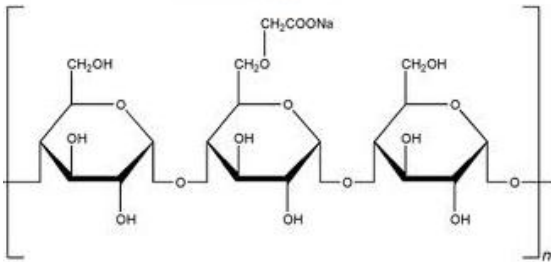# CHALMERS

Characterisation of Starch Properties in Retorted Products

Master of Science Thesis

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Göteborg, Sweden, 2013

THESIS FOR THE DEGREE OF MASTER OF SCIENCE

Characterisation of Starch Properties in Retorted Products

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Abstract

Starch is an important component in food industry; it contributes with energy for example in rice, wheat, maize etc. It is also significant in smaller doses to improve the properties of food such as viscosity, texture, thickness etc. However the starch granules hydrate easy, swell rapidly, lose viscosity, and produce weak bodies which create a very stringy and cohesive paste. Therefore most starches are modified to improve these properties.

The aim of this study is to find the target starch which is one that can either be used as a filling agent in the fillers or one that contributes with good viscosity and texture to the final product and remains constant during and after storage time.

The procedure of finding a suitable starch that fulfils all the requirements involves many steps. The first step is contacting different starch suppliers to get starch samples. Then the starch samples from suppliers with different modifications from different plant origins are studied by producing a basic starch soup. Different parameters are then changed by adding other ingredients to the basic starch soup to ensure that the starch can fulfil all the requirements. The soup is packed with Tetra Recart's packages and inserted in a retort for sterilization. Finally, the soups are analysed with different methods and at different occasions in order to find the target starch.

Before the retort all starches, except for native waxy maize and potato acetylated starch, contributed with high viscosity to the basic starch soups. After retort and during storage time the potato and waxy maize starches with modification E1442 (by treating the starch with propyleneoxide and phosphoric acid) contributed with highest viscosity to the soups. As for the native potato starch, it breaks down after retort and the soup becomes very thin, i.e. the native potato starch is a filling agent starch.

Keywords: Starch, modified starch, viscosity, retort

1   Introduction

Starch has been widely used in different industries due to its low cost, biodegradability and renewability. It is used in paper, fuel (ethanol) and food industry. This rapport is focusing on the latter one. Starch is an important component in food industry; it contributes with energy for example in rice, wheat, maize etc. It is also significant in smaller doses to improve the properties of food such as viscosity, texture, thickness etc. However the starch granules hydrate easy, swell rapidly, lose viscosity, and produce weak bodies which create a very stringy and cohesive paste. Therefore most starches are modified to improve these properties. [1]

There are ready to eat soups and sauces in Tetra Recart packages. Tetra Recart package is based on paperboard used for food canning. Canning of soups and sauces is a convenient way of having industrial food in no time. A good canned viscose soup or sauce contains starch that contributes with the high viscosity. However, the problem is that the soups and the sauces can lose their viscosity after processing time or during storage time. Therefore, this study is focusing on finding a suitable starch that can fulfil the criteria. [2]

1.1   Aim

The aim of this study is to find a suitable starch that Tetra Recart can recommend for their customers. The target starch is one that can either be used as a filling agent in fillers or one that contributes with good viscosity and texture to the final product. The filling agent is a starch that is viscous before retorting and after retort the starch granules should be destroyed. The filling agent starch is good for eliminating two steps in the filling process which means only one step is necessary for pumping the particles and the sauce into the packages. It is important that the viscosity remains constant during and after storage time.

1.2   Method

The procedure of finding a suitable starch that fulfils all the requirements involves many steps. The first step is contacting different starch suppliers to get starch samples. Then the starch samples are studied by producing a basic soup where different parameters are changed to ensure that the starch can fulfil the requirements. The production of the soup is performed in a pilot hall and then they are packed with Tetra Recart's packages. The packages are inserted in a retort for sterilization. Finally, the soups are analysed with different methods before production and after having been stored.

2   Literature overview

The following chapter aims to give some theory for understanding this project and contains relevant background information about starch properties and processing of product.

2.1   Starch

Starch is a polysaccharide produced by green plants as a carbon and energy storage material and it is a very common carbohydrate in our food. The green plants contain starch which is produced by condensation polymerisation with glucose and an enzyme that can synthesise starch (figure 1). Starch contains two types of polymers: a linear molecule chain called amylose and a branched polymer called amylopectin. The fraction of amylose and amylopectin are depending on the plant, but generally starch consists of 15-30 % amylose. Amylose molecules have two types of helices; a single and double helix. The helix has a hydrophobic core due to the CH-groups and hydrophilic surface due to OH-groups. The double helix forms by two single helixes interacting with each other and stabilizes by van der Waals and hydrogen bonds. While, amylopectin is a highly branched polymer with α-D-glucopyranosyl units and it is amorphous at the branched regions. [1]

$$6H_2O + 6CO_2 \longrightarrow C_6H_{12}O_6 + 6O_2$$

$$nC_6H_{12}O_6 + enzyme \longrightarrow (C_6H_{12}O_5)_n + nH_2O$$

Figure 1: The reaction formula of starch. [1]

Figure 2 is the microscopic pictures of different starch granules. Potato starch has the largest grain in the microscope with its oval shape (figure 2a) and thereby the highest swelling capacity. Meanwhile, wheat starch has two different grains structures; small grain (1-10 μm) and bigger grain (20-45 μm) see figure 2C. Wheat starch is produced in many countries but as a by-product from production of wheat gluten. The consumption in the world of wheat starch is about 1 million tons per year. Wheat and potato starch are mostly used in Europe. [3]

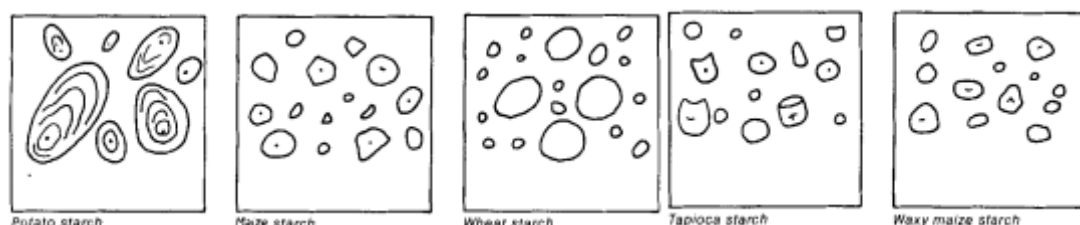Potato starch   Maize starch   Wheat starch   Tapioca starch   Waxy maize starch

Figure 2: Microscopic pictures of starch granules according to "AVEBE Stadex". [1]

Maize starch is mainly produced in the USA, around 50 % of the world production. It has medium sized grains, figure 2B, and a characteristic taste. Tapioca starch is produced from roots of tropic

plants called cassava or tapioca. In microscope the starch has round or angular shape, figure 2D. Tapioca starch is mainly produced in Thailand, Brazil, Malaysia, Philippines and Angola. The tapioca root starch is used for production of glue and food. Tapioca starch properties are similar to waxy starch. [3]

The waxy starch has different structure consist only amylopectin and no amylose, figure 2E. The amylopectin makes the starch gelatinisation easier than amylose. The gelatinisation process is when the starch becomes like paste and an increase of the viscosity occurs. Waxy starches are mainly produced in the USA from maize and potato by adding a hybrid of maize and potato, respectively. Waxy starch is mainly used in glue and as thickening agent in food industry. [1] [3]

Native Starch granules are insoluble in cold water due to hydrogen bonds between the hydroxyl group and the starch molecules. These hydrogen bonds are weak but many which make it difficult for water molecules to enter and dissolve the starch in water. At higher temperatures, 55°C to 80°C depending on which plant, the starch starts the procedure of gelatinisation which is increasing the viscosity in the mixture (figure 3). The gelatinisation point is when more hydration and irreversible expanding of the starch granules occur. The expanding occurs at the amorphous reigns, where the hydrogen bonds among the starch molecules are destroyed and the starch granules micelles absorb water and increase the viscosity. However, after the starch reaches the viscosity peak, figure 3, the granules are more destroyed and the viscosity starts to decrease. Therefore many starches are modified to keep the viscosity constant by delaying peak viscosity. Industrial starches are mainly made from potato, wheat, maize, tapioca and rice. The properties of the starch depends on the plant's origin. [1] [4]
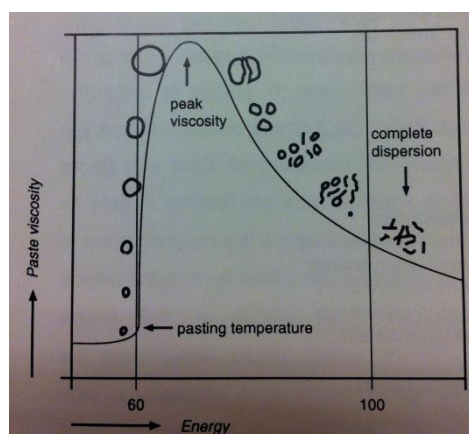

