Mayonnaise

Quality and Catastrophic Phase Inversion

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Abstract

In industrial production of mayonnaise it is important to both get a product of high quality and to avoid phase inversion. To investigate how the quality and phase inversion point are affected by changes in the production, a high shear mixer and an immersion blender were used to make mayonnaise. The stirring speed, temperature, egg content, egg type and oil content were changed to see how the quality of the mayonnaise was affected. The maximum oil flow rate that could be used without getting a phase inversion was investigated where changes in stirring speed, temperature, egg content, egg type and addition of the oil was made.

High quality mayonnaise has a firm texture and a small droplet size. Mayonnaise reaches a peak in texture and a minimum in droplet size during mixing. At longer mixing times the quality decreases and the mayonnaise becomes over-sheared. When the ingredients are cold and a high amount of egg yolk is used mayonnaise with a high quality can be made. Mayonnaise with an increased oil content has a firmer texture but is also more sensitive to over-shear.

Phase inversion occurs in two different ways: when the mixing is prolonged and when the oil is added too fast. Mayonnaise made with increased oil content undergoes phase inversion after prolonged mixing. Traditional phase inversion of mayonnaise occurs when the oil is added too fast which means that there is a maximum flow rate. It was found that the stirring speed does not affect the maximum flow rate that could be used. Changes in temperature and egg content only gave small changes while changing the egg type to egg yolk powder required a very low oil flow rate. The information obtained in this master thesis can be used to make mayonnaise of high quality where phase inversions can be avoided.

Preface

This master thesis was a collaboration between Tetra Pak Processing Systems and the Department of Food Technology, Engineering and Nutrition at Lund University faculty of Engineering (LTH). The duration of this master thesis was from January to June 2017. The aim of the master thesis was to investigate phase inversion and quality in mayonnaise.

We wish to thank our supervisors from Tetra Pak Prof. Fredrik Innings and Dr. Dragana Arlov and from LTH Prof. Björn Bergenståhl for all the support and encouragement. We also want to thank Dan Johansson from LTH and Ingrid Svedberg from Tetra Pak for all practical help in this thesis.

Contents

1 Introduction

Mayonnaise is a pale yellow sauce with a thick and creamy texture. Mayonnaise can be divided into two main types depending on the amount of oil that is used in the recipe. Low-fat mayonnaise contain around 30-65% oil while full-fat mayonnaise contains around 75-80%. In this thesis only full-fat mayonnaise was investigated. The oil is dispersed in a water phase to form an oil-in-water (O/W) emulsion. The ingredients that can be found in the water phase are: egg, vinegar, salt, sugar, mustard and water. The oil-water interfaces are stabilized by egg yolk which contains emulsifying agents.

In order to form the O/W emulsion the oil needs to be broken up into small droplets. This is achieved by applying high amount of energy in the form of stirring. Generally the more energy that is applied the smaller the droplets become and smaller droplets makes the emulsion more stable.

When making mayonnaise at home there is always a risk of a phase inversion, resulting in a water-in-oil (W/O) emulsion. Phase inversed mayonnaise, or broken mayonnaise, is characterized by a low viscosity which is close to the viscosity of oil. Every mayonnaise maker has their own tips and tricks for preventing this phenomenon, including everything from whipping technique to the temperature of the ingredients. The best tip according to both Jamie Oliver and Martha Stewart is to pour the oil in slowly (Oliver, 2013; Stewart, 2017). The problem with getting a broken mayonnaise can also occur in the industry, where a high shear mixer is used. Not much has been investigated when it comes to phase inversion of mayonnaise made in industrial scale. Information found in cookbooks is therefore sometimes used also in the industry and it will be the starting point of this master thesis as well.

1.1 Objective

The objective of this master thesis is to investigate how different parameters in full-fat mayonnaise production affect the formation and breakage of the emulsion. This is done by using a Tetra Pak High Shear Mixer, Pilot unit B200-100VA and an immersion blender. The results will be evaluated by measuring the texture and droplet size of the resulting mayonnaise. Mayonnaise that has undergone a phase inversion cannot be measured but information concerning when and how the phase inversion occurred will be noted. The following parameters are believed to have an impact on phase inversion of mayonnaise:

- Rotor tip speed during oil addition
- Oil inlet flow rate
- Amount and type of emulsifier
- Temperature of the ingredients
- Oil content

Furthermore the texture and droplet size of mayonnaise will be investigated by changing the emulsifier, emulsification tip speed, temperature and oil content.

2 Background

2.1 Basics of mayonnaise

Mayonnaise is an oil-in-water (O/W) emulsion with a dispersed oil phase and a continuous water phase containing egg, vinegar, salt, sugar, mustard and water. The oil-water interface is stabilized by egg yolk, which acts as an emulsifier. The oil content in mayonnaise can vary to give the original (full-fat) mayonnaise or low-fat mayonnaise. In full-fat mayonnaise the oil content is around 75-80% while the oil content in low-fat mayonnaise is around 30-65%. This master thesis will only focus on full fat mayonnaise. In this master thesis mayonnaise with high quality is characterized by a firm texture and a small droplet size.

Industrially made mayonnaise is often produced in a high shear rotor-stator mixer where the ingredients of the water phase are mixed together first to get a homogenous mix. The mixing process can then be divided into two steps: coarse emulsion and emulsification. The coarse emulsion is when the oil is added to the water phase at a controlled rate to get a coarse O/W emulsion. When all the oil has been incorporated into the emulsion the vinegar is added. The emulsification is then initialized and the increased shear introduced in this step reduces the droplet size of the existing emulsion, making it more stable. Not much research concerning how phase inversion is avoided has been done in industrial settings.

Kitchen-made mayonnaise can either be done by hand or by using an immersion blender. Both methods only include a coarse emulsion step, which is the step where the oil is added. When whisking by hand everything but the oil and vinegar are mixed before the oil is added slowly. After half of the oil has been incorporated into the emulsion the vinegar is added. The remaining oil can then be added a bit more rapidly and the mayonnaise is whisked until the preferred consistency is reached. When mayonnaise is made by using an immersion blender all ingredients are added to a high, narrow bowl. The oil, with its lower density, will be on top of the other ingredients. The immersion blender is put in the water phase at the bottom of the bowl and turned on. The blender is then moved around at the bottom and the oil on top will be dragged down, forming mayonnaise. When mayonnaise has been formed in nearly the entire bowl the blender can be lifted slowly to incorporate all the remaining oil.

Mayonnaise has a pale yellow color and the high fraction of dispersed phase makes it a highly viscous condiment. However, because of the high fraction of dispersed phase there is a risk of phase inversion, where the mayonnaise becomes broken. A broken mayonnaise is easy to distinguish from normal mayonnaise because of the very low viscosity (similar to the viscosity of oil) and darker yellow color. The differences between normal- and broken mayonnaise can be seen in Figure 2.1.

Figure 2.1: Left: picture of normal mayonnaise. Right: picture of broken mayonnaise.

Phase inversion of mayonnaise can occur either during the coarse emulsion step or the emulsification step. In this report the different phase inversion types are referred to as coarse emulsion phase inversion and emulsification phase inversion.

A coarse emulsion phase inversion is characterized by a very early phase inversion and as a result no mayonnaise is ever formed. As this type of inversion takes place during the coarse emulsion it is very sensitive to the oil addition rate. A low oil addition rate is therefore recommended in order to avoid getting broken mayonnaise. Since the coarse emulsion step is present in both kitchen-made and industrial production of mayonnaise, coarse emulsion phase inversion can occur in both of these production methods. In recipes for making kitchen-made mayonnaise it is stated that adding the oil slowly is crucial, especially in the beginning, in order to avoid phase inversion (Oliver, 2013; Smith, 2017). Other recommendations are to use ingredients with the same temperature (Ica Förlaget, 1995) and to keep the immersion blender in the bottom of the bowl.

An emulsification phase inversion is characterized by a formation of mayonnaise that is then broken during the emulsification step. As this type of phase inversion takes place after all the oil has been incorporated, the addition rate of oil is not of importance. Instead the emulsification phase inversion is a result of merging oil droplets to a point where the inversion occurs. Considering that kitchen-made mayonnaise does not include an emulsification step this type of phase inversion is not observed in this production method. Not much research concerning the avoidance of a phase inversion has been done in industrial settings and instead recommendations from cookbooks are used. Since kitchen-made mayonnaise is only at risk for coarse emulsion phase inversion this type of inversion has been investigated. However, emulsification phase inversion is only observed in industrially produced mayonnaise and is therefore relatively unexplored.

2.2 Ingredients

Mayonnaise is an O/W emulsion with an oil phase and an aqueous phase which consists of egg, vinegar, salt, sugar, mustard and water. However, mayonnaise from all over the world can have varying ingredients depending on the country of origin. Differences can be seen in choices like type of oil or emulsifier. The oil type can be changed because of economical reasons while different spices can be used to reflect the culture. The emulsifier used can vary between whole egg (common in USA), liquid egg yolk (common in Europe) and spray-dried egg yolk (common in Russia) (Cedergårdh, 2014).

2.2.1 Oil

Oil is the main ingredient in mayonnaise and therefore it has a large influence on the quality of the final product. The amount of oil dispersed in the mayonnaise contributes to the visco-elastic behavior, stability and high viscosity of the product. The oil also impacts the organoleptic properties by providing creaminess and flavor to the mayonnaise. It is therefore important to use an oil with neutral taste, like rapeseed oil, sunflower oil or grapeseed oil. The smooth texture and appearance is also dependent on the amount of oil present in the product. (McClements and Demetriades, 1998)

2.2.2 Egg

2.2.2.1 Composition of egg

Eggs are composed of three main parts, egg white (59%), egg yolk (31%) and the outer shell (10%) (Nys and Guyot, 2011). However, in mayonnaise only egg yolk and egg white are used as an emulsifying agent. Egg yolk is more commonly used compared to egg white because of its greater emulsifier properties.

The egg white or albumen is composed of water, protein, glucose and some minerals. The main part of albumen is water, 84-89% (Nys and Guyot, 2011), while the remaining part is dominated by globular glycoproteins (Li-Chan and Kim, 2008).

Egg yolk only contains 50% water and therefore has considerably higher dry matter content compared to albumen. The remaining 50% in egg yolk consists of lipids (33%), proteins (15%), carbohydrates (1%) and minerals (1%) (Li-Chan et al., 1995). To make all lipids soluble they are bound to proteins, or apoproteins, to form lipoproteins, see Figure 2.2. These structures can be divided into low-density lipoproteins (LDL) and high-density lipoproteins (HDL). LDL have a spherical shape with a diameter of around 35 nm and contain a higher amount of lipids (90%) than HDL (20%) and consequently have a lower density (Anton, 2007a; Li-Chan and Kim, 2008). HDL lacks the spherical shape that LDL has and instead resembles the structure of globular proteins. The structure of HDL is smaller than LDL, 7-20 nm, and it is also more rigid. (Anton, 2007a; Anton, 2007b)

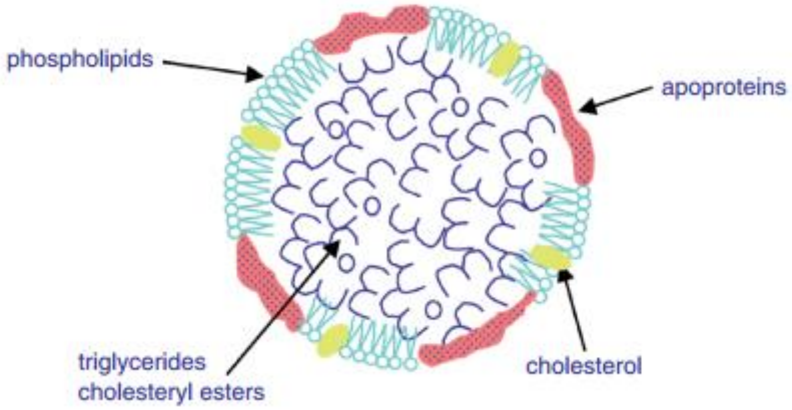

Figure 2.2: Suggested structure of Low-density-lipoprotein (LDL) (Anton, 2007a).

The standard way to classify egg yolk is by how it is separated during centrifugation, where the two fractions are plasma (supernatant) and granules (sediment). The plasma is the larger of the two fractions, responsible for 78% of the total egg yolk (Li-Chan et al., 1995). This fraction contains LDL and livetins (Anton, 2007a). The granules contain HDL, phosvitin and LDLg where LDLg is a smaller portion of LDL situated in the granules (Stadelman and Cotterill, 1995).

2.2.2.2 Emulsifying and stabilizing properties of egg

Egg yolk is the main emulsifying agent in egg because while egg components in egg white also show some emulsifying ability, egg yolk is four times as effective (Bergquist, 1995). The emulsifying properties of egg yolk is mainly determined by the amount of LDL, livetins and to some extent the granules and phosvitin (Anton, 2013; Vincent et al., 1966). Both the LDL and livetins can be found in the plasma and are soluble under nearly all conditions. Granules and phosvitin are complex bound through phosphocalcic bridges and consequently, this fraction is only soluble under certain conditions, such as natural pH and high ionic strength (Anton, 2013). Solubility of the emulsifier is of great importance since it needs to be soluble in order to get to and interact with the oil-water interface. Emulsifying properties of different substances are often measured as the decrease in interfacial tension. By comparing the decrease in interfacial tension between egg yolk (containing both granules and plasma) and only plasma it has been found that the contribution from granules was negligible (Vincent et al., 1966).

LDL is often mentioned as the primary reason for the excellent emulsifying properties of plasma, even though livetins also have been shown to be somewhat surface active (Vincent et al., 1966). This is mostly believed to be because of the relatively large fraction of LDL in plasma (85%) relative to livetins (15%). The adsorption mechanism for LDL has therefore been investigated thoroughly while the adsorption mechanism for livetins has been less investigated. The adsorption mechanism of LDL at an interface is characterized by a breakdown of the original structure, which is illustrated in Figure 2.3. The breakdown is initialized by anchoring of the outer apoprotein layer to the interface. Consequently, the structure is disrupted and the proteins start to spread along the surface (Martinet et al., 2003). The lipid core is then released and there may also be coalescence with the oil phase (Matsumura and Matsumiya, 2012). It is generally believed that only the first layer of LDL, which is in direct contact with the oil, unfolds completely. Consequent layers of LDL might only be partly disrupted or even retain the original structure (Kiosseoglou and Sherman, 1983). The surface load of egg yolk at the oil-water interface is around 1-3 mg/m^2 and is dependent on both salinity and pH (Le Denmat et al., 2000).

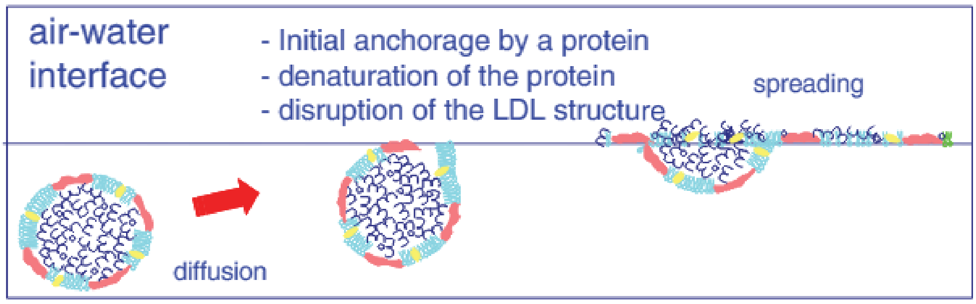

Figure 2.3: Adsorption mechanism for LDL which is the same for both air-water and oil-water interface (Anton, 2013).

The stability of an emulsion is determined by its ability to withstand destabilizing mechanisms like creaming, flocculation and coalescence. The stability of egg yolk emulsions is mainly provided by the granules (which contain HDL, phosvitin, LDLg) (Anton and Gandemer, 1997). This fraction contains more proteins than plasma which can contribute to a thick layer of protein on the droplet interface. This ensures the stability of an emulsion both by introducing more electrostatic- and steric repulsion and by allowing cross-linking between droplets (Anton and Gandemer, 1997; Ford et al., 1997). With plasma the emulsifying properties are very good but plasma alone lacks the stabilizing properties of an egg yolk emulsion. This is thought to be a consequence of the relatively small amount of protein available in the plasma fraction, since LDL only contains 10% apoprotein (Anton and Gandemer, 1997; Li-Chan and Kim, 2008). With less protein available, cross-linking between droplets is unlikely to occur and the emulsion becomes less stable when only plasma is used.

