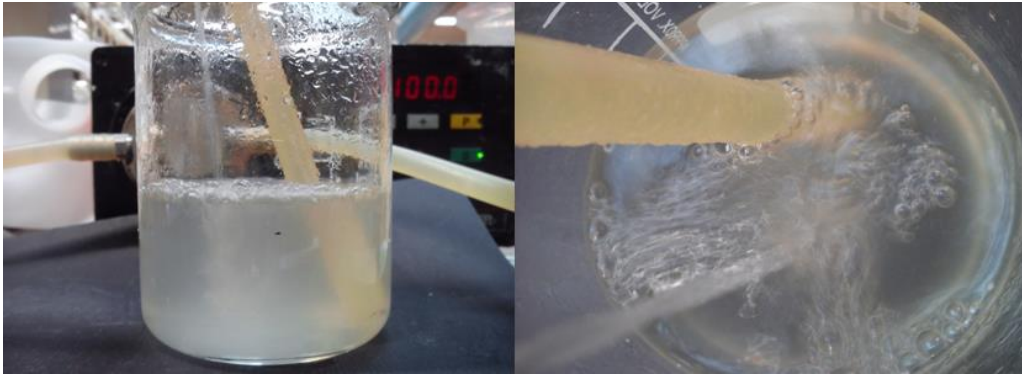


Figure 26 - Results obtained with the fresh starch/0120178.190 solution on the assimilating pump test method.

- 0120178.116:



Figure 27 - Results obtained with the fresh starch/0120178.116 solution on the assimilating pump test method.

- 0120178.110:



Figure 28 - Results obtained with the fresh starch/0120178.110 solution on the assimilating pump test method.