Figure 3: Swelling and dispersion of starch granules during gelatinization according to "AVEBE Stable food starches". [4]

Retrogradation of starch occurs when cooked starch is cooled; the starch paste thickens and sets rigid irreversible gel. The retrogradation reaction occurs in amylose molecule, mostly at double helix, by forming hydrogen bridges thereby retrograde and arrange to form crystalline structure. Amylopectin have branched molecules and thereby less tendency to retrogate. Modification reduces or enhances retrogradation deepens on required properties. [4]

2.2 Composition of starch

Table 1 presents the average values of starch components. The behaviour of the starch depends on these values. The moister content of the starch depends on how much the starch grains have absorbed water, relative humidity and temperature. Normally native starch contains 10-20% [1] of moister under regular atmospheric conditions. [3]

Table 1: The composition of native starch granules. [3]

Composition of Starch Granules (Average Values).

Starch Components	Potato starch	Maize starch	Wheat starch	Tapioca starch	Waxy maize starch
Moisture at 65% RH and 20°C.	19	13	13	13	13
Lipids (% on dry substance)	0.05	0.7	0.8	0.1	0.15
Proteins (% on d.s.)	0.06	0.35	0.4	0.1	0.25
Ash (% on d.s.)	0.4	0.1	0.2	0.2	0.1
Phosphorus (% on d.s.)	0.08	0.02	0.06	0.01	0.01
Amount of taste and odour substances	low	high relative	high relative	low	medium

Potato and tapioca starches contain the least amount of fatty substance (lipids); 0.05% and 0.1 % respectively, in comparison to other starches (maize, waxy and wheat) which contain 0.6 to 1.0 % of lipids (see table 1). The high amount of lipids reduces water binding capacity, swelling and stability of the starch. The lipids can also affect flavour and storage conditions of starch. Therefore potato and tapioca starches have the least flavour effect on the final product. [1] [3]

In cereal starches (i.e. maize, wheat, rice) the amount of proteins is 0.25% to 0.5% which is much higher comparing to potato (0.06%) and tapioca (0.1%). Proteins include peptides, amides, amino acids, enzymes and nucleic acids. The high amount of protein causes formation of a mealy flavour, foam and muddiness. [1] [3] Therefore when wheat and maize starches are dissolved in water they

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produce milky and un-diaphanous solution. In comparison to potato, tapioca and waxy maize starches they produce clear and transparent solution. [1]

Cereal starches contain almost no phosphor while the potato starch contains a very small amount of it (0.06%-0.1%). Even though it is a small amount of phosphor it makes the amylopectin cause repulsion of like charges in aqueous solutions. The phosphor groups can works as ion changers, which cause entanglement of polymer molecules and extend their sphere. [1] [3]

2.3   Production of starch

There are many different production ways of starch types, this part is focusing on maize starch production by wet milling. Maize starch is produced through wet milling from kernel; the techniques of the process include grinding, screening and centrifugation to separate purified starch from fiber, oil and protein, figure 4. First step is the softening of the kernel by diluting acid solution; the solution prevents fermentation and helps separation of the starch from protein. The kernel splits to remove oil containing germs by rough milling, then finer milling for separation of fiber from endosperm. The centrifugation is to separate less dense protein from starch. Finally, the starch is washed and dried and left in slurry for further processing. [5]
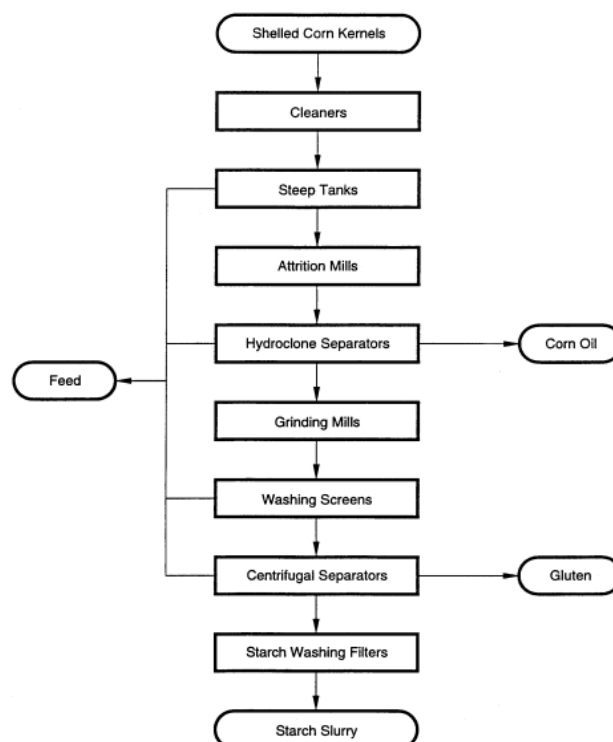

Figure 4: Schematic figure of wet milling process. [5]

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2.4   Modification of Starch

Native starches have limited use in food industry; the starch granules hydrate easy, swell rapidly, lose viscosity, and produce weak bodies which create very stringy and cohesive paste. However, modification of starch improves the inherent properties. The main modification methods are chemical, physical and enzymatical. [1]

2.4.1   Chemical modification

There are many different ways in chemical modification to improve the properties of the starch. Chemically, the modification occurs either by substituting the hydroxyl group in the starch chain or by breaking the chain into smaller fragments. The modification caused by the chemical substitution is measured by the "Degree of substitution" (DS). DS is the average value of substituted hydroxyl groups per glucose in starch chain. Each glucose can maximum bind to three hydroxyl groups which means DS can maximum be three. [6]

2.4.1.1   Acetylation

Acetylation, a chemical modification mechanism, is one of the most used in food industry, for example in baking, frozen food, sauces and baby food. The hydroxyl groups are replaced with acetyl groups (see figure 5) which create steric obstacles and make it difficult for the starch chain to form hydrogen bonds thus decrease the gelatinisation temperature. The acetylation reduces retrogradation and improves the stability at cooling and freezing points. [7]
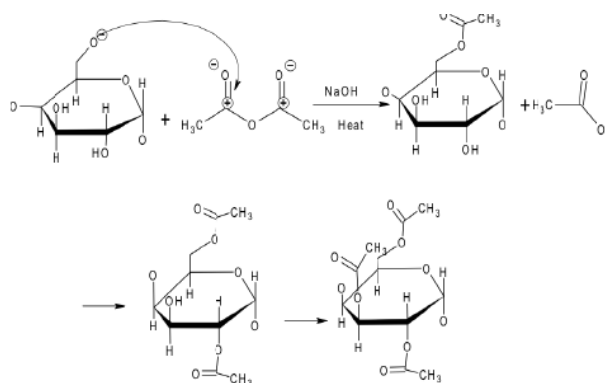

Figure 5: The mecanism of replacing hydroxylgroups according to the "Modification and Chemical Characterization of Barley Starch". [7]

2.4.1.2 Hydroxypropyl

The hydroxypropyl modified starch is also mostly used in food industry and has almost the same function as low acetylated starch, which was mentioned in previous paragraph. The modification occurs through reaction of the starch with propyloxide and base catalyst (NaOH), i.e. etherification through nucleophile substitution. The hydroxypropyl modification causes weaker interaction among the starch chains and decreases hydrogen bonds which makes it easier for the water molecule to assimilate. This function makes the starch granules absorb more water and swell before changing its structure. This modification improves the following properties: lower gelatinisation temperature, higher viscosity and reduction of retrogradation of starch. [8]

2.4.1.3 Acid/alkaline treatment

Acid treatment (E1401) is one of the oldest modification methods. The starch hydrolyzes by mixing with water and an acid. The acid hydrolyzes bonding between glucose molecules and then breaks down the starch chain into smaller fragments. However, the hydrolysis occurs mostly in the amorphous region which requires low heat temperatures. Thus, this modification is often used in frozen food. [9]

Alkaline treatment (E 1402) is referred to when starch granules are treated with an inorganic alkali such as sodium hydroxide and ethanol which is then neutralized with hydrochloric acid. This results in hydrolysis and negatively charged starch chain. Therefore the starch chain repulses and attracts sodium ions instead. This result in the starch granules absorbs water easily and has low gelatinisation temperature. [8] [9]