Liquid egg yolk can be spray-dried to improve the shelf-life but processing egg yolk in this way results in some protein denaturation. This influences the quality of mayonnaise and gives a more firm texture. (Guerrero and Ball, 1994)

2.2.3 Vinegar

Vinegar used in mayonnaise contributes to the flavor of the mayonnaise and it decreases the pH. By keeping the pH of the product low, the microbiological safety and preservation of the product increases. The low pH used in mayonnaise is close to the isoelectric point of the proteins from the egg yolk. The few charges on the proteins makes it possible for the proteins to be closer to each other and the droplets can be packed more tightly. (Duncan, 2004)

2.2.4 Salt and sugar

Salt contributes to the flavor and to the stability of the mayonnaise (Depree and Savage 2001). Salt helps to neutralize the charges of the proteins so they can adsorb more efficiently to the droplet interface. The more neutral droplet interface decreases the electrostatic repulsion between the droplets which induces flocculation. As a result of more flocculation, the packing of the mayonnaise becomes tighter and the viscosity increases. Sugar contributes to the flavor of the mayonnaise and is added mainly to counteract the flavor of vinegar. (Duncan, 2004)

2.2.5 Mustard

Mustard contributes to the flavor and color of mayonnaise. Most of the flavor in mustard comes from the isothiocyanates. The acid in the mayonnaise stabilizes these flavor compounds. (Depree and Savage, 2001) The mustard used in mayonnaise can be added as mustard flour instead of regular mustard if wanted (Duncan, 2004).

2.3 Emulsions

2.3.1 Structure

Mayonnaise is an oil-in-water (O/W) emulsion with oil droplets in a continuous water phase, see Figure 2.4. The oil droplets are present in a range of sizes, usually between 0.1 and 10 µm (McClements, 2016). The oil content in mayonnaise is usually around 75-80% oil, where a higher oil content gives a more firm structure. When the volume fraction of a monodispersed phase is higher than 64% the droplets interact more with each other and behave as elastic solids (Mason et al., 1995). This interaction makes the droplets less movable and they have formed a glass state. When the monodispersed phase is more than 74% the droplets become polyhedral instead of spherical, which contribute to greater interactions between droplets (Harrison and Cunningham, 1983). However, a polydisperse phase (like mayonnaise) can be packed more efficiently and both the glass state transition and droplet deformation occur at a higher volume fraction.

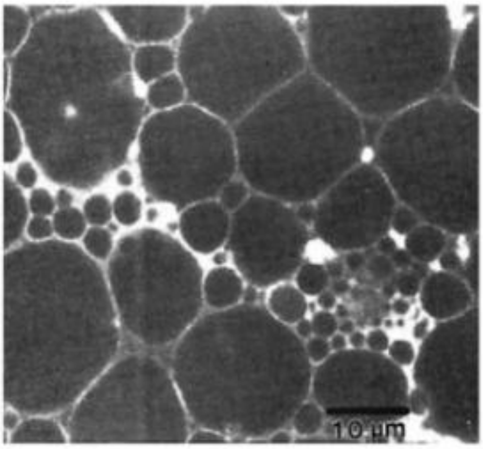

Figure 2.4: Tightly packed oil droplets surrounded by a continuous water phase. Picture taken by Confocal Laser Scanning Microscopy (CLSM). Fluorescent staining of both the interface and the continuous phase by Nile Blue. (Heertje, 1993).

At the interface between the oil and the water there is a very thin film where emulsifiers, coming from egg yolk plasma, are situated. The continuous water phase surrounds the deformed oil droplets. Here, the most abundant structure is the egg yolk granules but it also contains the flavoring substances of mayonnaise. The granules adhere to each other and to the apoproteins situated at the oil-water interface forming a network. The protein network helps to make the emulsion more stable and increases the viscosity of the product. The viscosity is also influenced by the adhesiveness between the oil droplets. The oil droplets are in a glass state with van der Waal attraction between them making them form a network of droplets. The strength of the droplet network is determined by how adhesive the droplets are to each other. (Depree and Savage, 2001)

When egg white is used together with egg yolk the mayonnaise becomes more viscoelastic. This suggests that proteins in egg white can interact with the egg yolk proteins to make the protein network between the oil droplets stronger. (Kiosseoglou and Sherman, 1983)

2.3.2 Droplet Formation

The process to form an emulsion is called emulsification, where the two phases are mixed together and smaller and smaller droplets of the dispersed phase are formed. The droplet size achieved is dependent on both the rate of droplet disruption and droplet coalescence.

In droplet disruption there is a balance between the interfacial forces that keeps the droplets together and the disruptive forces, created by the mixing, that break-up the droplets. The interfacial force that keeps the droplets spherical is the Laplace pressure ΔP_L which is described by Equation 2.1:

$$\Delta P_L = \frac{4\gamma}{d} \qquad [2.1]$$

Where γ is the interfacial tension between oil and water and d is the diameter of the droplet. The Laplace pressure increases with decreased droplet size which means that more energy is needed to disrupt a smaller droplet than a bigger one. In order for the droplets to break up, they first need to be deformed. Generally, the duration of the disruptive forces needs to be longer than the time the droplets need to deform, in order for the droplets to break up. (McClements, 2016)

The new droplet interfaces are covered with emulsifiers that are transported to the interface by convection. Many of the new droplets coalesce into bigger droplets again but if more new droplets are formed than the number that coalesce then the amount of droplets will increase. A schematic picture of the balance between droplet breakup and coalescence can be seen in Figure 2.5. As more droplets a broken up more emulsifiers are needed to cover the interface and the concentration of the emulsifier in solution decreases. To avoid having the emulsifier limit the amount of droplets that can be formed the emulsifier needs to be present in excess. (Walstra, 1993)

Emulsifiers attach to the interface and lower the interfacial tension which facilitates the droplet breakup. Different emulsifiers can lower the interfacial tension to different degrees. The lower surface tension an emulsifier gives the less energy input is needed to obtain a given droplet size. (Walstra, 1993; McClements, 2016)

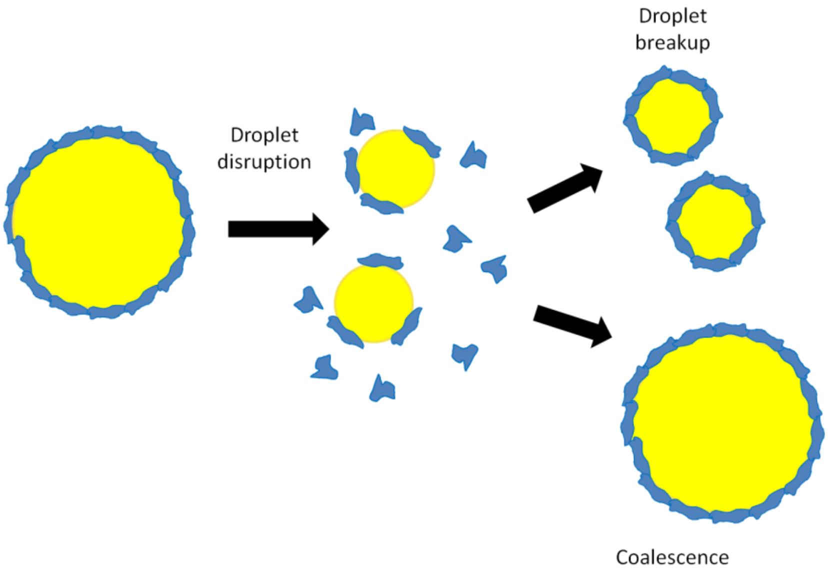

Figure 2.5: Schematic overview of droplet breakup and coalescence.

Emulsifiers are classified using the Hydrophile-Lipophile Balance (HLB) concept where the emulsifier is given a number based on the chemical structure. An emulsifier with many hydrophilic groups has a high number (10-18), an emulsifier with many lipophilic groups has a low number (3-6) and in the middle there are the intermediate emulsifiers (7-9). To get the right HLB number different emulsifiers can be mixed together. A high HLB number shows that the emulsifier is water soluble and therefore stabilizes O/W emulsions. (McClements, 2016) Egg yolk is water soluble and consequently has a relatively high HLB number which promotes an O/W emulsion during the formation of mayonnaise (Stauffer, 2002). An emulsifier with a low HLB number is oil soluble and instead stabilizes W/O emulsions. The intermediate ones can stabilize both types of emulsions and lowers the interfacial tension more than both high and low HLB number emulsifiers. With this very low interfacial tension the interfaces easily break, with coalescence as a result. (McClements, 2016)

2.3.3 Stability

Mayonnaise is a product that has a long shelf life and during this time the product needs to both keep its properties and remain safe for consumption. To keep the properties of the mayonnaise the emulsion needs to be stable. Three common phenomena destabilize emulsions: creaming, flocculation and coalescence. Creaming is when the emulsion separates because of a density difference where the lighter oil droplets rise to the surface. This is not a problem in mayonnaise since the volume of dispersed phase is high enough to keep the droplets from moving. By packing the system with droplets of dispersed phase the droplets cannot move because they are blocked in. (McClements, 2016) Flocculation is when the emulsion droplets aggregate and thereby form larger units (Tadros, 2013). In mayonnaise the droplets are always in contact with each other because of the glass state it is in so there is no traditional flocculation. It is however desired that the droplets have an attractive interaction with many junction points since this gives the mayonnaise a firmer texture. Coalescence is when smaller droplets merge together forming a larger droplet. This occurs when droplets come in contact with each other and the interfacial film is ruptured. This eventually results in phase separation and a ruined mayonnaise. (McClements, 2016)

If the oil used in mayonnaise production is kept at low temperatures there is a risk that crystals will be formed. These crystals promote coalescence by protruding out of the droplets and into the water phase. When the protruding part reaches another oil droplet partial coalescence is promoted. (McClements. 2016)

2.2.4 Phase inversion

Phase inversion occurs when the emulsion changes from being an oil-in-water (O/W) emulsion to a water-in-oil emulsion (W/O), or the other way around. At the time of inversion it is probable that a more complex structure like an oil-in-water-in-oil emulsion (o/W/O) or a water-in-oil-in-water (w/O/W) is formed as a transition state. (McClements, 2016) In the case of mayonnaise (which is an O/W emulsion), the transition state o/W/O can be observed during emulsification phase inversion where small pieces of mayonnaise are floating in oil.

Phase inversion can be either transitional or catastrophic. Transitional phase inversion is when the phase inversion is caused by a change in the formulation, like a change in temperature or salinity (Thakur et al., 2008). This type of phase inversion is reversible and shows very little hysteresis. For example, when the temperature is increased above the critical temperature there is a phase inversion and when the temperature is lowered passed the critical point again, the emulsion returns to the original state. In the case of mayonnaise changes in salinity are more probable to occur which affects the proteins in the egg yolk. As previously mentioned proteins in egg yolk are the emulsifiers. They provide electrostatic repulsion and therefore an increase in electrolytes can cause a transitional phase inversion of mayonnaise. This is because the electrical charges on the proteins become screened with increased amount of counterions. (McClements, 2016). The focus of this mater thesis will not be on transitional phase inversion.

Catastrophic phase inversion is when the phase inversion is caused by a change in the oil-water ratio (Thakur et al., 2008). When the dispersed phase volume becomes too high a phase inversion occurs. This type of inversion is dependent on the hysteresis of the system which makes the phase inversion irreversible (Kumar et al., 2015). This results in a hysteresis zone around the inversion line, meaning that the inversion occurs at different conditions depending on which direction the composition is changed. The phase inversion point is dependent on the intensity of the agitation and the rate of adding the dispersed phase. (McClements, 2016) For example, when making mayonnaise at home the oil must be added slowly in order to avoid a phase inversion. The high oil fraction in mayonnaise also makes the system more prone to undergo a phase inversion. The highest oil content that can be used in the production of mayonnaise is 84% (Duncan, 2004). At higher concentrations the droplets are packed too tightly and the interfacial film becomes very thin and fragile.

An overview of transitional and catastrophic phase inversion is shown in Figure 2.6.

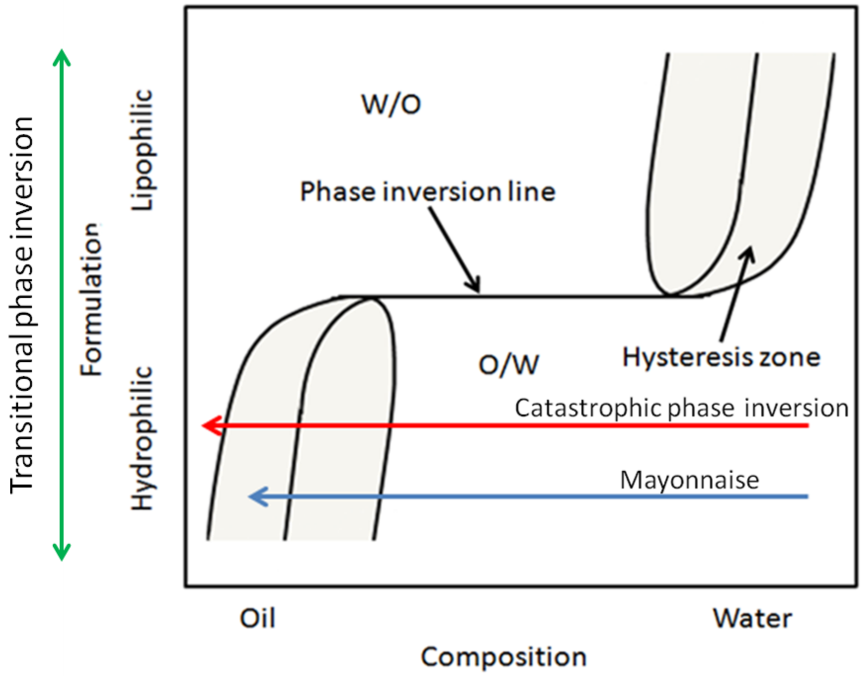

Figure 2.6: A schematic overview of which emulsion will be formed depending on formulation and composition of the system. The blue arrow represents how mayonnaise is made when oil is added to the system. The red arrow represents how mayonnaise with too much oil will undergo catastrophic phase inversion when it passes the hysteresis zone. Transitional phase inversion is illustrated by the green arrow. (Modified after original by Salager et al., 2001).

In Figure 2.6 it can be seen how a transitional phase inversion occurs when the formulation of the system is changed, or by moving vertical in the figure (green arrow). A catastrophic phase inversion, which is more interesting in mayonnaise production, occurs when the composition of the system is changed. At the start of mayonnaise production the system is only composed of a water phase. During the coarse emulsion oil is added and the composition changes, this is illustrated by the blue arrow in Figure 2.6. Because of the hysteresis of the system the phase inversion line can be crossed without causing a catastrophic phase inversion. Instead a catastrophic phase inversion occurs when the system have passed the hysteresis zone, illustrated by the red arrow in Figure 2.6. This can be done in two different ways: by having a high local oil fraction or by adding to much oil to the entire system. While it is easy to understand why a system with too much oil will undergo a phase inversion, understanding why a high local oil fraction is important is more difficult. This is because a high local oil fraction only influences the system when it occurs at the site of droplet formation. In the case of mayonnaise production the site of droplet formation is the rotor/stator combination. When this part of the mixer gets filled with a high oil fraction the local system in the rotor is pushed to the left of the hysteresis zone resulting in a catastrophic phase inversion.

The occurrence of a phase inversion is also dependent on the position of the rotor, more particularly if it is in the water phase or in the oil phase. When the rotor is in the water phase small amounts of oil will be dragged into the higher amount of water and an O/W emulsion will be formed. This is illustrated in Figure 2.7. If the rotor is in the oil phase the opposite will take place and a W/O emulsion will be formed. (Salager et al., 2001) However, even in the case of having the rotor in the water phase it is still possible to form local W/O emulsions. This might happen when too much oil gets into the rotor at a given time and thereby pushing the local system to the left of the hysteresis zone in Figure 2.6.

The final emulsion will consequently be determined by which phase the rotor spends the majority of the time in, which would be the water phase in the scenario illustrated in Figure 2.7. It is therefore critical to control the position of the rotor to avoid a phase inversion.

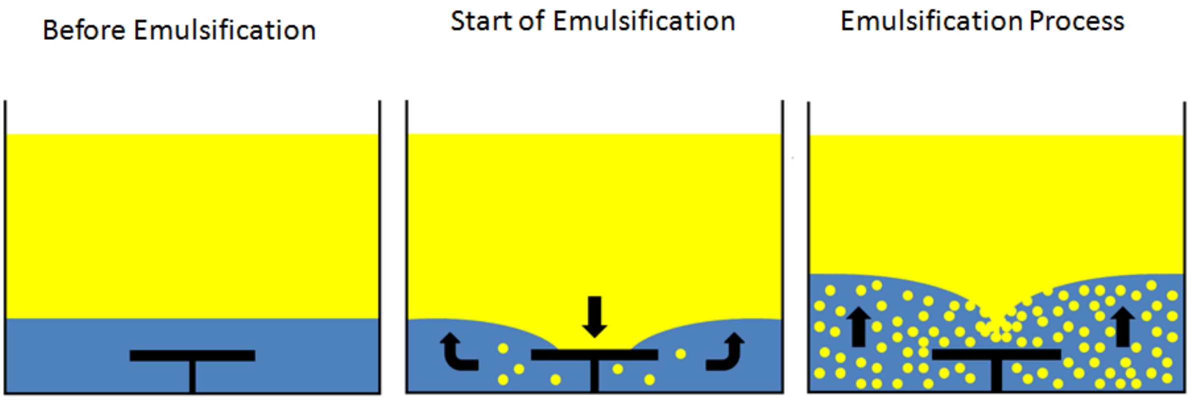

Figure 2.7: Illustration of the effect of the placement of the rotor. The blue on the bottom represents water while the yellow on top represents oil. When the stirring is started the oil will be dragged down to the water phase to form an O/W emulsion (Modified after original by Salager et al., 2001).

3 Method

3.1 Mayonnaise production

3.1.1 Mixers

3.1.1.1 High shear mixer

The mixer used was a Tetra Pak High Shear Mixer Pilot unit B200-100VA that can be seen in Figure 3.1 below. B200 shows that it is a batch mixer with a 200 mm rotor head. 100VA shows that is has a batch size of 100 l, that vacuum can be applied and that there is an agitator in the mixer. Different stators are possible to use in the mixer. The stator used for this thesis has 5x14 mm slots and a gap of 1 mm between the rotor and the stator. During a run the stator can be either up or down. When the stator is down all the flow created by the rotor must go through the stator giving a high shear force. When the stator is up the flow can go under it resulting in a lower shear force. The batch-size was modified to 33 kg in order to compare the result from this thesis with previous result by Andersson (2015).

Figure 3.1: Above: the mixer from the side. Left: stator with 5x14 mm slots (Andersson, 2015). Right: inside the vessel of the mixer with the rotor and stator marked.

The standard mixer settings used during the experiments can be seen in Table 3.1.

Table 3.1: Standard mixer settings.

Diameter vessel	51 cm
Height vessel	72 cm
Vacuum	500 mbar
Cooling water temperature	11 °C
Agitator speed	1 m/s

3.1.1.2 Immersion blender

The immersion blender used was of the type Bamix BAM493536 and the mixer head can be seen in Figure 3.2. The rotating part of the mixer-head had a diameter of 3.6 cm and experiments were run with the tip speed of 19 m/s. The flow pattern for an immersion blender is illustrated in Figure 3.2.

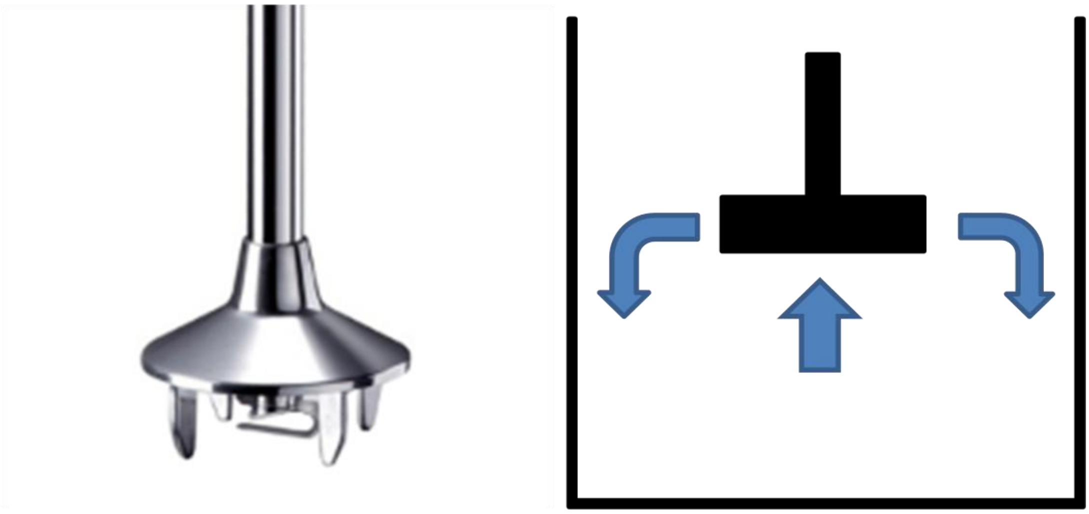

Figure 3.2: Left: Mixer head of the immersion blender (Elgiganten.se, 2017). Right: Flow pattern through an immersion blender.

3.1.2 Recipes

Four recipes were used, one with liquid egg yolk, one with decreased liquid egg yolk, one with egg yolk powder and one containing both liquid whole egg and liquid egg yolk, see Table 3.2 to 3.5. The standard temperature of the ingredients was 8 °C.

Table 3.2: The standard mayonnaise recipe.

Ingredient	**Fraction (w/w%)**	**Amount (kg)**
Water	5.80	1.91
Salt	0.300	0.099
Sugar	0.300	0.099
Mustard	2.50	0.826
Vinegar	2.10	0.693
Egg yolk, liquid (8% salt)	8.50	2.81
Rapeseed oil	80.5	26.6
Total	**100**	**33.0**

Table 3.3: The mayonnaise recipe with decreased egg content.

Ingredient	**Fraction (w/w%)**	**Amount (kg)**
Water	8.10	2.67
Salt	0.500	0.165
Sugar	0.300	0.099
Mustard	2.50	0.826
Vinegar	2.10	0.693
Egg yolk, liquid (8% salt)	6.0	1.98
Rapeseed oil	80.5	26.6
Total	**100**	**33.0**

Table 3.4: The mayonnaise recipe with egg yolk powder.

Ingredient	**Fraction (w/w%)**	**Amount (kg)**
Water	9.80	3.23
Salt	1.20	0.396
Sugar	4.00	1.32
Mustard flour	0.300	0.099
Vinegar	3.00	0.990
Egg yolk powder	2.70	0.891
Rapeseed oil	79.0	26.1
Total	**100**	**33.0**

Table 3.5: The mayonnaise recipe with whole egg.

Ingredient	**Fraction (w/w%)**	**Amount (kg)**
Water	7.10	2.34
Salt	1.04	0.344
Sugar	4.00	1.32
Mustard flour	0.300	0.099
Vinegar	3.00	0.990
Whole egg, liquid (0% salt)	3.61	1.19
Egg yolk, liquid (8% salt)	1.96	0.646
Rapeseed oil	79.0	26.1
Total	**100**	**33.0**

The ingredients used in this master thesis can be seen in Table 3.6 below.

Table 3.6: The ingredients used with name, company and city of origin.

Ingredient	**Name**	**Company**	**City**
Egg yolk, liquid (8% salt)	Äggula saltad 8% (pasteurized)	Källbergs	Töreboda
Egg yolk powder	Pasteurzed dried egg yolk	Källbergs	Töreboda
Mustard	Mayosenf hell ohne süßstoff	Kühne	Hamburg
Mustard flour	Senapspulver MUSTA0802	Fermia	Höganäs
Rapeseed oil	Svensk rapsolja	AAK	Karlshamn
Vinegar	Ättiksprit 12%	Druvan	Eslöv
Whole egg, liquid (0% salt)	Pastöriserade flytande ägg	Kronägg	Perstorp

3.1.3 Procedure

3.1.3.1 High shear mixer

Mayonnaise is made in three steps: premixing, coarse emulsion and emulsification. At the different steps the rotor speed, the time and the position of the stator are set to the values needed for the specific run. The steps are schematically illustrated in Figure 3.3.

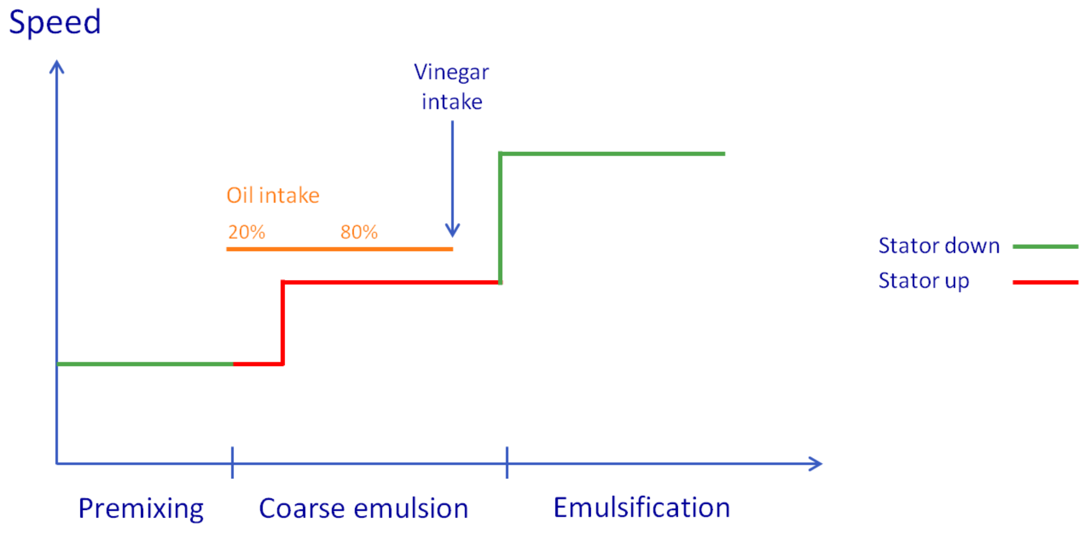

Figure 3.3: Schematic overview of the steps in mayonnaise production.

3.1.3.1.1 Premixing

The first step in mayonnaise production is the premixing step. In this step water, salt, sugar, mustard and egg yolk is mixed together for 30 s with applied vacuum and connected cooling water. The tip speed used is 5.5 m/s.

3.1.3.1.2 Coarse emulsion

The coarse emulsion step is the second step after the premixing where the oil is added to the water phase to form an emulsion. The oil is added form a funnel attached to the mixer-vessel using the vacuum, see Figure 3.1. When oil is initially added to the mixer the coarse emulsion tip speed is low (5.5 m/s) and the stator is raised to the low shear position. The addition of oil is done by using a vacuum of 500 mbar inside the mixing vessel. The first 20% of the oil is added at a lower rate (about 1 kg/s or lower when extremely low oil flows are used) in order to avoid splashing. After the first 20% oil has been added the coarse emulsion tip speed is increased (generally to 8.3 or 13.0 m/s) and the oil inlet flow rate is increased to the value set by a second valve. When all the oil has been added to the mixer, the vinegar is added from a second funnel attached to the back of the mixer-vessel, using the vacuum. This is followed by a 10 s mixing step to evenly distribute the vinegar in the formed mayonnaise.

3.1.3.1.2 Emulsification and sampling

The last step is the emulsification step where the emulsion droplets decrease in size in order to form a more stable mayonnaise. The stator is lowered to the high shear position and the rotor tip speed is increased. After 20 s the mixer is stopped and a sample is taken. The mixer is then run for another 20 s and the next sample is taken. The mixer takes 5 s to accelerate up to the right rotor tip speed and this time is not included in the 20 s. Samples are generally taken at 20 s, 40 s, 60 s, 80 s, 100 s and 150 s. The samples are taken from the center of the mayonnaise and put in a plastic container. They are then stored in room temperature until analysis.

The time the samples are taken are made into passages in order to make it easier to scale the process. Passages are a measure of how many times a droplet has theoretically passed through the rotor. At a set mixer speed the rotational speed can be calculated by Equation 3.1:

$$N = \frac{v}{D \cdot \pi} \qquad [3.1]$$

Where N is the rotational speed, v is the tip speed of the rotor and D is the diameter. From this the flow through the rotor can be calculated by using Equation 3.2:

$$Q = N_f \cdot N \cdot h \cdot D^2 \qquad [3.2]$$

Where Q is the mass flow through the rotor, N_f is a constant, N is the rotational speed, h is the height of the rotor and D is the diameter of the rotor. The circulation time can be calculated by Equation 3.3:

$$Circ. time = m/Q \qquad [3.3]$$

Where m is the mass of the batch and Q is the mass flow through the rotor. By using the emulsification time the number of passages can be calculated by using Equation 3.4:

$$Passages = emul. time\ /circ. time \qquad [3.4]$$

Since passages takes the time, the mixer and the batch size into account it becomes easier to compare with experiments made in different set-ups, compared to if only time where used.

3.1.3.2 Immersion blender

The mayonnaise production was started adding water, salt, sugar, mustard and egg yolk to a beaker. The ingredients were then mixed for around 10 s with a tip speed of 19 m/s. The oil was then added though a funnel with an oil addition flow rate of 3.3 g/s while using the same tip speed of 19 m/s. After all the oil had been incorporated the vinegar was added and the final mayonnaise was mixed for an additional 10 s.

3.2 Mayonnaise analysis

The samples of mayonnaise obtained were analyzed the day after production.

3.2.1 Texture

The texture was analyzed by measuring the force needed to compress the mayonnaise. This gives a value called the Stevens value. A Texture Analyser (TA-XT2i, Stable Microsystems, UK, Godalming) together with a Brookfield Engineering probe was used during the measurements, see Figure 3.4. The mayonnaise was transferred to a 100 ml beaker by using a syringe with a cut-off-tip. The probe was then lowered into the beaker and a measurement was started. The speed of the probe was 2.0 mm/s and the trigger force was 5.0 g. The average of the measured force between 5 and 11 s gave the Stevens value, see Figure 3.4. The procedure was repeated three times for each sample. For more details see Cedergårdh (2014).

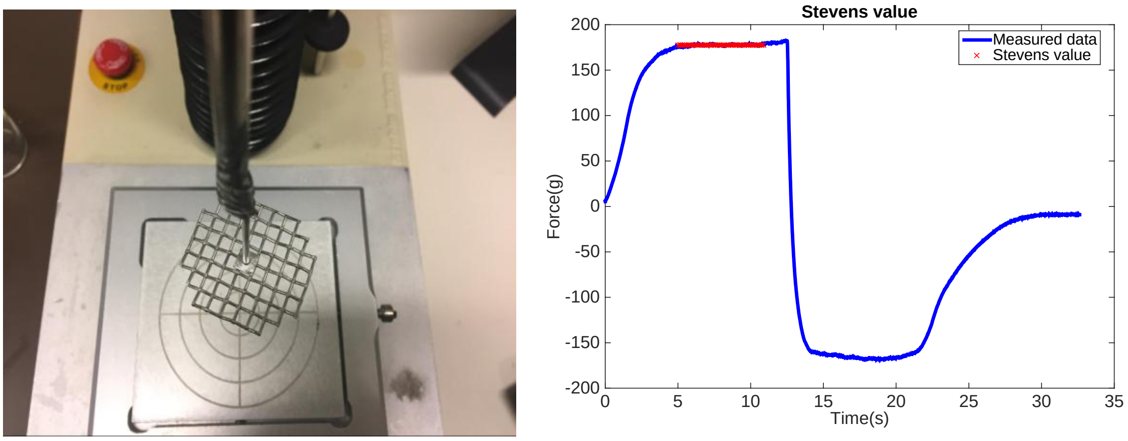

Figure 3.4: Left: The probe used for measuring Stevens value. Right: Example of texture curve obtained when measuring a sample. The values used for making the average are highlighted in red. (Andersson, 2015)

3.2.2 Droplet size

The droplet size of the oil droplets was measured using laser diffraction. First the mayonnaise was dispersed in a surfactant solution. 2 ml mayonnaise was added to a beaker containing 300 ml 0.01% SDS (sodium dodecyl sulphate). The mayonnaise was then dispersed by using an Ultra Turrax (IKA-Labortechnik, Germany, Staufen) for 15 s. To remove air bubbles the sample was transferred to a vacuum proved flask and a diaphragm pump (Vacuumbrand, Germany, Wertheim) was used for 1 min. The prepared sample was then analysed with a Laser diffraction instrument (Mastersizer 2000, Malvern Instruments, UK, Workshire) to obtain the droplet size distribution. The absorption was set to 0.0001 and the refractive index for rapeseed oil was set to 1.474. For more details see Cedergårdh (2014).

From the results of the droplet size measurement the mode value of the surface weighted size distribution. The mode value is the highest point of the peak obtained and represents the most common droplet size, see Figure 3.5. The mode value is more stable than the d(3,2) and d(4,3) values since it is not affected by a few large impurities, like air bubbles, in the sample.