2.4.1.4 Oxidation

The production of oxidized starch occurs by reaction with an oxidation agent. The oxidation agent is usually sodium hypochlorite (NaCIO), but even NCO_2, ozone, hydrogen peroxide H_2O_2 and SO_2 can be used. The hydrogen groups in the starch chain are de-polymerized into carbonyl and carboxyl groups and this resulting in shorter polymer of the starch chain. The oxidation degree is higher at the amylose parts than at the amylopectin ones. [9] [10] [11]

Oxidized starch produce a homogenous gel with good retrogredation stability due to the carbonyl and carboxyl groups that create obstacles and prevent the starch molecule from rotating and changing the structure. However, the shorter polymer chain reduces the strength of the gel [8]. The

oxidized starch (E1404) is used in food industry as a replacement for acacia gum in candy and bread baking. [10] [11]

2.4.1.5   Stabilization

Stabilization is a modification method occurs by reaction of the starch chain with bulky groups. These groups stabilize the starch chain by take up space and create steric hinder, thereby prevent the starch chain from rotating. The stabilization effectiveness depends on the number and nature of the bulky groups. [8] The DS level is also important, as the level increases the interaction among the starch chain is increased, this resulting in hydration and gelatinisation at lower cooking temperatures and thereby loss of viscosity. Usually, stabilization is used in conjugation with crosslinking modification (see following paragraph). Stabilization is often used to prevent retrogradation and improve shelf-life of the starch. [1] [9]

2.4.1.6   Cross-linking modification

Cross-linking is an important way of strengthening the starch granules and it is performed by multifunctional reagents that can bind to hydroxyl groups through esterification or etherification. Cross-linked bonding is usually stronger than hydrogen bonds which make it easier for the starch molecule to keep its structure and increase tolerance to shear stress, high boiling temperatures and acid hydrolysis. However, cross-linking makes the starch granules more sensitive to retrogradation and therefore the cross-linking is often combined with phosphate reagents to stabilize and prevent retrogradation. Generally, as the number of cross-links increases, the starch becomes more resistant to gelatinisation. [12]

Di-starch phosphates (E1412) and Phosphorylated di-starch phosphate (E1413) are used in food industry, mostly in frozen food, puddings, sauce, etc. The starch is produced by esterification with $POCl_3$ at controlled environment; temperature 25 C and pH 10. [1] The solution is then neutralized and the starch granules are filtrated and dried. The PO_3 is not assimilated in the starch granules; it is instead dominated at the surface preventing the starch from expanding and changing its structure. [1]

Combining cross-linking and stabilization is a way of optimizing the properties of the starch. It decrees the swelling of the starch granules and improves the viscosity of the mixture. [1] Hydroxypropyl di-starch phosphate (E1442) and acetylated distarch adipate (E1422) are two examples of double modification which are normally used in food industry. [9]

2.4.2 Physical and enzymatic modification

Physical modification occurs by de-polymerization or trans-polymerization resulting in changing the chemical composition of the starch. In food industry the physical modification process is based on heat treatment, ultrasound treatment or high pressure treatment. Drum drying and extrusion are processes to produce pre-gelatinisation starch. Enzymatic treatment that is used in food industry can be performed with different enzymes such as endoamylase, exoamylase and trans fat. [13]

2.5 Processing the soup at Tetra Recart

Tetra Recart is the world´s largest carton base package company for canning of food. The carton base packaging is used in food industry instead of cans and glass jars, since it is more convenient due to light weight, efficient distribution and shelf storage and minimum environmental impact. With Tetra Recart's packages, vegetables, beans, sauces and soups can be packed and stored up till 24 months. [2]

The retort (autoclave) process can be generalized into three different phases: come-up, cook-up and cooling. The come-up is the time for the retort to reach the sterilization point, the cooking time is the time at the sterilization point and the last part of the process is the cooling phase where the product is cooled down to about 35 ° C, figure 6. The canning procedure of low acid food is based on high-pressure saturated steam at ~120 °C operating for 15 to 60 minutes to reach the cook-up phase. The cooking time depends on the size of the load and the viscosity of the product in the retort. When the product reaches the desired lethality (F-value) the retort is put into cooling temperature. Since food needs to be sterilized in order to eliminate spores, the steam in the retort is operating above 100 °C. The steam has high heat transmission coefficients compered to air and water. The starch soup is heated by convection where heat particles are transferred with fluids. Therefore the viscosity is very important for processing time, the higher the viscosity of the product, the longer the processing time for reaching required temperature. [14] During the retorting process a thin metal probe is inserted in one or two of the packages for measuring the temperature and F_0-value of the soup (figure 6). The F_0-value is a measurement of the lethality of the applied process; this is equivalent to the time at 121°C. When the product reaches sterilization value (F_0) at 6 then the retort sets on cooling to room temperature (~25°C). This value depends on the viscosity of the product, the more viscose the product, the longer time to reach the F_0-value. Figure 6 is a graph from a measurement in the retort, it shows the temperature curves of the retort and the F_0-value of the product. [15]
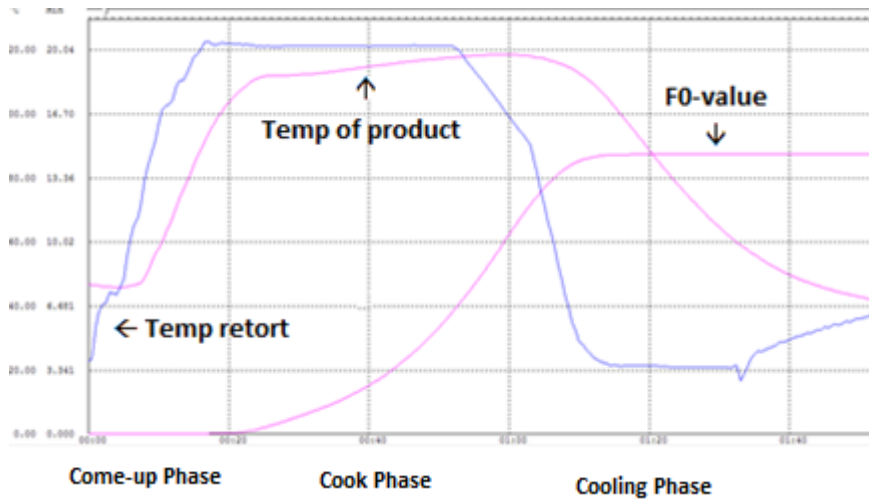
Come-up Phase   Cook Phase   Cooling Phase

Figure 6: A graph from retorting program, where the x-axis is the time and the y-axis is the temperature. The blue line is the temperature inside the retort while the pink line is the temperature of the product. The pink line that starts at the x-axis is the line of F-value. The F_0 –value is when the product have reaches the sterilization point. The three phases that products undergo during retort process are presented below the graph.

Figure 7 below is a schematic figure of Tetra Recart's packages. The Tetra Recart Junior version (J5) seals the packages. The package has longitudinal sealing (LS) on the backside of the package also on a transversal sealing (TS) on the bottom and on the top. The packages are sealed on the top first then they are filled with the product then sealed on bottom. [14]
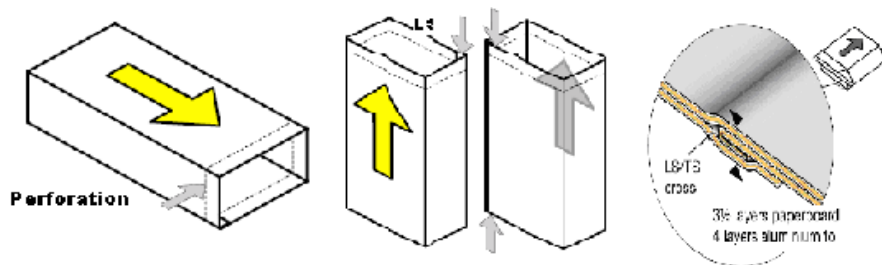

Figure 7: The carton package sealing procedure [14]

3 Starches from suppliers

The starch samples are from different suppliers, the suppliers are AVEBE, Culinar AB, Innovation (former National Starch) and Procudan. Table 2 present the starch samples from the four suppliers, as for the two starches that are marked with TRC are Tetra Recarts currently used starches. Table 3 is summary table of all starch samples with modification specification.