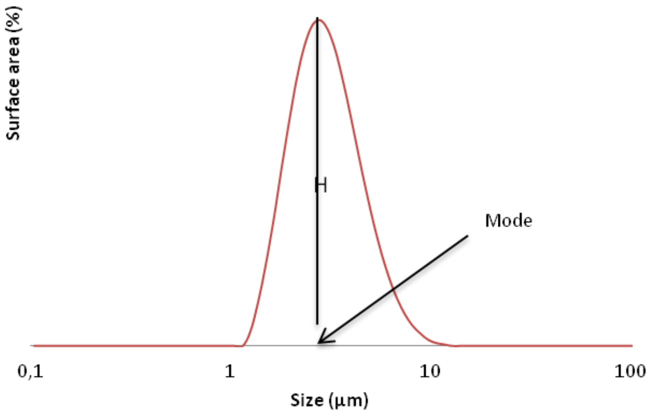

Figure 3.5: Droplet size distribution with the mode value marked. (Andersson, 2015)

4 Experimental design and results

An overview of the experiments performed is presented in Figure 4.1.

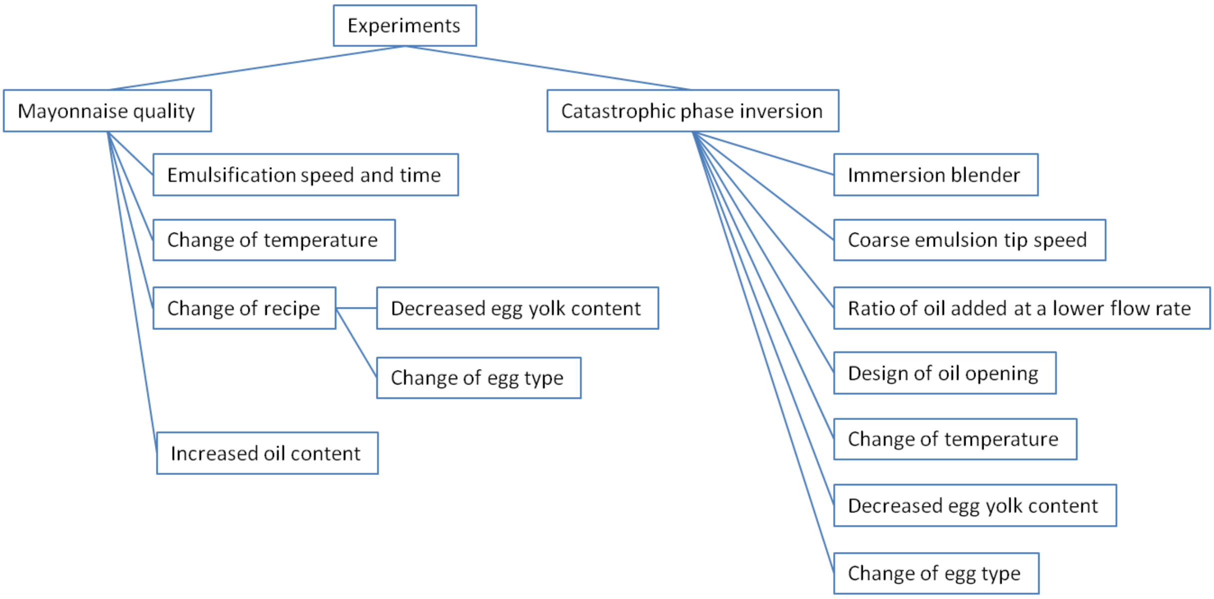

Figure 4.1: Overview of the experiments performed.

The settings for all the runs made are found in Appendix in section 8.1 *Mixer settings*. The results from the texture and droplet size measurements are found in Appendix in section 8.2 *Analysis results* and the results for the experiments about phase inversion can be seen in Appendix in section 8.3 *Phase inversion results*.

4.1 Statistical analysis

The reproducibility of the results was established by making three runs using the same settings and times, see Table 8.1 in section Appendix 8.1 *Mixer settings*. The runs were made on different days in order to get a standard deviation between batches. The measurement on both the texture and droplet size was repeated three times in order to establish a standard deviation within the batch. Two one-way ANOVAs were performed on the results within a batch and between the batches to get the variances. The variances from the ANOVAs were then compared by performing a F-test to see if there was a significant difference between the variances. From the variances the standard deviation was calculated. The statistical analysis was done in the same way for both the texture and the droplet size results.

The F-test of the Stevens value showed that there was no significant difference between the variances from within and between batches. Therefore, the largest variance was chosen. In this case the largest variance came from within a sample. The standard deviation originating from within a sample was therefore used together with triplicate measurements.

For the droplet size measurements there was a significant difference between the variance from within and between batches. Once again the largest variation was used, which in this case was the variance stemming from between batches. Therefore, the standard deviation from between batches was used together with triplicate measurements. The standard deviations can be found in Table 4.1.

From the standard deviation a confidence interval was made by using the t-distribution with significance level of α=0.05, see Equation 4.1 below:

$$\mu = x \pm t_{\alpha/2} \cdot \frac{s}{\sqrt{n}} \quad [4.1]$$

Where µ is the interval, x is the mean, t is the t-distribution for α=0.05, s is the standard deviation and n is the number of samples which is three for Stevens value and one for droplet. The range of the confidence interval can be seen in Table 4.1. The results from this statistical analysis were used for future results.

Table 4.1: The standard deviation and confidence intervals used for the Stevens value and the droplet size with α=0.05 and n=3 for Stevens value and n=1 for droplet size.

Method	**Standard deviation**	**Confidence interval (α=0.05)**
Stevens value	5.2	13
Droplet size [µm]	0.10	0.65

4.2 Mayonnaise quality

4.2.1 Emulsification speed and emulsification time

The ingredients for the water phase were mixed for 30 s in the pre-mix step. In the coarse emulsion step 20% of the oil was added with the tip speed 5.5 m/s and the flow 0.4 kg/s while the remaining 80% was added with the tip speed 13.0 m/s and the flow 0.61 kg/s. After the oil had been added the vinegar was added and mixed for 10 s. In the emulsification step the tip speeds low, medium and high were used. The samples were taken at the normalized passages seen in Table 4.2. For this experiment the *standard pipe* was used to add the oil, see Appendix 8.5 *Pipes used for oil addition* for more information. The result of the texture analysis can be seen Figure 4.2.

Table 4.2: The normalized passages the samples were taken at and which time they correspond to.

Time [s]	Normalized passages for low	Normalized passages for medium	Normalized passages for high
20	0.094	0.11	0.12
40	0.19	0.21	0.23
60	0.28	0.32	0.36
80	0.37	0.43	0.48
100	0.47	0.53	0.60
150	0.70	0.80	0.91

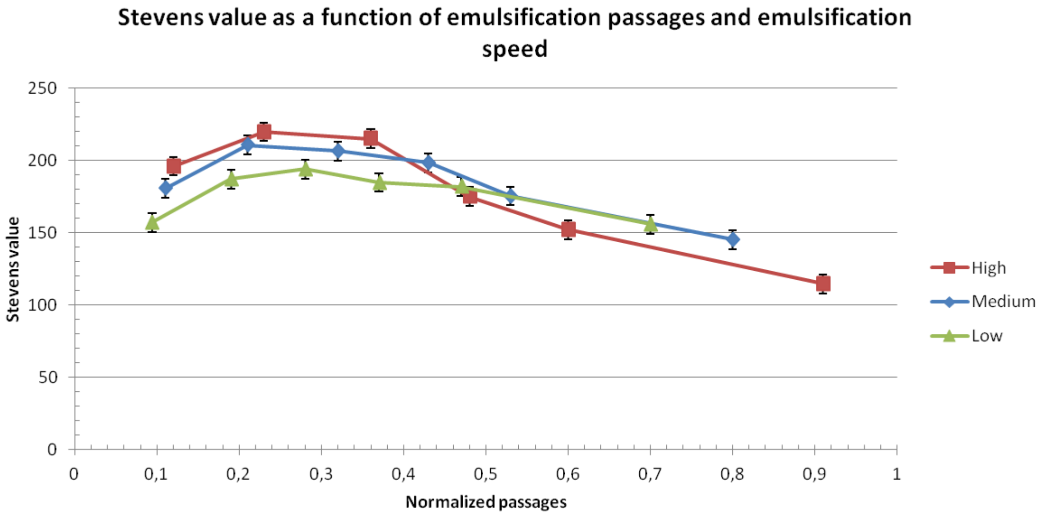

Figure 4.2: Stevens value as a function of the amount of normalized emulsification passages for the three different emulsification tip speeds (low, medium and high).

From this figure it can be seen that the Stevens value increase in the beginning, before reaching an optimum and thereafter decreasing. This behavior can be seen for all three emulsifications tip speeds. The peak texture is higher for higher emulsifications tip speeds and the decline in texture seems to be more rapid compared to the lower tip speeds. The peak in Stevens value occur around 0.3 normalized passages independent of the emulsification tip speed.

The effect of both the emulsification speed and emulsification time on the droplet size of mayonnaise can be seen in Figure 4.3.

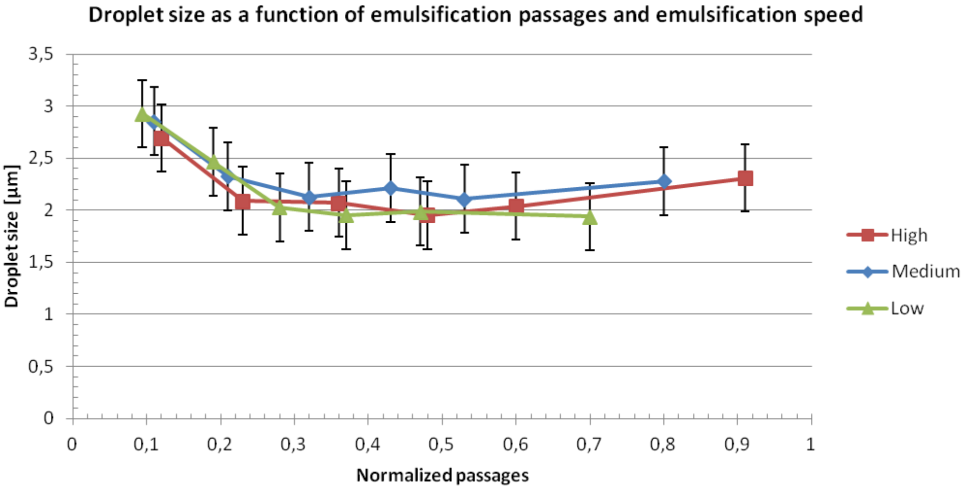

Figure 4.3: Droplet size as a function of the amount of normalized emulsification passages for the three different emulsification speeds (low, medium and high).

From this figure it can be seen that the droplet size decreases in the beginning before reaching a minimum and thereafter increasing. This behavior can be seen for all three emulsification speeds but no significance difference for the increase in droplet size can be observed. A relationship between the Stevens value (Figure 4.2) and the droplet size (Figure 4.3) can be seen where a higher Stevens value coincide with a smaller droplet size. The minimum in droplet size occur around 0.3 normalized passages which coincides with the maximum in Stevens value. Mayonnaise that has been mixed well beyond the optimum of 0.3 normalized passages has a lower quality and is referred to as over-sheared.

Using a higher emulsification speed gives mayonnaise with a firmer texture in a shorter time, which can decrease the production time. However, it is important to remember that mayonnaise which is produced at a higher emulsification speed is more sensitive to over-shear.

4.2.2 Change of temperature

The temperature of the ingredients was changed to different combinations, see Table 4.3.

Table 4.3: The temperatures tested for the oil and the remaining ingredients. The experiment with 8 °C oil and 8 °C ingredients is the standard.

Oil temperature [°C]	Ingredients temperature [°C]
1	8
8	8
20	8
8	20

The ingredients for the water phase were mixed for 30 s in the pre-mix step. In the coarse emulsion step 20% of the oil was added with the tip speed 5.5 m/s and the flow 0.4 kg/s while the remaining 80% was added with the tip speed 13.0 m/s and the flow 0.61 kg/s. After the oil had been added the vinegar was added and mixed for 10 s. The samples were taken at 0.11, 0.21, 0.32, 0.43, 0.53 and 0.80 normalized passages and the texture results can be seen in Figure 4.4. For this experiment the *standard pipe* was used.

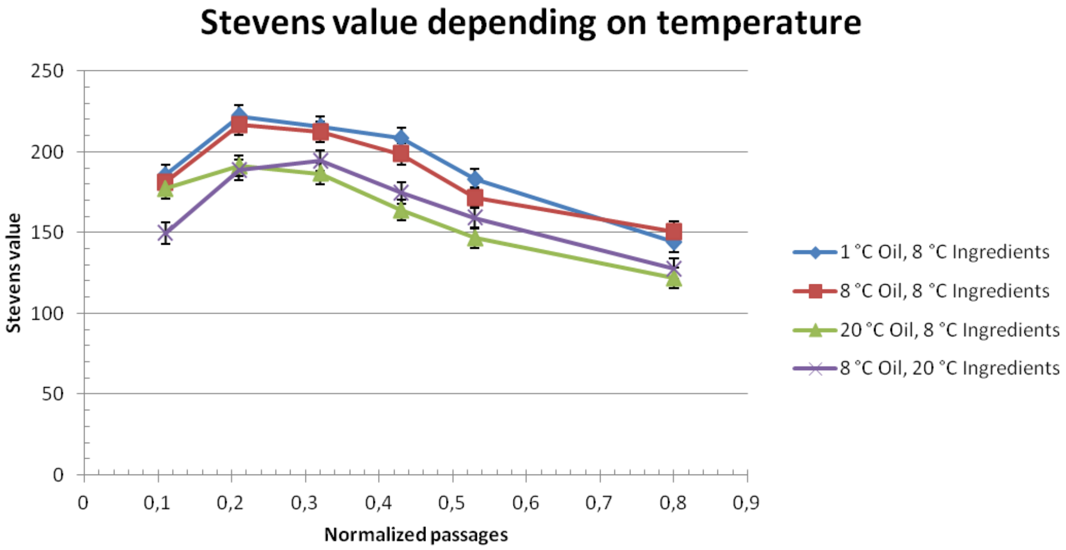

Figure 4.4: Stevens value as a function of the amount of normalized emulsification passages for the four different temperature combinations 1 °C oil with 8 °C ingredients, 8 °C oil with 8 °C ingredients, 20 °C oil with 8 °C ingredients and 8°C oil with 20 °C ingredients.

Mayonnaise made with the oil temperatures 1 °C and 8 °C, with the ingredient temperature of 8 °C, is similar and shows no significant difference between the measure points. Both the mayonnaise made with 20 °C oil and 8 °C ingredients and the mayonnaise made with 8 °C oil and 20 °C ingredients have lower Stevens values than the two experiments previously mentioned.

The droplet size results can be seen in Figure 4.5.

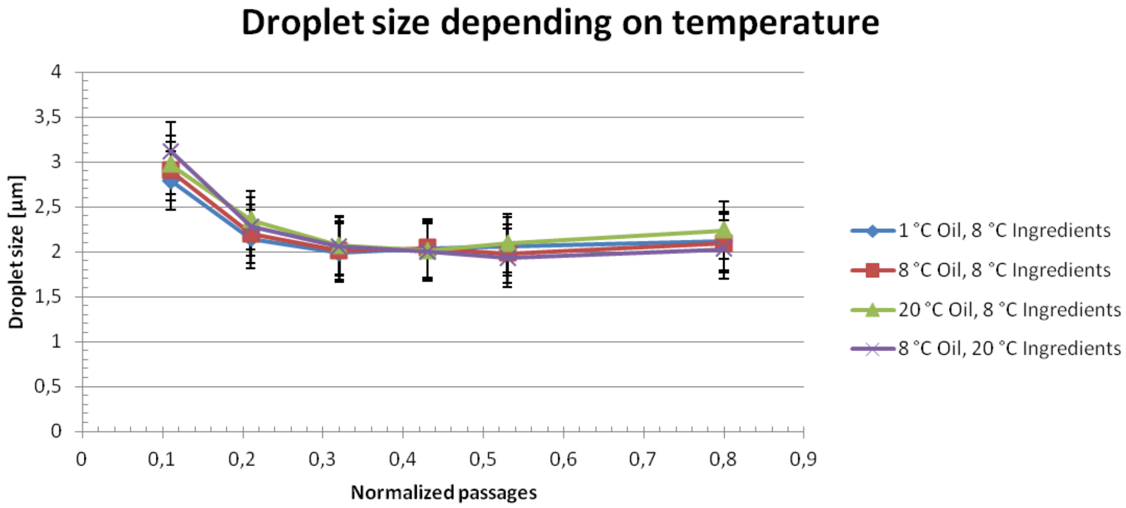

Figure 4.5: Droplet size as a function of the amount of normalized emulsification passages for the four different temperature combinations 1 °C oil with 8 °C ingredients, 8 °C oil with 8 °C ingredients, 20 °C oil with 8 °C ingredients and 8°C oil with 20 °C ingredients.