Table 2: Starch samples from suppliers

	Starch origin	Modification way
Starch A	Waxy maize	E 1442 (Hydroxypropyl di-starch phosphate)
Starch B	Tapioca	E 1442 (Hydroxypropyl di-starch phosphate)
Starch C	Waxy maize	Native
Starch D	Potato	E 1420 (Starch acetate)
Starch E (TRC)	Waxy maize	E 1422 (Acetylated di-starch adipate)
Starch F	Potato	E 1442 (Hydroxypropyl di-starch phosphate)
Starch G	Potato	E 1442 (Hydroxypropyl di-starch phosphate)
Starch H (TRC)	Potato	Native
Starch I	Potato	E 1442 (Hydroxypropyl di-starch phosphate)
Starch J	Waxy maize	E 1442 (Hydroxypropyl di-starch phosphate)
Starch K	Waxy maize	E 1422 (Acetylated di-starch adipate)

Table 3: Summary table of the starch samples with their modification specifications.

Starch origin	Modification way	Aim of modification
Waxy maize	Native E1422: treating the starch with acetic acid anhydride and adipic acid anhydride [16] E1442: treating starch with propyleneoxide and phosphoric acid [16]	E1422: resistant against stirring and high temperatures [16] E1442: more stable against acid, alkaline and starch degrading enzymes. It also provides better colour and shine to products and has very good freeze-thaw properties [16]
Potato	Native E1420: Prepared by treating starch with acetic acid anhydride [16] E1442: treating starch with propyleneoxide and phosphoric acid [16]	E1420: more stable at high temperatures and low pH [16] E1442: more stable against acid, alkaline and starch degrading enzymes. It also provides better colour and shine to products and has very good freeze-thaw properties [16]
Tapioca	E1442: treating starch with propyleneoxide and phosphoric acid [16]	E1442: more stable against acid, alkaline and starch degrading enzymes. It also provides better colour and shine to products and has very good freeze-thaw properties [16]

4 Experiment

The experiment of starch soup contains two parts; the first is preparation and production of the soup and the second is analysis of the product.

Different analysing methods were used in order to find the target starch: a filling agent starch or one for good final texture of soups, sauces etc. The following parameters were investigated: viscosity with and without applied shear rate with DV-E Brookfield viscometer, pH with Inolab PH Level 1 pH meter, the structure of the starch granules with a microscope and finally the flow of the soup with Bostwick instrument. These measurements were performed at different occasions: before the retorting process, one day after retort, two weeks after retort, etc. to have an overview of how starch behave over longer time period. Even appearance and taste of the starch soups were evaluated by a test method called sensory test.

4.1 Viscosity- With and without applied shear rate

Viscosity is the measure of a fluid's internal friction, which is the resistance of the flow. The greater the friction, the greater shearing, which is the amount of force required to cause this movement. Highly viscous materials need more force than low viscous ones. Viscosity measurement is performed by the Brookfield instrument; it measures the friction of liquids and semi-solids. Product quality and efficiency are often combined with viscosity measurements. The sample is inserted under the viscometer with a suitable spindle size; the higher the viscosity of the sample, the smaller the spindle used. [17]

The flow behaviour can be classified with combination of applied shear rate and viscosity. There are two main flow behaviours; Newtonian and non-Newtonian (see figure 8). The Newtonian fluid is constant viscosity with changed shear rate. The Non- Newtonian fluid is more complex, it is defined as none constant and the viscosity changes with respective shear rate. Some polymers and salt solutions have this behaviour. [18]The viscosity drag (resistance flow) is proportional to the spindle's speed of rotation and is related to the spindle's size and shape. The drag increases as the spindle size and/or rotational speed increases. The identification of liquid behaviour, when a stress is applied to it, is useful information for processing, formulating changes, aging etc. The applied stress can be used in processing for example pumping liquids in a machine; this information can be useful to set the required force for pumping the liquid. The measurement of viscosity with applied shear rate is also performed by the Brookfield instrument with same procedure as viscosity measurements but with smaller sample size (10-16 ml) and with changed speed of the spindle. [17]
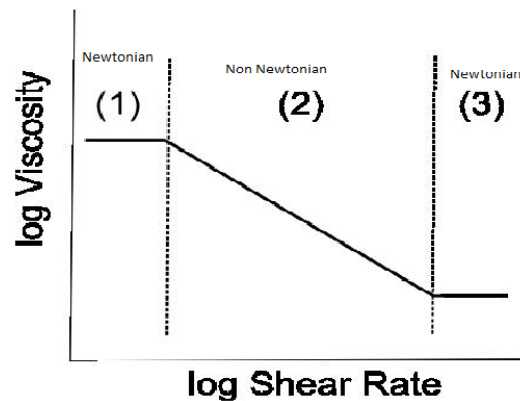
Figure 8: A graph of liquid behavior with shear rate. The y-axis is log viscosity and the x-axis is log shear rate. [19]

Many factors affect the rheological behaviour, for example temperature, time and pressure. Increasing temperature will decrease viscosity, some materials are more sensitive than other. Aging is a very common phenomenon during which there is change in viscosity; it especially occurs after retort process. Therefore it is important to control the measuring conditions. In this case the viscosity is investigated to find out how much the starch granules have expanded and aged before processing and during storage time. Also it is very important to be aware of the behaviour of the starch when a stress is applied to it. [19]

4.2 Bostwick

The texture of viscous material can be defined as the rate of the flow and it is measured with the Bostwick instrument (figure 9). The Bostwick can only be used for viscous material for example sauces, paint or cosmetics. The measurement procedure is very simple, first the sample is poured in the Bostwick instrument then the gate is opened and the timer is started as the sample runs along the axis. The further the sample runs along the slope the less viscous is the material. [20]

Figure 9: The Bostwick instrument measures the rate of the flow.

4.3 pH

The acidity affects starch conditions by disruption of the hydrogen bonds and thereby makes the starch granules swell easier and lose their structure easily resulting in loss of viscosity. [8] Therefore the pH was measured at each analysis occasion with Inolab PH Level 1 pH meter. The pH meter measures the hydrogen cat ions in the sample with glass electrode and the voltage of the sample is then displayed on the screen. The pH meter must calibrate with a known buffer solution before each measurement. [21]

4.4 Microscopy

The shape and the size of the starch granules can be determined with a microscope. The starch soups are analyzed with a microscope to see how much the granules have expanded or dried. The reaction of iodine with amylose and amylopectin creates characteristic blue and red colour respectively, this information is used to indicate amylose or amylopectin in the microscope. [1] [22]The analysis process is very simple; one drop of the starch soup and one drop of iodine solution are added on the object glass. Each starch type has its characteristic shape, size and color in the microscope. [22]

4.5 Sensory test

Sensory test is a very common analysis method that is used in food industry for evaluating the products. The test is performed by a test panel which is a group of people using their senses; in this case taste and sight, for the evaluation.

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In this report sensory tests were performed by two test panels; experts (food technologists) and non-experts. The test panels analyzed the appearance and evaluated the taste of the starch soups A-K. The data was collected by answering a questionnaire.

Production of the starch soup

The production of the starch soup with Tetra Recart's package is summarized in figure 10.
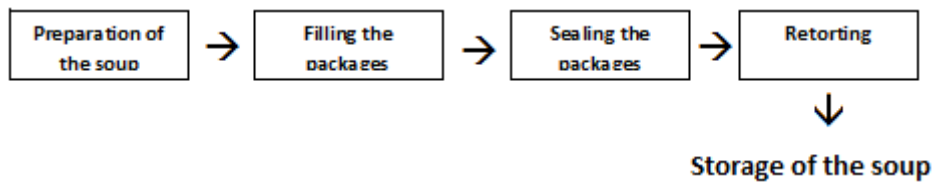

Figure 10: The production procedure of starch soup

The starch samples in table 2 were investigated by preparing a basic starch soup for each starch that contain simple ingredients which might affect the properties of the starch. The recipe of the soup with all the ingredients for the starch soup is in table4. The recipe was also modified by adding some ingredients to find out how they affect the texture, the viscosity and processing time of the product. Therefore, the following parameters were changed:

- Lower pH by adding citric acid to the sauce
- Higher dosage of starch, 8% (table 12, appendix)
- Adding citric acid and higher dosage (table 13, appendix)
- Mixing two different starches

Table 4: The recipe of the basic starch soup

Package content	
Ingredient	**Weight [g]/package**
Sauce	350
Diced carrots	40
Sauce Content	**[%] /Package**
Sunflower oil	2.69
Water	80.63
Salt	0.54
Sugar	0.9
Starch sample	3.59
Vegetable bouillion	1.35
Ascorbic acid	0.04

Preparation of the soup

For each starch 10-25 packages of starch soup were produced. The starch soup is heated before it is filled in the packages. First, all the ingredients for the sauce content are weighed according to the recipe (table 4). Then all the sauce ingredients are mixed and heated up except for the starch sample and 1L of water. The starch sample is mixed separately in a bowl with cold water to avoid lumps, when it is completely dissolved it is added to the sauce and heated up to pasteurizations temperature ($75^{o}C$). While the sauce is heating up, packages with 40 g of carrots each are prepared. Then each package is filled with 350 g of sauce and sealed with Junior (J5). After the sealing procedure the packages are inserted into AR092 JBT- autoclave for sterilisation and a probe is inserted into one or two of the packages to have an overview of the generation of the heat in the packages and of the F_0-value.