Figure 4.5 shows that there is no difference in droplet size between the results for the different temperatures. This shows that the higher temperature during the 20 °C run affects the texture without affecting the droplet size. This indicates that the lower Stevens value is not because of coalescence.

The cloud point of rapeseed oil is 6 °C and the run at 1 °C was made to test if cloudy oil affected the mayonnaise negatively since crystals in the oil promote partial coalescence. The oil was cloudy but no effect could be seen in these results. It is possible that more effect could be observed if the oil had been kept at a low temperature for a longer period of time. It was kept at low temperature only for a few hours and it is possible that more crystals could have been formed if the oil had been kept at a low temperature for twelve hours or longer.

4.2.3. Change of recipe

In the standard recipe liquid egg yolk were used. How the mayonnaise changed when using recipes with less liquid egg yolk, egg yolk powder and liquid whole egg were investigated. The recipes used can be found in section 3.1.2 *Recipes*. For all experiments in this section the *standard pipe* was used.

4.2.3.1 Decreased egg yolk content

In this experiment the amount of liquid egg yolk was decreased from 8.5% to 6% (w/w%). The amount of added salt and water was increased to keep the salt concentration and the total amount of water phase the same as in the standard recipe.

The ingredients for the water phase were mixed for 30 s in the pre-mix step. In the coarse emulsion step 20% of the oil was added with the tip speed 5.5 m/s and the flow 0.4 kg/s while the remaining 80% was added with the tip speed 13.0 m/s and the flow 0.61 kg/s. After the oil had been added the vinegar was added and mixed for 10 s. The samples were taken at 0.11, 0.21, 0.32, 0.43, 0.53 and 0.80 normalized passages and the texture results can be seen in Figure 4.6.

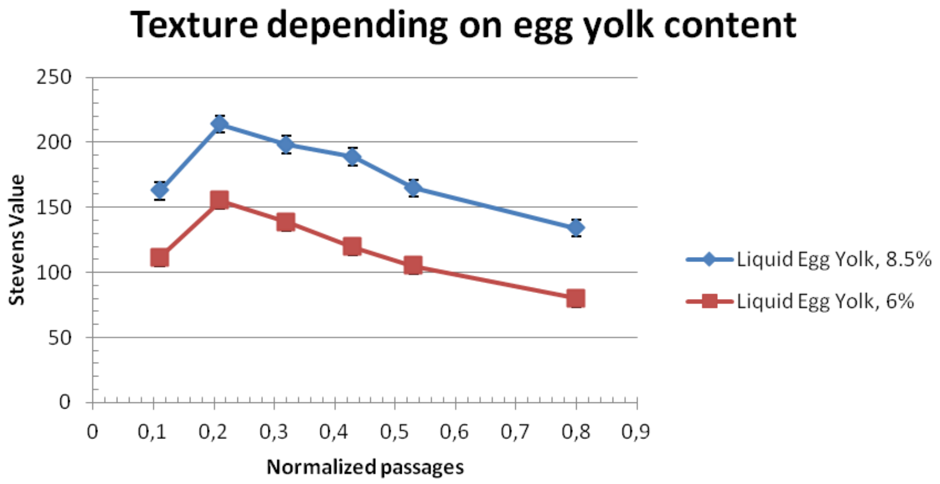

Figure 4.6: Stevens value as a function of the amount of normalized emulsification passages for mayonnaise made with both 8.5% and 6% liquid egg yolk.

In Figure 4.6 the results from using the lower amount of 6% liquid egg yolk is compared with the standard of 8.5% liquid egg yolk. The curves follow parallel to each other and have the same basic appearance. The curve for 6% liquid egg yolk show significantly lower values at all measured passages and are about 50 units lower. The mayonnaise with 6% egg yolk had the same appearance as the mayonnaise with 8.5% egg yolk. It was only looser in its consistency as was reflected by the lower Stevens value.

The results of the droplet size measurements can be seen in Figure 4.7.

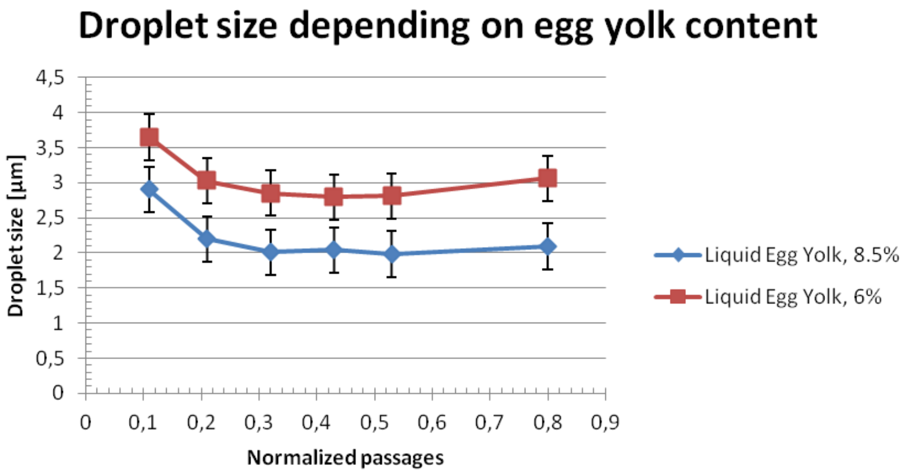

Figure 4.7: Droplet size as a function of the amount of normalized emulsification passages for mayonnaise made with both 8.5% and 6% liquid egg yolk.

In Figure 4.7 it can be seen that the droplets size also show a parallel behavior between the 8.5% liquid egg yolk and the 6% liquid egg yolk. The curve for 6% liquid egg yolk show significantly larger droplets at all measured passages. This agrees well with the results obtained by Andersson (2015) where the interpolated value for 6% liquid egg yolk gave a Stevens value just above 100 and a droplet size around 4 µm after 0.11 passages. A logical explanation for the lower Stevens values and the larger droplet size could be that there were not enough emulsifiers to cover all possible interfaces. However, by assuming a surface coverage of 2 mg/m^2 (Le Denmat et al., 2000) and a mean diameter of 2 µm there should be enough proteins to cover all interfaces.

4.2.3.2 Change of egg type

In the experiment the egg type was changed to both egg yolk powder and liquid whole egg with added liquid egg yolk. The recipes can be found under section 3.1.2 *Recipes*.

When the egg type was changed to egg yolk powder the amount of egg yolk corresponded to 5.6% (w/w%) liquid egg yolk. The ingredients for the water phase were mixed for 30 s by hand in the mixer vessel and then for 30 s by the mixer. This was repeated two more times until no more lumps were visible. The coarse emulsion step for this experiment used the flow rate of 0.23 kg/s for the first 20% at 5.5 m/s tip speed and the remaining 80% used the same flow rate of 0.23 kg/s with the tip speed of 8.3 m/s. After the oil had been added the vinegar was added and mixed for 10 s. Samples were taken at 0.053, 0.11, 0.21, 0.32, 0.43, 0.53 and 0.80 normalized passages.

When the egg type was changed to liquid whole egg with added liquid egg yolk the amount of egg yolk corresponded to a total content of 3.4% (w/w%). The ingredients for the water phase were mixed for 30 s in the pre-mix step. In the coarse emulsion step 20% of the oil was added with the tip speed 5.5 m/s and the flow 0.4 kg/s while the remaining 80% was added with the tip speed 13.0 m/s and the flow 0.61 kg/s. After the oil had been added the vinegar was added and mixed for 10 s. The samples were taken at 0.11, 0.21, 0.32, 0.43, 0.53 and 0.80 normalized passages.

The results from the texture analysis for the experiments with egg yolk powder and whole egg can be seen in Figure 4.8 together with mayonnaise made with 6% liquid egg yolk. This was done to give a better comparison between the different recipes, since the egg yolk powder recipe correspond to a liquid egg yolk content of 5.6% and the whole egg recipe corresponds to a liquid egg yolk content of 3.4%. Keep in mind that the oil contents are not the same where 79% oil was used in both the egg yolk powder recipe and the whole egg recipe while the 6% liquid egg yolk contained 80.5% oil.

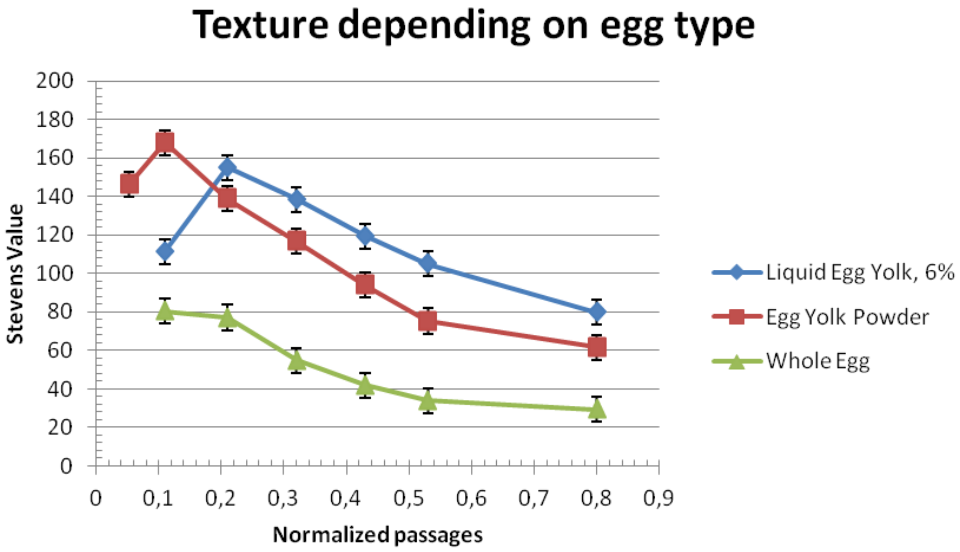

Figure 4.8: Stevens value as a function of the amount of normalized emulsification passages for mayonnaise made with 6% liquid egg yolk, egg yolk powder and liquid whole egg. The recipe containing egg yolk powder and liquid whole egg corresponds to a recipe with 5.6% and 3.4% liquid egg yolk respectively and has an oil content of 79%. The recipe with 6% liquid egg yolk has an oil content of 80.5%.

From these results it can be seen that both the recipe containing egg yolk powder and the recipe containing whole egg behaves differently than the recipe containing 6% egg yolk.

The recipe based on egg yolk powder has a higher peak even though it contains less amount of egg yolk (5.6%) than the recipe with 6% egg yolk. The peak occurs earlier and the decline in texture is steep. This shows that mayonnaise made with egg yolk powder is more sensitive to over-shear.

The mayonnaise based on whole egg showed significantly lower Stevens values at all measured points. For this type of mayonnaise no peak in Stevens value could be seen, instead there was a slight plateau before the decrease in Stevens value began. It is possible that the peak occurred earlier and was not caught in this experiment.

The droplet size for the three different recipes can be seen in Figure 4.9.

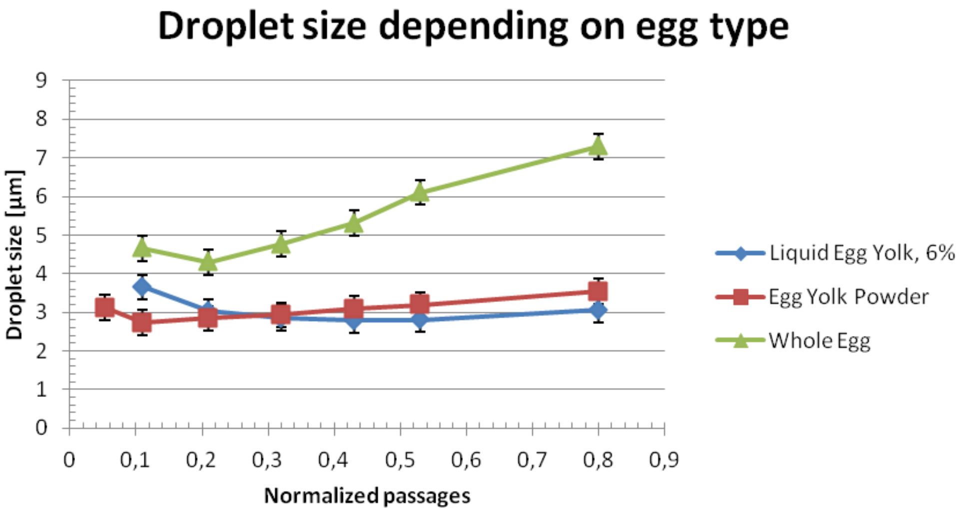

Figure 4.9: Droplet size as a function of the amount of normalized emulsification passages for mayonnaise made with 6% liquid egg yolk, egg yolk powder and liquid whole egg. The recipe containing egg yolk powder and liquid whole egg corresponds to a recipe with 5.6% and 3.4% liquid egg yolk respectively and has an oil content of 79%. The recipe with 6%liquid egg yolk has an oil content of 80.5%.

From the figures above it can be seen that the egg yolk powder is functioning very well in the beginning with a low droplet size and a high Stevens value. This suggest that egg yolk powder is a more efficient emulsifier than liquid egg yolk, which agrees very well with what was found in the literature, see section 2.1.2.2 *Emulsifying properties of egg*. The recipe based on egg yolk powder only contained 79% oil which was lower than the recipe with 6% liquid egg yolk, which contained 80.5%. According to Andersson (2015) mayonnaise with 79% oil content should have a Stevens value of around 120 and a droplet size of around 3.5 µm. However, these values were obtained using ingredients with a starting temperature of 20 °C and not 8 °C. By using the information obtained in section 4.2.2 *Change of temperature* the Stevens value would be closer to 150 while the droplet size remains unchanged at 3.5 µm, if ingredients with a starting temperature of 8 °C were used. Therefore a Stevens value of 167 and a droplet size of 2.7 µm after 0.11 normalized passages that was observed for egg yolk powder was better than expected.

The recipe containing whole egg shows a very large droplet size. All measured values are significantly higher than the values of both the 6% liquid egg yolk and egg yolk powder recipes. A small decrease in droplet size can be seen before the increase. However, the smallest droplet size achievable with this recipe was still very large. This would suggest that this recipe lacks the amount of emulsifier needed to cover all possible interfaces during emulsification. By assuming a surface load of 2 mg/m^2 (Le Denmat et al., 2000) and a mean droplet diameter of 2 µm there should be enough proteins from the egg white and the egg yolk to cover all possible interfaces. But this only holds true if all proteins show some emulsifying behavior which is probably not the case for the proteins in egg white. It is therefore reasonable to assume that this recipe does not contain enough emulsifiers.

For both the recipe containing egg yolk powder and whole egg a certain sliminess was observed. Compared to the standard recipe both mayonnaises had a long texture which made their consistency more slimy and elastic. This is not a wanted quality of mayonnaise and it is therefore possible to say that this mayonnaise do not fulfill the requirements of proper mayonnaise.

4.2.4 Increased oil content

In order to find the highest oil content that gives mayonnaise and to investigate the stability of mayonnaise with increased oil content, different oil contents were tested starting at 94%. When the oil content was increased the amount of water phase was decreased to keep the batch size of 33 kg constant. The fraction within the water phase was kept relative to each other which mean that the total amount of egg yolk was decreased. The experiments were started with an oil content of 94% which was decreased until mayonnaise was obtained. The remaining oil contents that were tested were 88%, 87%, 86%, 85%, 84%, 83%, 82% and 80.5%. The recipe with 80.5% oil is the standard recipe and the remaining recipes for the experiments with increased oil content can be found in Appendix 8.4 *Recipes for increased oil content*.

The ingredients for the water phase were mixed for 30 s in the pre-mix step. In the coarse emulsion step 20% of the oil was added with the tip speed 5.5 m/s and the flow 0.4 kg/s while the remaining 80% was added with the tip speed 13.0 m/s and the flow 0.61 kg/s. After the oil had been added the vinegar was added and mixed for 10 s. The samples were taken at 0.11, 0.21, 0.32, 0.43, 0.53 and 0.80 normalized passages. For this experiment the *standard pipe* was used.

The recipe with 94% oil broke during the oil addition step and therefore underwent a coarse emulsion phase inversion. The recipes with 88% and 87% oil remained and O/W emulsion through the coarse emulsion step but broke during the emulsification step before 0.11 normalized passages was reached. Both of these recipes underwent emulsification phase inversion. The highest oil content that gave mayonnaise after 0.11 normalized passages was 86%.

The experiments with 80.5-86% oil gave mayonnaise and both texture and droplet size could be measured. The results from the texture analysis can be seen in Figure 4.10

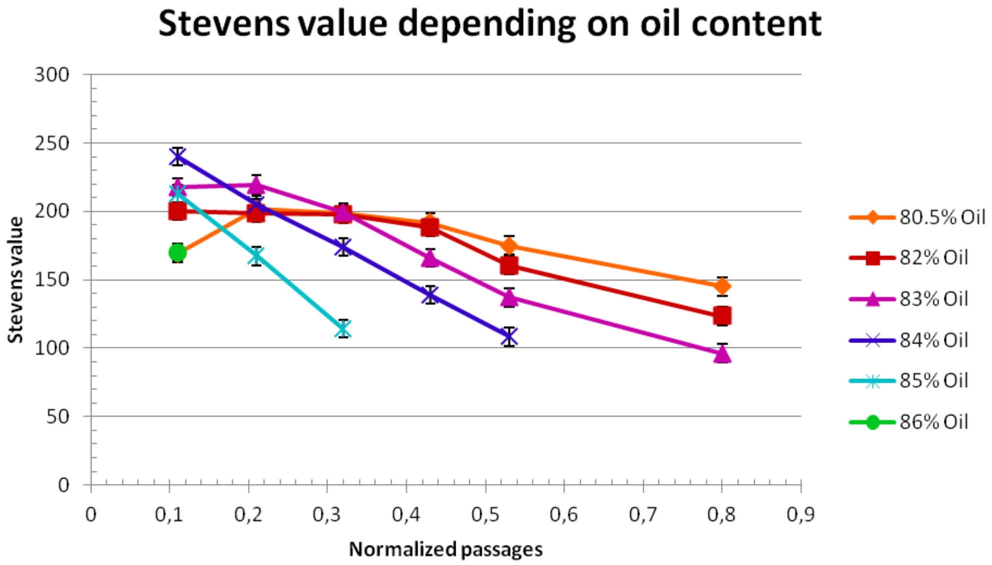

Figure 4.10: Stevens value as a function of the amount of normalized emulsification passages for mayonnaise made with 80.5%, 82%, 83%, 84%, 85% and 86% oil.

The oil content of 80.5% is the standard recipe and the peak in Stevens value occur around 0.3 normalized passages. As the oil content is increased the peak occurs at fewer passages and the decline in texture is steeper. For 80.5%, 82%, 83% and 84% a trend could be observed where an increase in dispersed phase gives a higher maximum Stevens value. The high Stevens value can be explained by the increase of dispersed phase, where more oil results in more droplets which gives more junction points and thereby a firmer texture. The trend of increased dispersed phase giving a firmer texture does not hold for 85% and 86% oil. It is possible that for mayonnaise with this very high fraction of dispersed phase, the peak in texture occurs too early to be caught in these experiments.

Mayonnaise made with 83% oil was the highest oil content that gave proper mayonnaise, since this mayonnaise still followed the expected texture-curve. The peak in Stevens value for mayonnaise with 84-86% could not be captured in these experiments and as a result the mayonnaises measured are labeled as over-sheared. Mayonnaise with a higher oil content show more sensitivity towards over-shear, which can be seen by a steeper decline in texture with increasing oil content. They also undergo emulsification phase inversion where higher oil content results in an earlier phase inversion.

The results from the droplet size analysis can be seen in Figure 4.11.

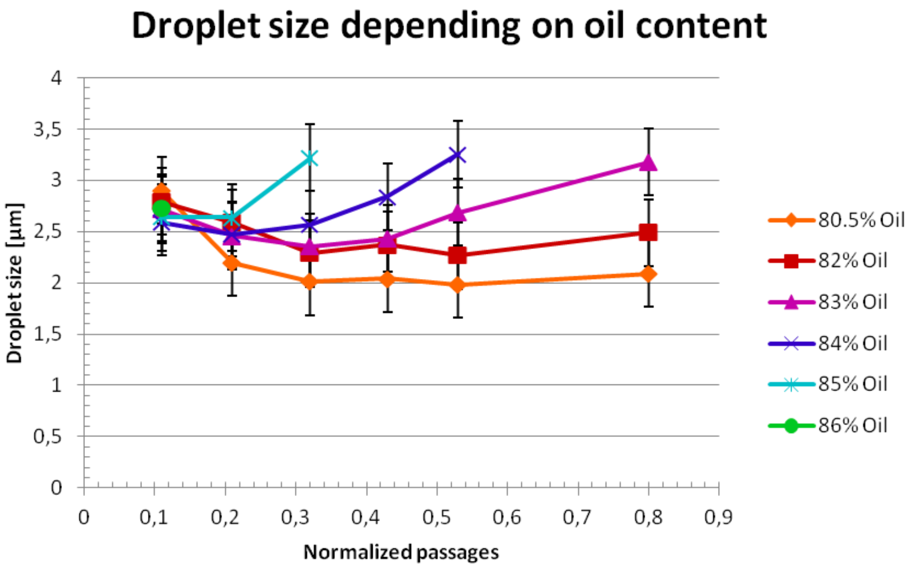

Figure 4.11: Droplet size as a function of the amount of normalized emulsification passages for mayonnaise made with 80.5%, 82%, 83%, 84%, 85% and 86% oil.

From Figure 4.11 it can be seen that higher oil content results in a faster increase in droplet size, with increased number of passages. In the standard recipe, with 80.5% oil, there is only a slight increase in droplet size with increased passages which indicates that the droplets are stable at this oil content. The increase in droplet size at the high oil contents show that there was coalescence amongst the droplets indicating an unstable emulsion.

As previously mentioned the highest oil content that gave proper mayonnaise was 83%. This is supported by the droplet size measurements which also show that 83% oil is the last concentration that shows the same kind of pattern as proper mayonnaise, even though it rises quite fast. According to literature the highest oil content that can be used is 84%, see section 2.2.4 *Phase inversion*. This is close to the value obtained here which makes the results more reliable.

There is a risk when increasing the oil content, and consequently decreasing the water phase, that the amount of emulsifier in the mayonnaise become so low that the amount of interface that can be formed is affected. By assuming that the adsorbed layer of egg yolk protein is 2 mg/m^2 (Le Denmat et al., 2000) there is enough protein to cover all interfaces of a system containing droplets with a diameter of 2 µm, even in the case of 86% oil.

4.3 Catastrophic phase inversion of mayonnaise

4.3.1 Kitchen-made mayonnaise made with immersion blender

To test the sensitivity of a mayonnaise made with the standard recipe a kitchen scale experiment was made using an immersion blender. The recipe was scaled down to a batch size of 0.5 kg. The effect of the position of the mixer head was investigated in several different set-ups. The mixer head was either lifted upwards to keep it just below the surface, kept still at the bottom, rotated at the bottom or was circulated in the vessel according to cookbook recommendations.

When the mixer head was raised to remain just below the surface, mayonnaise was initially formed. When about a third of the oil had been added the mayonnaise broke. The immersion blender was not strong enough to create a flow profile that reached the entire vessel, see Figure 4.12.

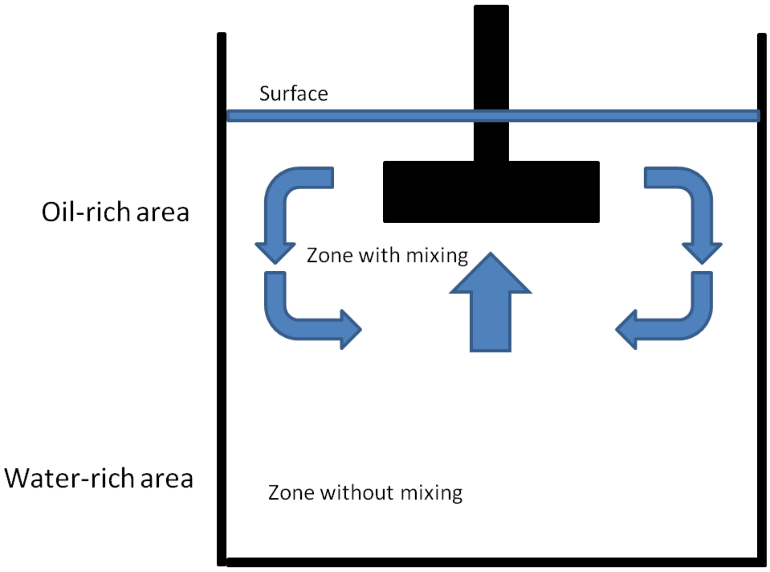

Figure 4.12: The flow in the vessel when the mixer head of the immersion blender was kept just below the surface.

When the mixer head was kept still at the bottom mayonnaise was formed around it. As the amount of oil increased, an oil layer started to form on top of the mayonnaise, see Figure 4.13. The same problem occurred when the blender was moved around the bottom.

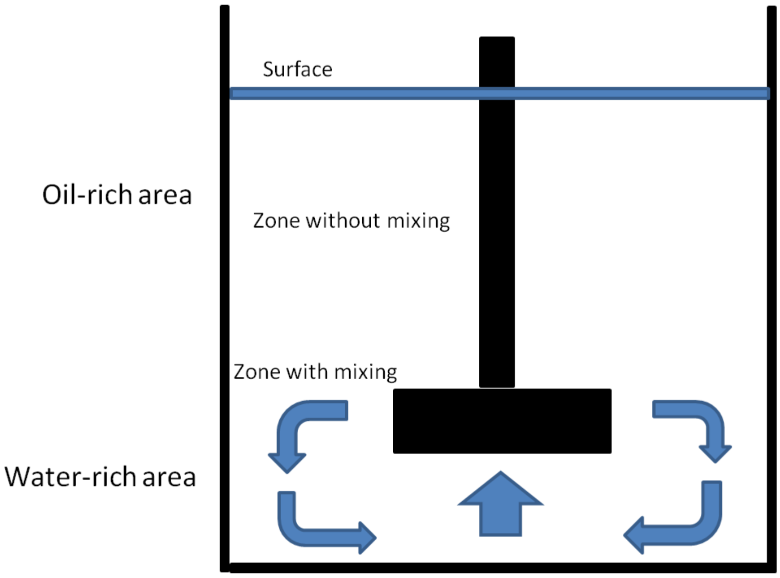

Figure 4.13: The flow in the vessel when the mixer head of the immersion blender was kept at the bottom of the vessel.

Cookbooks recommended moving the immersion blender on the bottom and then lift it only a little bit and then lowered again. When the blender was moved in this type of pattern mayonnaise was made in the entire vessel. However, in one experiment the mixer head was lifted a bit too high so that it reached an oil-rich area and the mayonnaise broke. The emulsion could not be saved by trying to bring the mixer head back to the already formed mayonnaise. Instead this resulted in broken mayonnaise in that part as well.

4.3.2 Coarse emulsion tip speed

Between which oil inlet flow rates the phase inversion point is situated for different coarse emulsion tip speeds was investigated in this experiment. The ingredients for the water phase were mixed for 30 s in the pre-mix step. In the coarse emulsion step the tip speeds 1.4, 2.8, 8.3, 13.0, 19.3 and 24.9 m/s were tested. For the experiments made with 8.3, 13.0, 19.3 and 24.9 m/s the first 20% of the oil were added using the tip speed of 5.5 m/s and then the tip speed was increased to the higher speed for the last 80% oil. For the tip speeds 1.4 and 2.8 m/s the tip speed was kept constant through the entire coarse emulsion step. The oil inlet flow rates ranged from 0.61 kg/s to 4.9 kg/s. The first 20% of the oil was added at a low speed, 1 kg/s, and was then increased to the higher flow rate that was to be tested. When an oil inlet flow rate to be tested was 0.61 kg/s then the initial flow rate were about 0.4 kg/s instead of 1 kg/s. After the oil had been added the vinegar was added and mixed for 10 s. Samples were taken at 0.11, 0.21, 0.32, 0.43, 0.53 and 0.80 normalized passages. For this experiment the *standard pipe* was used.

It was also tested if it is possible to get mayonnaise if the oil is put into the mixer together with the other ingredients from the start. This was done to test if there is a lower limit of the coarse emulsion tip speed. Since the oil was added from the start no coarse emulsion step was used. Instead the emulsification step was started directly. Between which flow rates the phase inversion points are situated are shown in Figure 4.14.

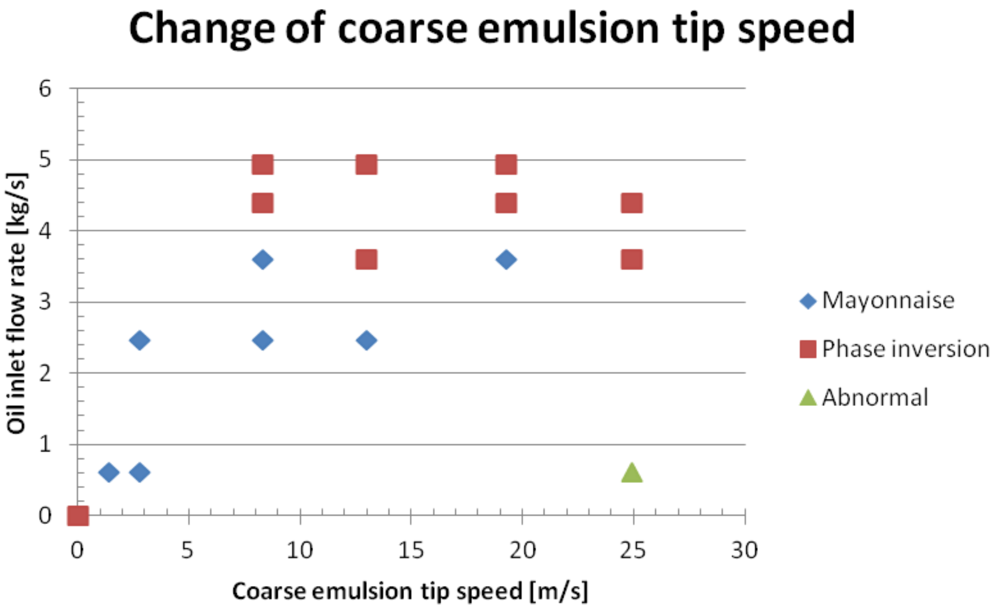

Figure 4.14: Plot of when the mayonnaise breaks depending on oil inlet flow rate and coarse emulsion tip speed. The blue rhombi represent the runs that resulted in a proper mayonnaise. The red squares represent the runs that resulted in a broken, or phase inversed, mayonnaise. The green triangle represent runs that resulted in mayonnaise with a high initial Stevens value but a very fast decrease compared to regular mayonnaise. The red square in origo is an experiment run with all the ingredients added without any stirring.

From Figure 4.14 it can be seen that if the oil is added too quickly the emulsion breaks and there is a coarse emulsion phase inversion. This agrees well with the recipe instructions that can be found in household cookbooks. No apparent correlation between the coarse emulsion tip speed and the oil inlet flow rate could be found. The same oil inlet flow rate can be used at a tip speed of 8.3 m/s and 19.3 m/s to get mayonnaise while a slightly lower oil inlet flow rate could be used for 13.0 m/s. The quality of the final mayonnaise formed during the emulsification step was not affected by the different coarse emulsion tip speeds and oil inlet flow rates but had the same texture and droplet size for all samples. It is logical to assume that a higher tip speed would allow for a higher oil inlet flow rate, since the stirring is more intense and could disperse the oil more efficiently. This was, however, not observed during these experiments and consequently other factors must be responsible for limiting the highest oil inlet flow rate that can be used.

From observation of the process it was seen that it was the first part of the oil addition that was important, if the emulsion did not break then it never broke.

In one experiment oil was added to the mixer at the start together with all the other ingredients, instead of having a coarse emulsification step. This did not work and the mayonnaise broke. This experiment is represented by the red square in origo in Figure 4.14. To make mayonnaise it is necessary to add the oil in a coarse emulsion step with stirring.

At the very low coarse emulsion tip speed of 1.4 m/s mayonnaise could be made. In this case, the mayonnaise was runny after the coarse emulsion step but after the emulsification step it had achieved the same texture as regular mayonnaise. A comparison of regular mayonnaise and the mayonnaise made at 1.4 m/s can be seen in Figure 4.15. This is interpreted as a very large oil drop size and it is thought that the maximum oil inlet flow rate that can be used at 1.4 m/s is lower than for the other tip speeds.

Figure 4.15: Mayonnaise after the coarse emulsion step. Left: Standard mayonnaise made with the coarse emulsion tip speed of 8.3 m/s. Right: Mayonnaise made with the coarse emulsion tip speed of 1.4 m/s.

When the highest coarse emulsion tip speed was used (24.9 m/s) there was a change in the flow field in the mixer where the liquid had a parabolic shape and the surface was below the rotor. This affected the emulsion formation. When a low oil inlet flow rate was used mayonnaise was obtained but it was abnormal because of a very firm initial texture that decreased rapidly with increasing passages. Because of the change in flow field the tip speed of 24.9 m/s is not recommended for production of proper mayonnaise.