5 Result

This part presents the evaluation of the starch soups that were investigated. However, starch I was insoluble in cold water (figure 11) even though the amount of water was increased. Therefore starch I is not in the evaluation procedure.
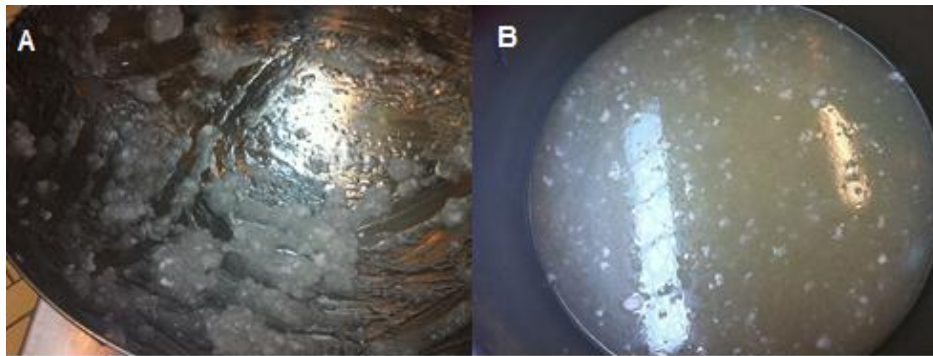

Figure 11: A) Starch I insoluble in cold water. B) Starch I insoluble in cold water with higher amount of water.

5.1 Viscosity, Bostwick and pH

All the starch samples were tested through their respective basic starch soup, recipe in table 4. Table 5 shows the results of the measurements (Bostwick, viscosity, pH and F_0 value) on the production day and after 15 weeks of storage. The packages were stored after the production day at room temperature (~23 °C) for 15 weeks.

Table 5: The results of basic starch soup from the production day and after 15 weeks of storage

| Starch soup | Bostwick [cm/15s] at 22 °C | Viscosity [mPas] at 22 °C | | pH | | F_0 Value [min] |
	15 weeks after retort	Before retort	15 weeks after retort	Before retort	15 weeks after retort	
Starch A	12	417	9 038	3.7	4.48	21
Starch B	17	720	938	3.95	4.62	14
Starch C	18	39	485	3.44	4.5	53
Starch D	too thin	25	8	3.98	4.45	15
Starch E	14.5	2 070	2 825	4.35	4.43	17
Starch F	13	50	5 705	3.99	4.7	14
Starch G	12.5	55 490	5 639	4.12	4.56	24
Starch H	too thin	2 327	20	4.16	4.58	14
Starch J	12.2	440	4 864	4.5	4.64	12
Starch K	14.5	1 409	2 120	4.24	4.46	15

Figure 12 shows the graph of the viscosity change as the shear rate increases after 15 weeks of storage. The graph presents the basic starch soups (A-K) except for starch soup D and H due to too thin to perform viscosity measurement with applied shear rate.
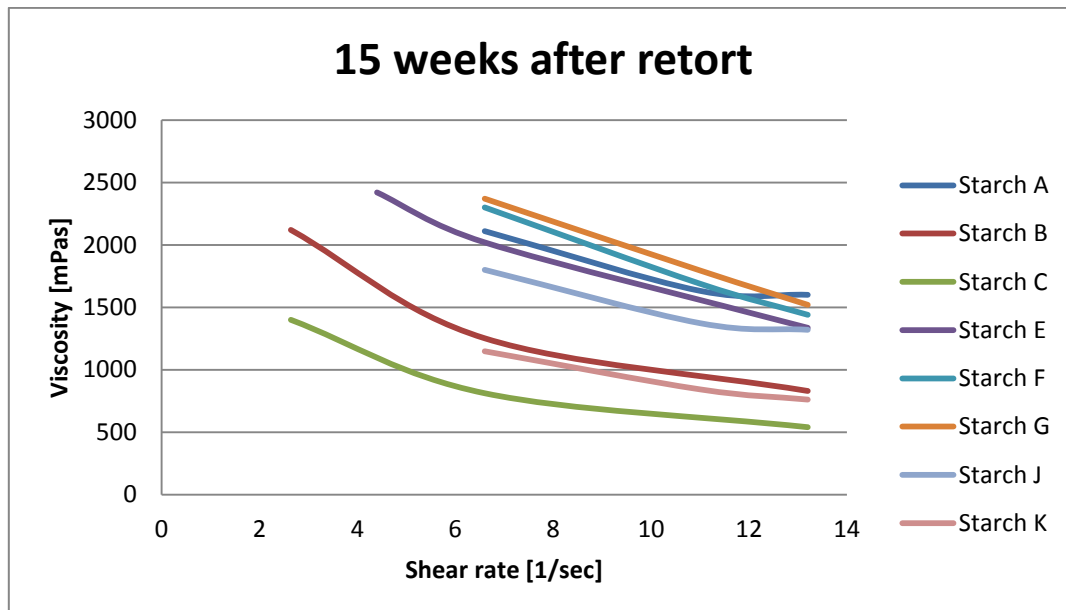

Figure 12: The relation between viscosity and shear rate for basic starch soups A-K (except for starch soups D and H), 15 weeks after retort. The y-axis is the viscosity and the x-axis is the shear rate.

Normally, the soups that are produced at Tetra Recart contain ~4% starch per package, in this test the amount of starch was doubled (8%). The starch samples (A-K) were tested through their respective starch soup with higher dose of starch (recipe in table 11, appendix). The measurements (Bostwick, viscosity, pH and F value) on the production day and after 6 weeks of storage are in table 6.

Table 6: The results of starch soups with higher dose of starch on the production day and 6 weeks after retort

Starch Soup	Bostwick [cm/15s] at 22 °C		Viscosity [mPas] at 22 °C		pH		F_0 Value [min]
	Before retort	6 weeks after retort	Before retort	6 weeks after retort	Before retort	6 weeks after retort	
Starch A	5.4	7.5	19 300	9 718	4.13	4.56	13
Starch B	3	too thin	99 500	67	4.01	4.43	17
Starch C	12.5	14.5	840	4 439	4.08	4.45	53
Starch D	too thin	too thin	45	9	4.06	4.5	15
Starch E	5.5	7	57 530	98 980	4.37	4.57	8
Starch F	4.5	11.5	99 080	17 800	4.19	4.69	16
Starch G	4	11.5	107 900	11 240	4.1	4.55	12
Starch H	7.5	too thin	147 200	38	4.28	4.43	20
Starch J	8	6.5	16 840	14 140	4.57	4.68	14
Starch K	7	8	5 600	8 368	4.4	4.6	12

Figure 13 shows the graph of the viscosity change as the shear rate increases after 6 weeks of storage. The graph presents all the starch soups with higher dose of starch except for starch soups D and H due to too low viscosity.
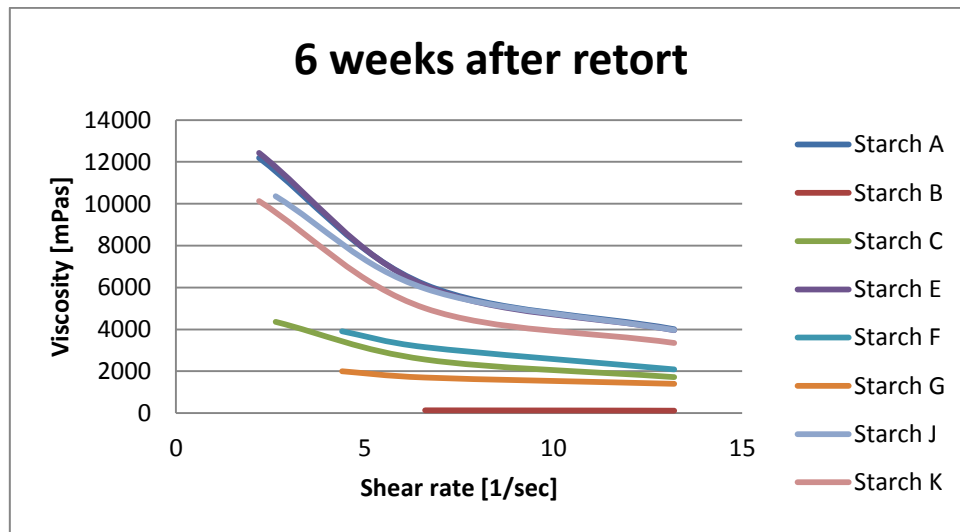

Figure 13: presents the relation between viscosity and shear rate for higher dose of starch soups A-K (except for starch D and H) 6 weeks after retort. The y-axis is the viscosity and the x-axis is the shear rate.