4.3.3 Ratio of oil added at lower flow rate

In this experiment the ratio of oil that was added slowly in the beginning was changed from the standard 20% to 0% and 40%. The ingredients for the water phase were mixed for 30 s in the pre-mix step. In the standard set-up 20% of the oil was added with the tip speed 5.5 m/s and the flow 1 kg/s while the remaining 80% was added with the tip speed 8.3 m/s and the flow being varied. In this experiment this ratio was first changed to 0% of the oil added at 1 kg/s which means that 100% was added at a fast flow rate that was varied. The ratio was also changed to 40% of the oil added at 1 kg/s and the remaining 60% being added at a varied flow rate. After the oil had been added the vinegar was added and mixed for 10 s. Samples were taken at 0.11, 0.21, 0.32, 0.43, 0.53 and 0.80 normalized passages. For this experiment the *standard pipe after flow changed* was used. Between which flow rates the phase inversion points are situated are shown in Figure 4.16.

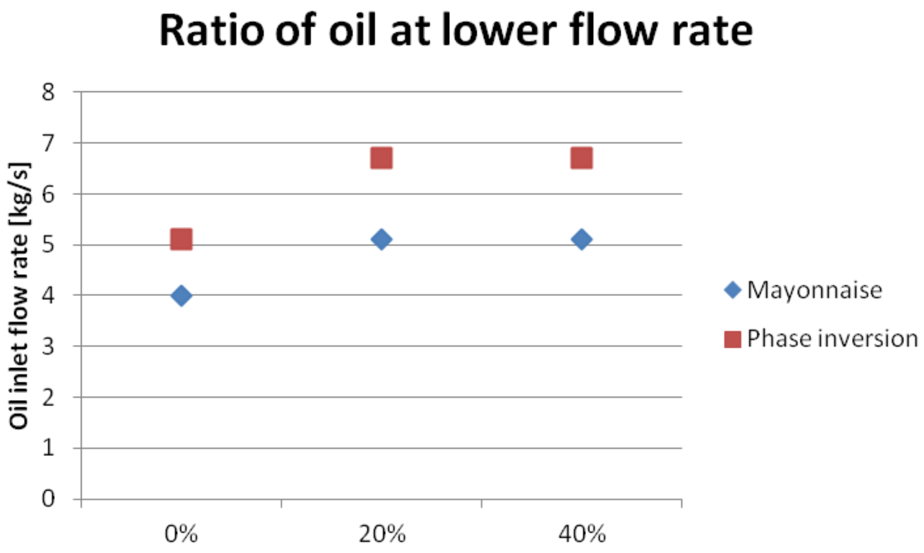

Figure 4.16: Plot of when the mayonnaise breaks depending on oil inlet flow rate and percentage of oil let in slowly. The blue rhombi represent the runs that resulted in a proper mayonnaise. The red squares represent the runs that resulted in a broken, or phase inversed, mayonnaise.

From Figure 4.16 it can be seen that higher oil flow rates can be used when taking in a part of the oil at lower rate before going to a higher flow rate compared to using the higher flow rate from the start. This shows that mayonnaise is more sensitive in the beginning than it is after the first emulsion has been formed. No difference in the oil inlet flow rate could be seen between letting in 20% or 40% of the oil slowly.

That a lower oil flow rate is needed in the beginning than when the emulsion has formed to avoid a phase inversion agrees well with the tips found in recipes for kitchen made mayonnaise. Here, it is often stated that the oil flow can be increased after the mayonnaise has started to thicken.

The results obtained here can be used to optimize the time needed to add the oil in the mixer. When 0% of the oil was added slowly it took 7 s to let in all the oil. When 20% of the oil was let in slowly, with an initial oil flow of 1 kg/s, the addition of oil took 9 s. When adding 40% of the oil slowly it took 14 s.

Since the 0% experiment showed that mayonnaise can be made from an oil inlet flow rate of 4.0 kg/s, this flow rate could theoretically be used for the first 20% oil added to the mixer. Since mayonnaise could be obtained with a flow rate of 5.1 kg/s after 20% of the oil had been added slowly, this flow can be used for the remaining 80% oil. Using a flow of 4.0 kg/s for the first 20% oil and a flow rate of 5.1 kg/s for the remaining 80% oil would shorten the total oil addition time to 6 s. With the batch size used in these experiments the difference in time does not make a big difference but when large batches are made the time becomes more important. For example if a batch size of 2000 kg is used the total oil addition time for adding 0% of the oil slowly would be 6.7 min while the optimized addition time would be 5.6 min.

4.3.4 Design of oil opening

In the experiments done with high oil inlet flow rates the amount of splashing was high. This is shown in Figure 4.17.

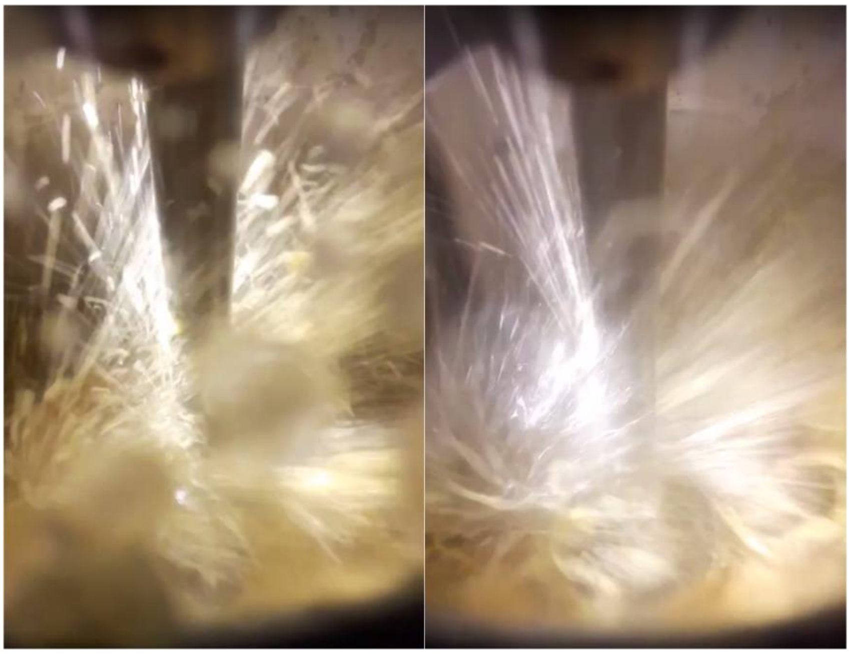

Figure 4.17: Splashing when the oil enters the mixer, a second before the oil covers the window. Left: Oil inlet flow rate of 3.6 kg/s and tip speed 8.3 m/s. This experiment resulted in proper mayonnaise. Right: Oil inlet flow rate of 4.4 kg/s and tip speed 8.3 m/s. This resulted in broken mayonnaise.

Since the velocity of the oil is high the oil enters the mixer in a stream that goes through the mayonnaise and hits the lid from which the oil rains down into the mixer. If a higher or lower velocity of the added oil affect the oil inlet flow rate that can be used was tested by changing the size of the oil inlet pipe. This allows the oil inlet velocity (m/s) to be changed while the oil inlet flow rate (kg/s) remains the same. With a smaller opening the oil enters with a higher velocity and with a wider opening the oil enters with a lower velocity. The higher velocity was achieved by using the butterfly valve in the bottom of the mixer to set the flow. This resulted in a crescent shaped oil inlet with a smaller area than if the valve was completely open. This is referred to as the *smaller opening*. To get a lower velocity a larger opening in the mixer was used where the butterfly valve was opened completely. This is referred to as the *larger pipe*. More details on the pipes and openings used can be found in Appendix 8.5 *Pipes used for oil addition*.

In the beginning a pipe without connection (*standard pipe*) was used and in order to connect it to the larger opening a connection was added. When the connection was added the valve was affected and the oil flow rate through the pipe was changed. New experiments were made using the altered pipe (*standard after flow changed*).

In the experiments the ingredients for the water phase were mixed for 30 s in the pre-mix step. In the coarse emulsion step 20% of the oil was added with the tip speed 5.5 m/s and the flow 1 kg/s while the remaining 80% was added with the tip speed 8.3 m/s and the flow being varied. After the oil had been added the vinegar was added and mixed for 10 s. Samples were taken at 0.11, 0.21, 0.32, 0.43, 0.53 and 0.80 normalized passages. Between which flow rates the phase inversion point is situated are shown in the Figure 4.18.

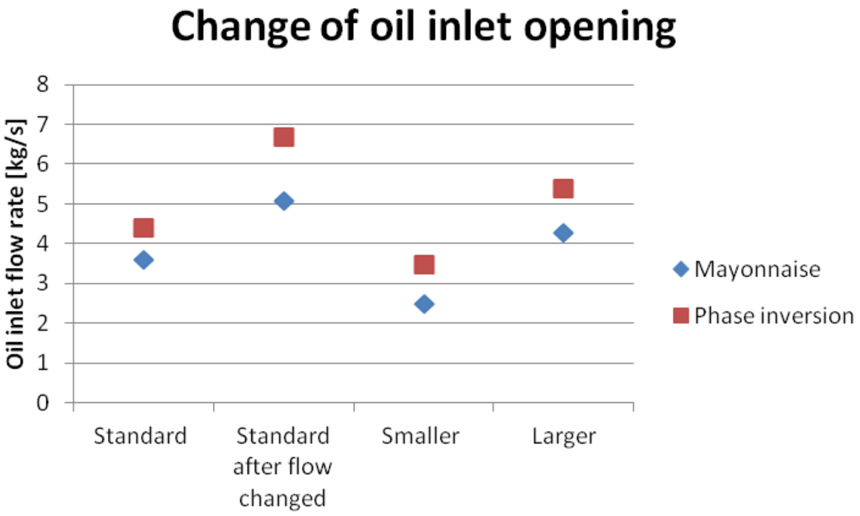

Figure 4.18: Plot of when the mayonnaise breaks depending on oil inlet flow rate and oil inlet opening. The blue rhombi represent the runs that resulted in a proper mayonnaise. The red squares represent the runs that resulted in a broken, or phase inversed, mayonnaise.

The smaller opening required a lower oil inlet flow rate to be used compared to the others. No change could be seen form changing to a larger opening.

Unexpectedly, there was a change of the flow in the standard pipe that resulted in that mayonnaise could be made at a higher oil inlet flow rate than before, as can be seen in Figure 4.18. The area of the oil inlet opening remained the same but since the mayonnaise was affected the flow profile must have changed. It is possible that the oil does not fill the entire pipe when it enters the mixer, but instead it takes up less area and has a higher speed. After the flow profile had changed it is possible that the oil fills up more of the pipe which allows the oil to enter at a lower speed than before. To test this theory the velocity of oil and the height of the liquid column was calculated and compared to the splashing pattern observed.

The speed of the flow rates marked in Figure 4.18 was calculated. When the opening was circular and the area known, the continuity equation, Equation 4.2, was used to calculate the velocity:

$$v = \frac{Q}{A \cdot \rho} \qquad [4.2]$$

Where v is the velocity, Q is the mass flow of oil, A is the area of the pipe and ρ is the density of the oil (920 kg/m^3). The smaller opening used was crescent shaped and the velocity was calculated from the pressure drop, assuming the pressure was converted to velocity in the valve. This was done using Equation 4.3:

$$\Delta P = \frac{\rho \cdot v_2^2}{2} \qquad [4.3]$$

Where ΔP is the pressure drop which is assumed to be 500 mbar, ρ is the density of the oil and v is the velocity. The calculated velocities are shown in Table 4.4.

The heights that the liquid columns would have reached if they had not hit the roof of the mixer can be calculated with Equation 4.4:

$$\frac{\rho v^2}{2} = \rho g h \qquad [4.4]$$

Where ρ is the density, v is the velocity, g is the gravitational acceleration and h is the height of the liquid column. The calculated heights are shown in Table 4.4.

Table 4.4: The oil flow, the velocities and the heights of the liquid columns for the different openings.

Opening		Oil flow [kg/s]	Oil velocity [m/s]	Liquid column height [m]
Standard	Proper	3.6	1.9	0.18
	Broken	4.4	2.3	0.27
Standard after flow changed	Proper	5.1	2.7	0.37
	Broken	6.7	3.6	0.66
Smaller	Proper	2.5	10.5	5.6
	Broken	3.5	10.6	5.7
Larger	Proper	4.3	1.5	0.11
	Broken	5.4	1.9	0.18

From Table 4.4 it can be seen that the speed for the smaller pipe is between 4-7 times larger than the other velocities. However, the oil inlet flow rate that can be used to get mayonnaise is only 1.5 to 2 times lower than the flow rates for the other openings. This indicates that the oil flow is of greater importance than the oil inlet speed.

From Table 4.4 it can also be seen that only the liquid columns from the smaller opening and the 6.7 kg/s flow rate in the standard after flow changed should be able to reach the lid 0.5 m up, theoretically. In reality all of the flow rates gave splashing that reached the lid, resulting in a minimum velocity of 3.1 m/s. This shows that the actual speed is higher than calculated which indicates that the pipes were not completely filled with oil after the valve, as was suspected when the flow changed in the standard pipe.

If the pipe is not entirely filled when the oil is added but instead enters in thin jet streams the system would be more unstable and sensitive to small changes in the pipe. When the connection was added to the pipe there was a slight change in the diameter at the connection site. This small change could be enough to change the flow pattern in the pipe and thereby also the flow into the mixer. How the streams could be affected by the connection is schematically illustrated in Figure 4.19. To avoid having a system that is unstable and sensitive to small changes it is important to make sure that the oil-addition pipe is filled. Alternatively, the oil addition can be done by pumping in the oil instead.

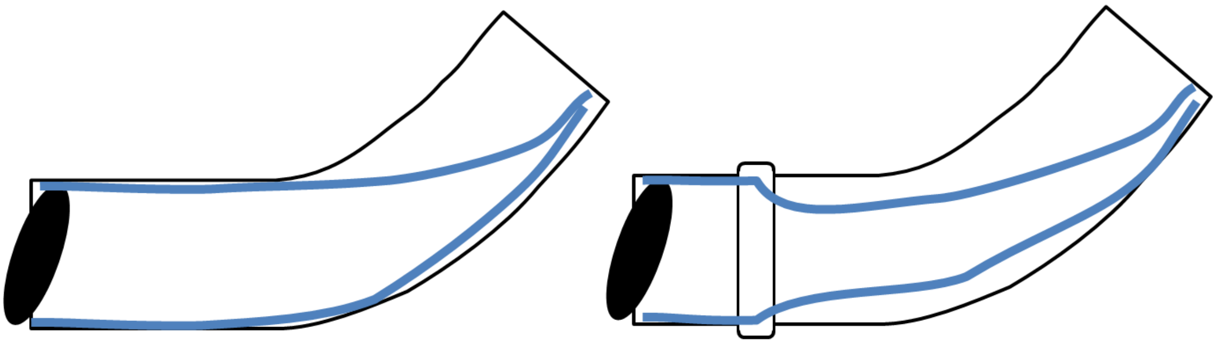

Figure 4.19: Possible flows in pipe. Left: Before the connection was added (standard). Right: After the connection was added (standard after flow changed).

If the actual velocity of the oil is higher than the calculated velocity it is possible that the difference in speed could be closer to the same ratio that was observed in the flow rates difference. This would mean that speed would be as important as the flow rate. To investigate this, experiments with controlled speeds must be performed.

4.3.5 Change of temperature

The temperature of the ingredients was changed to different combinations, see Table 4.5.

Table 4.5: The temperatures tested for the oil and the remaining ingredients. The experiment with 8 °C oil and 8 °C ingredients is the standard.

Oil temperature [°C]	Ingredient temperature [°C]
8	8
8	20
20	20

The ingredients for the water phase were mixed for 30 s in the pre-mix step. 20% of the oil was added with the tip speed 5.5 m/s and the flow 1 kg/s while the remaining 80% was added with the tip speed 8.3 m/s and the flow being varied. After the oil had been added the vinegar was added and mixed for 10 s. Samples were taken at 0.11, 0.21, 0.32, 0.43, 0.53 and 0.80 passages. For this experiment the *standard pipe after flow changed* was used. Between which flow rates the phase inversion points are situated are shown in Figure 4.20.