The starches A-H, except for D, were tested through their respective low pH starch soup by adding citric acid (table 13, appendix). Starch D was not in investigation process due to the low viscosity value in the basic soup recipe. As for the starches J and K the samples arrived after the investigation time. The results of the measurements (Bostwick, viscosity, pH and F value) from the production day and after one weeks of storage are presented in table 7. The low pH starch soups were not considered for further measurements due to low viscosity value. Since the low viscosity values of the soups were not able to perform viscosity measurements with applied share rate.

Table 7: The results of low pH starch soup on the production day and one week after retort

Starch soup	Bostwick [cm/15s] at 22 °C		Viscosity [mPas] at 22 °C		pH		F_0 Value [min]
	Before retort	one week after retort	Before retort	one week after retort	Before retort	one week after retort	
Starch A	11	too thin	6 366	17.3	2.83	3.3	28
Starch B	15	too thin	1 775	7.6	2.76	3.24	21
Starch C	21	too thin	222	7.4	2.84	3.36	12
Starch E	9.2	too thin	6 921	6.2	3	3.4	11
Starch F	8	too thin	18 700	8.7	2.87	3	20
Starch G	8	too thin	49 790	8.6	2.83	3.28	33
Starch H	16.5	too thin	821	5.5	2.91	3.2	19

Starch G and J contributed with good viscosity results in the basic soup and in the higher dose of starch soup. Therefore these two starches were investigated in lower pH and higher dose of starch soup. The aim from this investigation is to find out if the increased amount of starch can survive the low pH. Table 8 present the results of the measurements (Bostwick, viscosity, pH and F value) on the production day and after 5 weeks of storage. Unfortunately, the starch soups were too thin to perform viscosity measurements with applied shear rate.

Table 8: The results of higher dose of starch and low pH soup on the production day and 5 weeks after retort

Starch soup	Bostwick [cm/15s] at 22 °C		Viscosity [mPas] at 22 °C		pH		F_0 Value [min]
	Before retort	5 weeks after retort	Before retort	5 weeks after retort	Before retort	5weeks after retort	
Starch G	4.1	too thin	47 290	10	2.92	3.46	76
Starch J	6.5	too thin	2 945	6	3.11	3.66	28

Two different starch samples are mixed in same soup. The aim of this investigation is to find out if the starches with different properties can affect the final product. Table 9 present the measurements (Bostwick, viscosity, pH and F value) from the production day and after 5 weeks of storage.

Table 9: The results of mixing starch samples soup on the production day and 5 weeks after retort

Starch soup	Bostwick [cm/15s] at 22 °C		Viscosity [mPas] at 22 °C		pH		F_0 Value [min]
	Before retort	5 weeks after retort	Before retort	5 weeks after retort	Before retort	5 weeks after retort	
Starches H & F	9	too thin	18 920	70	4.13	4.62	16
Starches J & C	too thin	15	183	1 357	4.28	4.6	13

Figure 14 shows the graph of the viscosity change as the shear rate increases for starch soups with mixed starch sample.
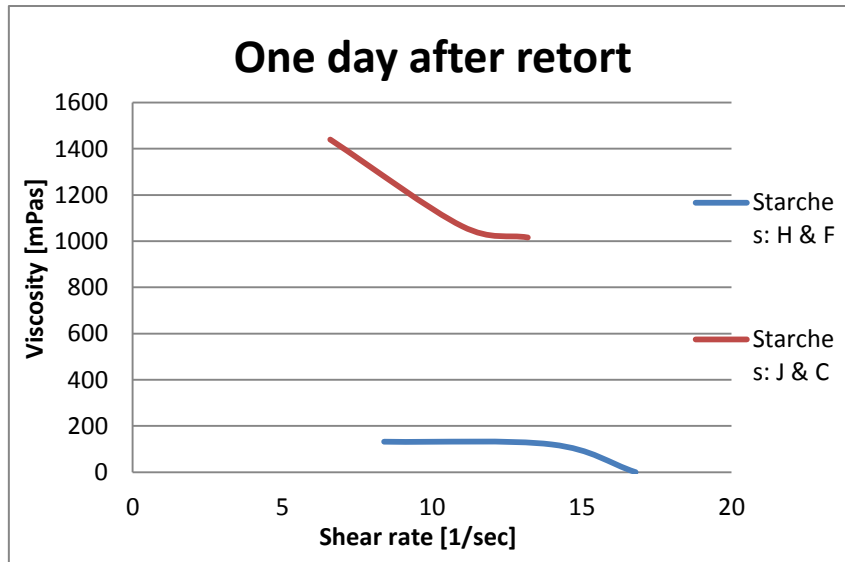
One day after retort

Figure 14: presents the relation between viscosity and shear rate for mix starch samples soup, one after retort. The y-axis is the viscosity and the x-axis is the shear rate.

5.2   Microscopy

All the starch soups (basic, higher dose of starch and low pH) were analysed with the microscope. The figures 15 and 16 present starch soups G and H. Starch soup G is one of the starches that contribute with good viscosity to the final product, therefore it shows an obvious structure of the starch granules in the microscope pictures. However, there is no trace in the microscopic pictures of the starch granules in figure16. Starch H is a filling starch; therefore the granules are destroyed after retort.
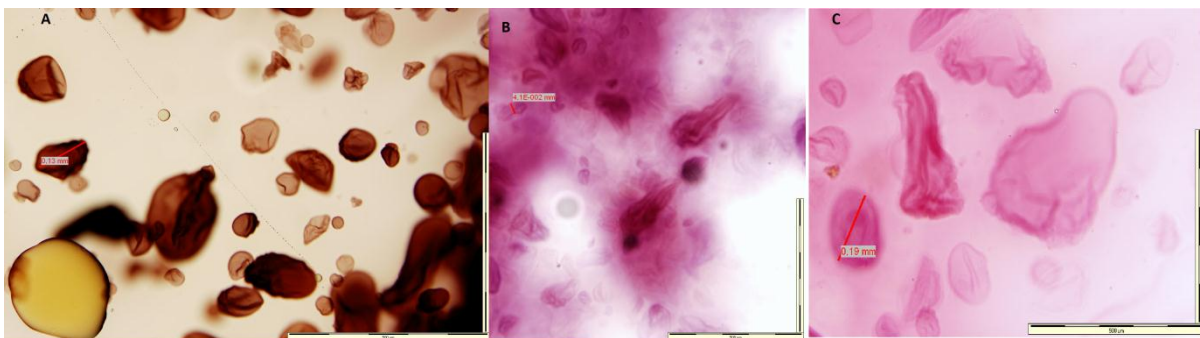

Figure 15: Microscopic pictures of the starch granules G. A) the basic starch soup G B) the starch soup with higher dose of starch C) The starch soup G with low pH
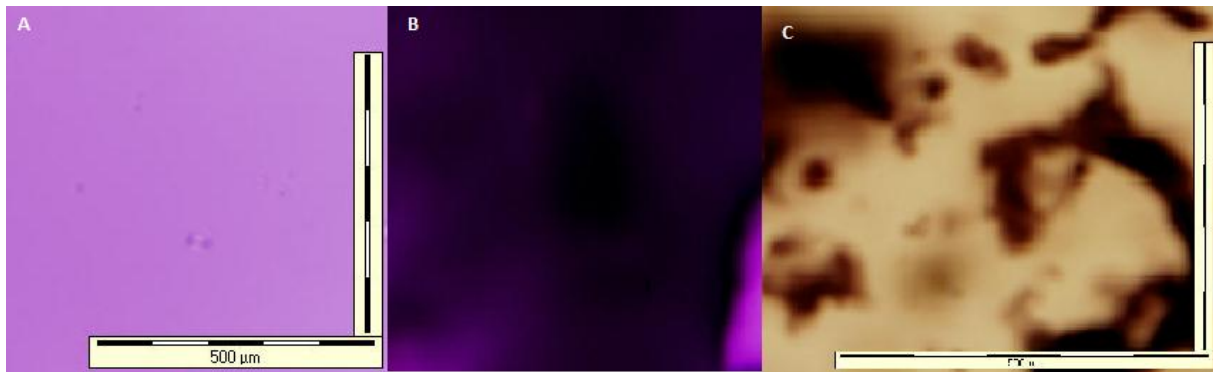
Figure 16: Microscopic pictures of the starch granules H. A) Low pH starch soup B) basic Starch soup C) higher dose starch soup

5.3 Sensory test

The evaluation of the sensory test was done by two panels: experts and non-experts. The panels evaluated the appearance and the taste of the basic starch soups A-K, recipe in table 3. Table 10 presents the highest results for appearance and taste while the lowest results are presented in table 11.

Table 10: Sensory test results for basic starch soups

Highest result for appearance		Highest results for taste	
Experts	**Non-experts**	**Experts**	**Non-experts**
Starch K	starch G	Starch G	Starch K
Starch F	Starch F	Starch E	Starch J
Starch J	Starch H	Starch J & K	Starch E

Table 11: Sensory test results for basic starch soups

Lowest result appearance		Lowest results taste	
Experts	**Non-experts**	**Experts**	**Non-experts**
Starch D	Starch J	Starch D	Starch C
Starch B	Starch C	Starch B	Starch D
Starch C	Starch D	Starch F	Starch B

6 Discussion

Before the retort all starches, except for native waxy maize and potato acetylated starch, contributed with high viscosity to the basic starch soups. After retort the native potato starch, breaks down and the soup becomes very thin, i.e. the native potato starch is a filling agent starch. While the native waxy maize starch is relatively thin before retort and increased slightly in viscosity after retort. After 15 weeks of storage the starch soups A, F and G have the highest viscosity. These starches are modified in the same way, i.e. E1442 by treating the starch with propylene oxide and phosphoric acid. The phosphoric acid makes the starch molecule anionic and thereby easier to react with water. While the propylenoxide stabilize the starch molecule by creating steric hinder to prevent the starch from rotating.

The E1420 potato starch (starch D) was very thin before and after retort. This starch is an acetylated starch with a CH_3CO-group. The acetyl groups work as obstacle on amylopectin to stabilize the chain. Since potato starch contain around 20 % amylopectin this modification type is mostly used in frozen food. The acetylated di-starch adipate (E1422) have similar modification to the acetylated potato starch but with adipic acid to stabilize pH. Since waxy maize starch contains 100 % amylopectin, it showed average results in viscosity and sensory measurements.

Figure 12 shows that all waxy maize and tapioca starches have exponential reduction in viscosity as the shear rate increases. In comparison, the potato starches have a straight reduction line in viscosity as the shear rate increases, figure 12. The slope of waxy maize and tapioca starches is much bigger than the one of potato starches.

With higher dose of starch, all starches, except for acetylated potato starch, contribute with high viscosity results before retort. After retort the waxy maize and potato starches with modification E1442 contributed with the highest viscosity, same as the basic starch soups. The tapioca starch, starch B, breaks down and becomes very thin and works as a filling agent when the dosage of starch is increased.

Figure 12 shows that waxy maize starches still have exponential reduction in viscosity but with bigger slope. As for potato starches, they still have straight reduction in viscosity but with much smaller slope. As the shear rate increases, the distribution of the viscosity values of the starches decreases.

None of these starches were able to withstand the low pH after retort, table 7. The starch granules were destroyed even though the dose of starch was increased, table 8. The medium cross-linking degree is the best modification method for starches to survive the acidity. However, this modification method is sensitive to high temperatures.

The mixture of native and modified potato starches contributed with high viscosity to the soup before retort and become very thin after retort. The properties of native potato starch dominated in this case. While the mixture of native and modified waxy maize starches contributed with relatively high viscosity after retort.

The evaluation of these soups is very individual, some people prefer soups with low viscosity and some prefer thick and high viscosity soups. According to the expert panel the modified waxy maize starches are best in taste and appearance. The non- expert panel considered the modified waxy maize starches also to be the best in taste, while modified potato starches are best in appearance. The starch soups that were the lowest in the evaluation are those with low viscosity. The modified tapioca starch got the lowest result in both taste and appearance by the panels. The tapioca starch has almost no taste due to the low composition values in protein and lipids.

7   Conclusion

In the basic starch soup and the high dose of starch soup the potato and waxy maize starches with modification E1442 gave the best results in viscosity, texture and sensory tests, also since the fluctuation in their values was very low during the measuring occasions.

The E1420 potato starch (starch D) is an acetylated starch with a CH_3CO-group. The acetyl groups work as obstacle on amylopectin to stabilize the chain. Since potato starch contain around 20 % amylopectin this modification type is mostly used in frozen food. The acetylated di-starch adipate (E1422) have similar modification to the acetylated potato starch but with adipic acid to stabilize pH. Since waxy maize starch contains 100 % amylopectin, it showed average results in viscosity and sensory measurements

The pH was increased for all starches after retorting. Since the soups were in the retort at high pressure steam and with oxygen the pH should decrease. In this case, the experimental results do not support the hypothesis.

The tapioca starch showed very interesting results when the dose of starch was increased. The starch granules were destroyed after retorting and the starch worked as a filling agent. Thus, the native potato starch is a filling agent which was shown in the experiments.

8   Suggestions for further work

There are several suggestions on how this study could be further expanded and continued. To start with, the stored soups should keep being analysed, perhaps after 6 months and after 1 year. Also, investigation of starch soups with lower dose of citric acid and finding the critical point before the starch granules are destroyed could result in interesting findings.

Acknowledgements

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Appendix

Package content	
Ingredient	**Weight [g]/package**
Sauce	350
Diced carrots	40
Sauce Content	**[%] /Package**
Sunflower oil	2.69
Water	77.04
Salt	0.54
Sugar	0.90
Starch sample	7.18
Vegetable bouillion	1.35
Ascorbic acid	0.04

Package content	
Ingredient	**Weight [g]/package**
Sauce	350
Diced carrots	40
Sauce Content	**[%] /Package**
Sunflower oil	2.69
Water	80.48
Salt	0.54
Sugar	0.90
Starch sample	3.59
Vegetable bouillion	1.35
Ascorbic acid	0.04
citric acid	0.15

Before retort				
Starch soup	Viscosity [mPas] at 22 °C	Brookfield Settings		pH
		Speed [RPM]	spindle	
Starch A	417	100	62	3.7
Starch B	720	100	62	3.95
Starch C	39	100	62	3.44
Starch D	25	100	61	3.98
Starch E	2 070	12	62	4.35
Starch F	50	100	62	3.99
Starch G	55 490	0.5	62	4.12
Starch H	2 327	12	62	4.16
Starch J	440	60	62	4.5
Starch K	1 409	100	62	4.24

One week after retort					
Starch soup	Bostwick [cm/15s] at 22 °C	Viscosity [mPas] at 22 °C	Brookfield Settings		pH
			Speed [RPM]	spindle	
Starch A	14	4 659	60	62	4.55
Starch B	16.5	1 270	20	62	4.6
Starch C	17.9	529	50	62	4.48
Starch D	too thin	12	100	62	4.48
Starch E	12.3	4 459	6	62	4.61
Starch F	15	5 423	5	62	4.66
Starch G	12.5	5 519	5	62	4.49
Starch H	too thin	22	100	62	4.64
Starch J	12.5	4 599	6	62	4.66
Starch K	14.5	1 414	20	62	4.56

6 weeks after retort					
Starch soup	Bostwick [cm/15s] at 22 °C	Viscosity [mPas] at 22 °C	Brookfield Settings		pH
			Speed [RPM]	spindle	
Starch A	10	5 750	5	62	4.57
Starch B	15.5	805	30	62	4.61
Starch C	17	404	60	62	4.52
Starch D	too thin	8	100	61	4.5
Starch E	14	2 384	12	62	4.61
Starch F	15.5	2 230	12	62	4.68
Starch G	15	2 936	10	62	4.62
Starch H	too thin	22	100	62	4.67
Starch J	12.2	4 864	6	62	4.64
Starch K	14.5	2 120	12	62	4.46

Table 17: Present the result of basic starch soup 15 weeks after retort. The table present the pH, viscosity and used setting of the viscometer, Brookfiled, for all starches.

15 weeks after retort					
Starch soup	Texture [cm/15s] at 22 °C	Viscosity [mPas] at 22 °C	Brookfield Settings		pH
			Speed [RPM]	spindle	
Starch A	12	9 038	3	62	4.48
Starch B	17	938	30	62	4.62
Starch C	18	485	60	62	4.5
Starch D	Too thin	8	100	61	4.45
Starch E	14.5	2 825	10	62	4.43
Starch F	13	5 705	5	62	4.7
Starch G	12.5	5 639	5	62	4.56
Starch H	too thin	20	100	61	4.58

Table 18: Present the result of low pH starch soup before retort. The table present the pH, viscosity and used setting of the viscometer, Brookfiled, for all starches.

Analyse1: Before retort					
Starch soup	Texture[cm/15s] at 22 °C	Viscosity [mPas] at 22 °C	Brookfield Settings		
			Speed [RPM]	spindle	pH
Starch A	11	6 366	4	62	2.83
Starch B	15	1 775	12	62	2.76
Starch C	21	222	100	62	2.84
Starch E	9.2	6 921	4	62	3
Starch F	8	18 700	1.5	62	2.87
Starch G	8	49 790	0.6	62	2.83
Starch H	16.5	821	30	62	2.91

Table 19: Present the result of low pH starch soup one day after retort. The table present the pH, viscosity and used setting of the viscometer, Brookfiled, for all starches.

One day after retort					
Starch soup	Texture [cm/15s] at 22 °C	Viscosity [mPas] at 22 °C	Brookfield Settings		
			Speed [RPM]	spindle	pH
Starch A	too thin	13	100	61	3.37
Starch B	too thin	6.5	100	61	3.61
Starch C	too thin	7	100	61	3.6
Starch E	too thin	9	100	61	3.67
Starch F	too thin	6.8	100	61	3.56
Starch G	too thin	9	100	61	3.88
Starch H	too thin	6	100	61	3.76

Table 20: Present the result of low pH starch soup one week retort. The table present the pH, viscosity and used setting of the viscometer, Brookfiled, for all starches.

Analyse 3: one week after retort			Brookfield Settings		
Starch soup	Bostwick [cm/15s] at 22 °C	Viscosity [mPas] at 22 °C	Speed [RPM]	spindle	pH
Starch A	too thin	17.3	100	61	3.3
Starch B	too thin	7.6	100	61	3.24
Starch C	too thin	7.4	100	61	3.36
Starch E	too thin	6.2	100	61	3.4
Starch F	too thin	8.7	100	61	3
Starch G	too thin	8.6	100	61	3.28
Starch H	too thin	5.5	100	61	

Table 21: Present the result of higher dose of starch, starch soup before retort. The table present the pH, viscosity and used setting of the viscometer, Brookfiled, for all starches.

Before retort			Brookfield Settings		
Starch soup	Bostwick [cm/15s] at 22 °C	Viscosity [mPas] at 22 °C	Speed [RPM]	spindle	pH
Starch A	5.4	19 300	6	63	4.13
Starch B	3	99 500	5	64	4.01
Starch C	12.5	840	60	25	4.08
Starch D	too thin	45	60	31	4.06
Starch E	5.5	57 530	0.5	62	4.37
Starch F	4.5	99 080	0.3	62	4.19
Starch G	4	107 900	1	63	4.1
Starch H	7.5	147 200	0.3	63	4.28
Starch J		16 840	6	63	4.57
Starch K		5 600	20	63	4.4

Table 22: Present the result of higher dose of starch, starch soup two weeks after retort. The table present the pH, viscosity and used setting of the viscometer, Brookfiled, for all starches.

Two weeks after retort			Brookfield Settings		
Starch soup	Bostwick [cm/15s] at 22 °C	Viscosity [mPas] at 22 °C	Speed [RPM]	spindle	pH
Starch A	7.3	18 520	1.5	62	4.54
Starch B	Too thin	160	100	62	4.48
Starch C	14	150	100	62	4.6
Starch D	Too thin	16	100	62	4.54
Starch E	7.8	5 675	20	63	4.63
Starch F	12.5	1 940	60	63	4.68
Starch G	12	3 475	30	63	4.61
Starch H	Too thin	42	100	61	4.65
Starch J	7.5	9 168	30	63	4.68

| Starch K | 7.5 | 9 310 | 30 | 63 | 4.55 |

6 weeks after retort					
			Brookfield Settings		
Starch soup	**Bostwick [cm/15s] at 22 °C**	**Viscosity [mPas] at 22 °C**	**Speed [RPM]**	**spindle**	**pH**
Starch A	7.5	9 718	12	63	4.56
Starch B	too thin	67	100	62	4.43
Starch C	14.5	4 439	6	62	4.45
Starch D	too thin	9	100	61	4.5
Starch E	7	98 980	0.3	62	4.57
Starch F	11.5	17 800	1.5	62	4.69
Starch G	11.5	11 240	2.4	62	4.55
Starch H	too thin	38	100	62	4.43
Starch J	6.5	14 140	6	63	4.68
Starch K	8	8 368	12	63	4.6

			Brookfield Settings		
	Bostwick [cm/15s] at 22 °C	**Viscosity [mPas] at 22 °C**	**Speed [RPM]**	**spindle**	**pH**
Before retort					
Starch G	4.1	47 290	12	64	2.92
Starch J	6.5	2 945	100	64	3.11
One day after retort					
Starch G	too thin	8	100	61	2.99
Starch J	too thin	8	100	61	3.5
One week after retort					
Starch G	too thin	7.14	100	61	3.57
Starch J	too thin	10.2	100	61	
Four weeks after retort					
Starch G	too thin	10	100	61	3.46
Starch J	too thin	6	100	61	3.66

Table 25: is the result of mixing two starches, starch soup. The table present the pH, viscosity and used setting of the viscometer, Brookfiled, for all starches.

| Starch soup	Bostwick [cm/15s] at 22 °C	Viscosity [mPas] at 22 °C	Brookfield Settings		pH
			Speed [RPM]	spindle	
Before retort					
Starches H & F	9	18 920	1.5	62	4.13
Starches J & C	Too thin	183	100	62	4.28
One day after retor					
Starches H & F	Too thin	92	100	62	4.22
Starches J & C	15	2 145	12	62	4.43
One week retort					
Starches H & F	too thin	85	60	61	4.6
Starches J & C	15	1 477	20	62	4.65
five weeks after retort					
Starches H & F	too thin	70	100	62	4.62
Starches J & C	15	1357	10	62	4.6
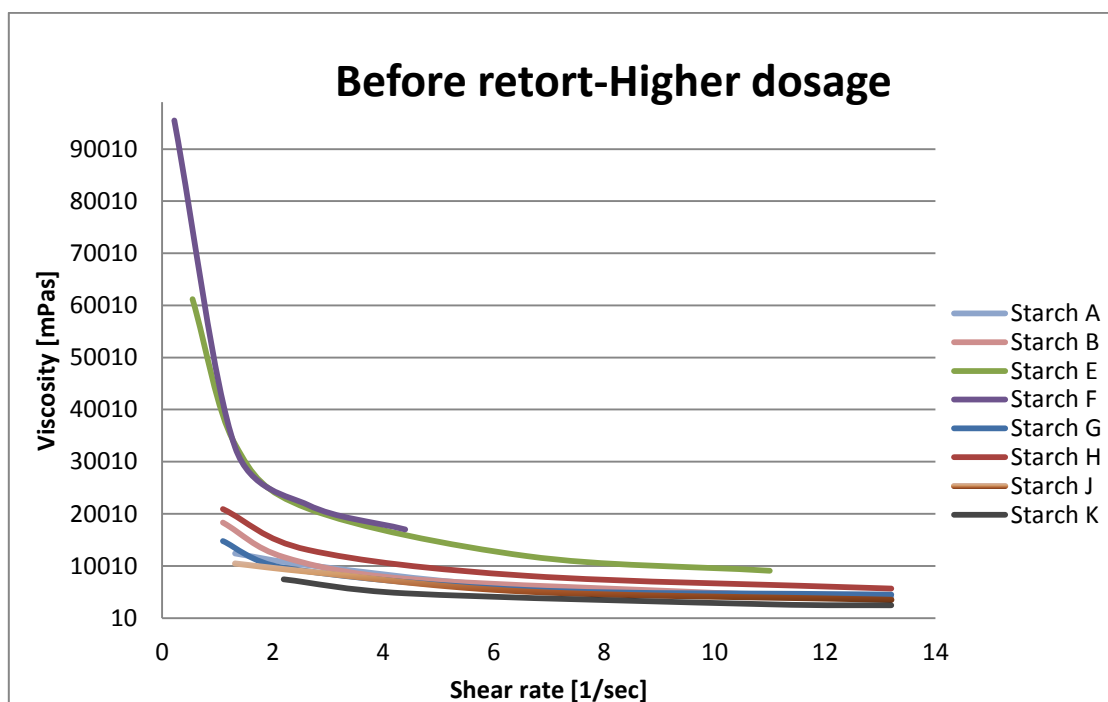
Figure 17: the relation between viscosity and shear rate before retort for high dose of starch soup. On the y-axis is the viscosity and x-axis is the shear rate.