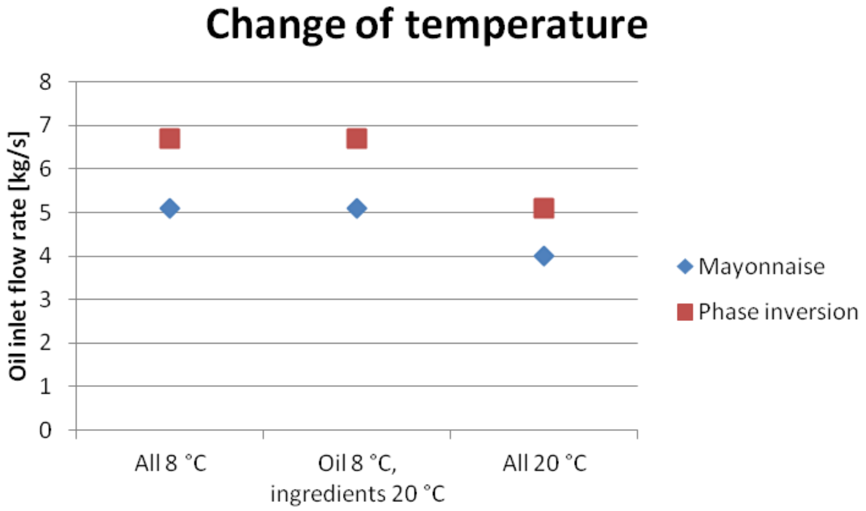

Figure 4.20: Plot of when the mayonnaise breaks depending on oil inlet flow rate and temperature of ingredients. The blue rhombi represent the runs that resulted in a proper mayonnaise. The red squares represent the runs that resulted in a broken, or phase inversed, mayonnaise.

From the figure it can be seen that mayonnaise made with warm ingredients became more sensitive. Mayonnaise made with only cold ingredients and mayonnaise made with cold oil and remaining ingredients warm showed no difference in sensitivity. In order to avoid coarse emulsion phase inversion it is recommended to use cold (8 °C) oil.

Cookbooks recommend using ingredients with the same temperature to avoid a phase inversion. The results from these experiments do not support this claim.

4.3.6 Decreased egg yolk content

In the experiment the amount of liquid egg yolk was decreased from 8.5% to 6%. The recipe can be found under section 3.1.2 *Recipes*. The amount of salt and water was compensated to keep both the salinity and the amount of water phase constant. The ingredients for the water phase were mixed for 30 s in the pre-mix step. 20% of the oil was added with the tip speed 5.5 m/s and the flow 1 kg/s while the remaining 80% was added with the tip speed 8.3 m/s and the flow being varied. After the oil had been added the vinegar was added and mixed for 10 s. Samples were taken at 0.11, 0.21, 0.32, 0.43, 0.53 and 0.80 normalized passages. For this experiment the *standard pipe after flow changed* was used. Between which flow rates the phase inversion point is situated are shown in Figure 4.21.

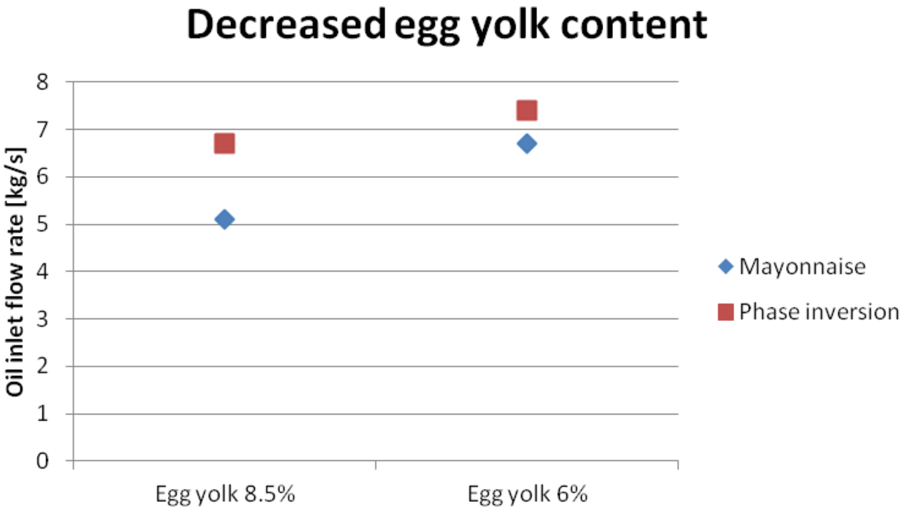

Figure 4.21: Plot of when the mayonnaise breaks depending on oil inlet flow rate and liquid egg yolk content. The blue rhombi represent the runs that resulted in a proper mayonnaise. The red squares represent the runs that resulted in a broken, or phase inversed, mayonnaise.

As can be seen from the figure a mayonnaise made with decreased egg yolk content is less sensitive than mayonnaise made from the standard recipe. A higher oil inlet flow rate could be used. This is unexpected since the egg yolk protein is the emulsifier and having more emulsifier present in the system should be beneficial to the stability of the emulsion.

4.3.7 Change of egg type

In the experiment the egg type was changed from liquid egg yolk to egg yolk powder. The recipe can be found under section 3.1.2 *Recipes*. The ingredients for the water phase were mixed for 30 s by hand in the mixer vessel and then for 30 s by the mixer. This was repeated two more times until no more lumps were visible. In the coarse emulsion step in the standard run 20% of the oil was added with the tip speed 5.5 m/s and the flow 1 kg/s while the remaining 80% was added with the tip speed 8.3 m/s and the flow being varied. However, in this experiment very low oil flow rates were used so the same flow rate was used for the first 20% at 5.5 m/s tip speed and for the remaining 80% at 8.3 m/s tip speed. After the oil had been added the vinegar was added and mixed for 10 s. Samples were taken at 0.11, 0.21, 0.32, 0.43, 0.53 and 0.80 normalized passages. For this experiment the *standard pipe after flow changed* was used. Between which flow rates the phase inversion point is situated are shown in Figure 4.22.

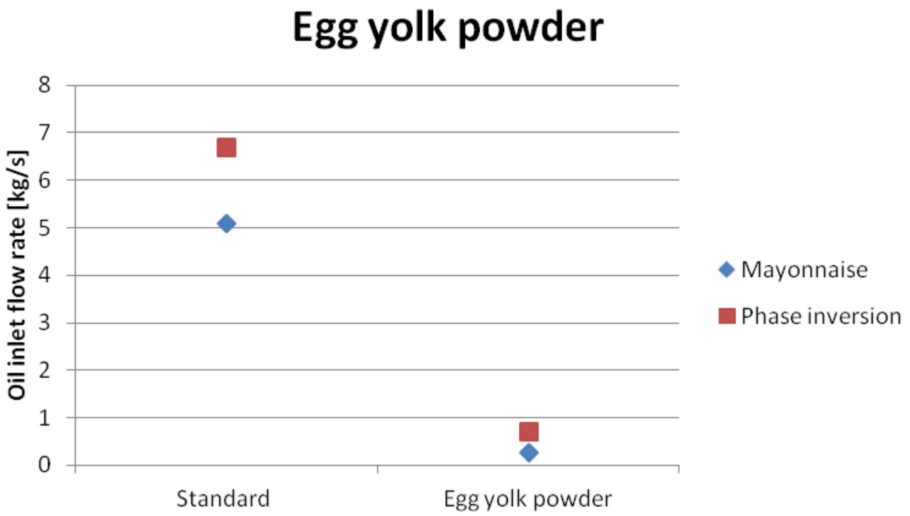

Figure 4.22: Plot of when the mayonnaise breaks depending on oil inlet flow rate and type of emulsifier. The blue rhombi represent the runs that resulted in a proper mayonnaise. The red squares represent the runs that resulted in a broken, or phase inversed, mayonnaise.

The oil inlet flow rate needed to be very low in order to get mayonnaise with the egg yolk powder recipe. Even the low speed of 0.73 kg/s gave a phase inversion.

There is a possibility that the proteins in the powder need more time to dissolve in the water phase to be able to act as an effective emulsifier at the oil-water interface. To investigate this optical microscopy pictures were taken of the dispersed egg yolk powder, the premix phase and the mayonnaise. An optical microscope (Olympus Light BX50 with a FireWire 400 Color Industrial Camera DFK 41AF02, Sony) with the objective UMPlanFl 10x/0.3 was used. The microscopy pictures are shown in Figure 4.23.

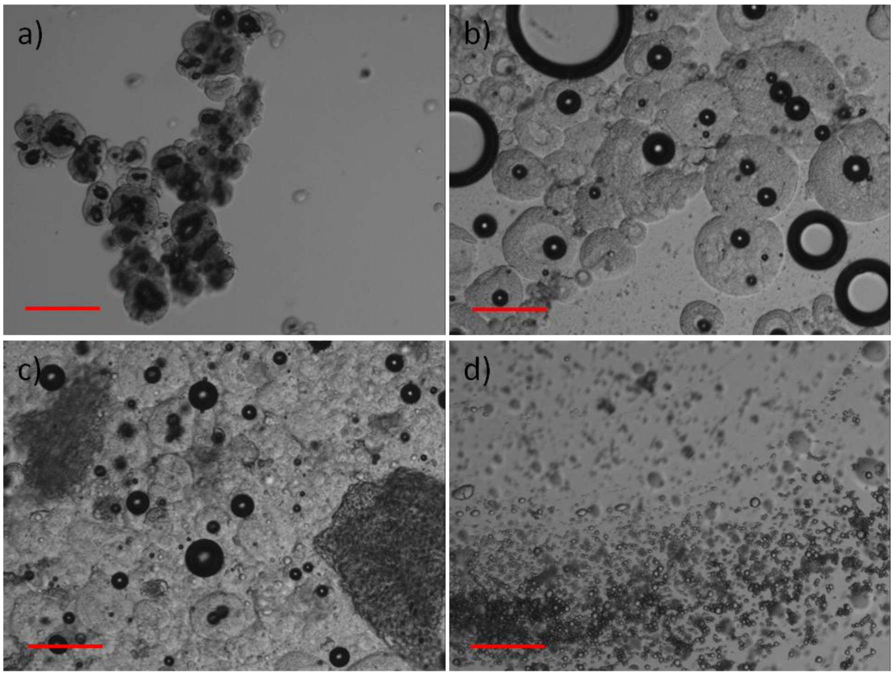

Figure 4.23: Microscopy pictures where the red bar is 100 µm: a) Egg yolk powder dispersed in oil. b) Pre-mix phase mixed for 3 min. The big black circles are air bubbles. c) Pre-mix phase mixed for 30 min. The dark objects are air bubbles and mustard particles. d) Mayonnaise made with egg yolk powder.

From Figure 4.23 it can be seen that the powder structure from the egg yolk is present in the premix phase (b). Prolonging the time egg yolk powder spends in the water to 30 min (c) did not dissolve the powder structure. When the mayonnaise has been formed the powder structure has been broken up by the high shear and is no longer visible in the microscopy picture (d). The remaining powder structure in the pre-mix phase can explain why such a low oil inlet flow rate was needed, since the protein was not freely available to act as an emulsifier.

5.3.8 Compilation of changes

The effects of different changes, in both recipe and set-up, on the sensitivity of mayonnaise to undergo coarse emulsion phase inversion have been tested. Figure 4.24 show a summary of all effects tested in the same order as they have been presented in the report.

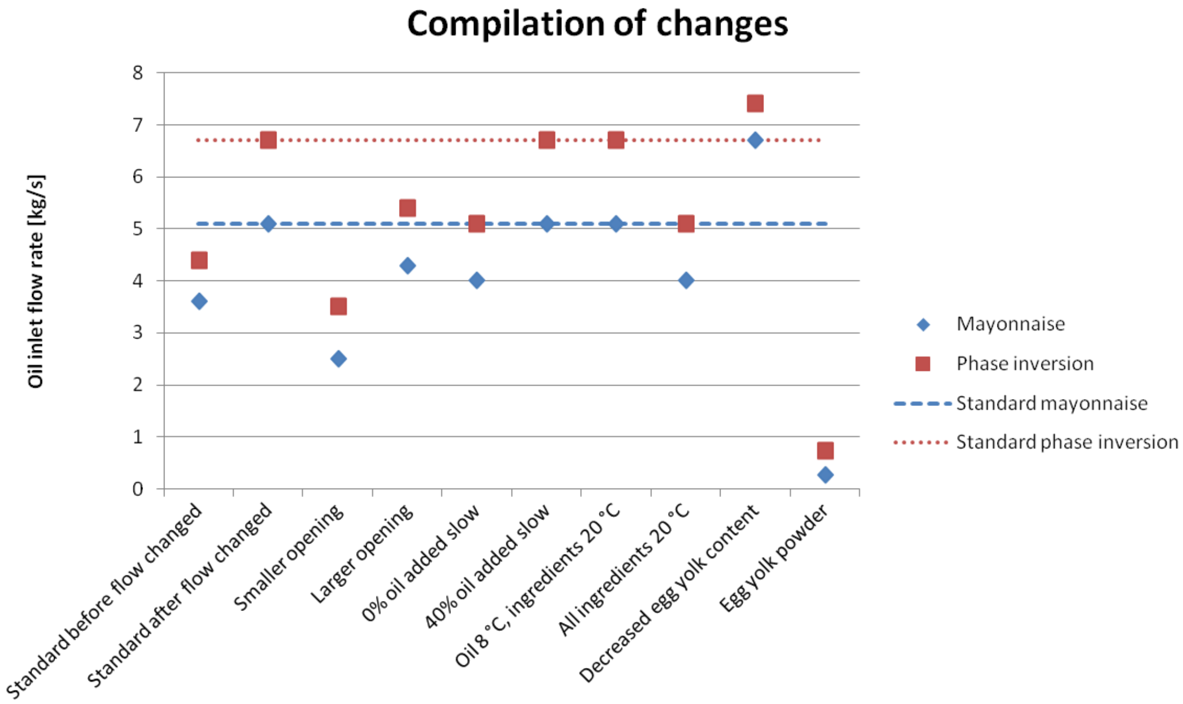

Figure 4.24: Summary of when the mayonnaise breaks depending on oil inlet flow rate. The blue rhombi represent the runs that resulted in a proper mayonnaise. The red squares represent the runs that resulted in a broken, or phase inversed, mayonnaise. The blue dashed line represent highest oil inlet flow rate that gave mayonnaise for the standard settings. The red dotted line represent lowest oil inlet flow rate that resulted in phase inversion for the standard settings.

From Figure 4.24 it can be seen that the changes that made the mayonnaise more sensitive to phase inversion were: using a smaller oil inlet opening, adding 0% of the oil slowly, using only warm ingredients and using a recipe based on egg yolk powder. The largest negative impact on the phase inversion point was found when using the recipe containing egg yolk powder. Since the difference in phase inversion sensitivity is very large, it is of great importance to use a low oil inlet flow rate when producing mayonnaise made with egg yolk powder. Using a smaller oil inlet opening gave the second largest negative impact on the phase inversion point. Adding 0% of the oil slowly and using only warm ingredients gave a phase inversion at a lower oil flow rate, but the difference from the standard was not that great.

The only change that made the mayonnaise less sensitive to coarse emulsion phase inversion was decreasing the egg yolk content. The difference in the oil inlet flow rate that could be used was not large. No reasonable explanation to why this would make the mayonnaise less sensitive could be found.

The phase inversion point is located somewhere between the proper- and broken mayonnaise points in Figure 4.24 but exactly where is not known. There is probably a deviation in the location of the phase inversion point but since the span is large this deviation should be smaller than size of the interval. Because of the large span, it is possible that there is a difference of the phase inversion point between some parameter changes that cannot be seen in these experiments. However, the position of those phase inversion points would be close to the standard phase inversion point and will therefore not affect the method of how mayonnaise is made.

5 Discussion

5.1 Over-shear

The quality of mayonnaise is changing during the emulsification process, exhibiting an optimum with both a peak in texture and a minimum in droplet size. Mayonnaise that has been processed well beyond the optimum becomes over-sheared and is characterized by a lower Stevens value and a larger droplet size. This seems to occur more rapidly for mayonnaises produced with a higher tip speed, see left side of Figure 5.1. A reasonable assumption can therefore be that over-shear is related to the amount of shear applied during the emulsification step. When mayonnaise is exposed to high shear for an extended period of time the protein in egg yolk seems to be destroyed irreversibly, resulting in a lower Stevens value and larger droplet size. As the oil content in mayonnaise is increased the mayonnaise becomes more and more sensitive to over-shear, see right side of Figure 5.1. With an increase in dispersed phase more junction points between droplets exists giving a more firm texture and a higher viscosity. A higher viscosity could make the shear more intense resulting in a more rapid destruction of egg yolk proteins. If the conclusion with destruction of egg yolk proteins is followed to the end, there should be a point in time where all the egg yolk proteins have been destroyed and emulsification phase inversion occurs. This was observed in the experiments with an oil content higher than 83%.

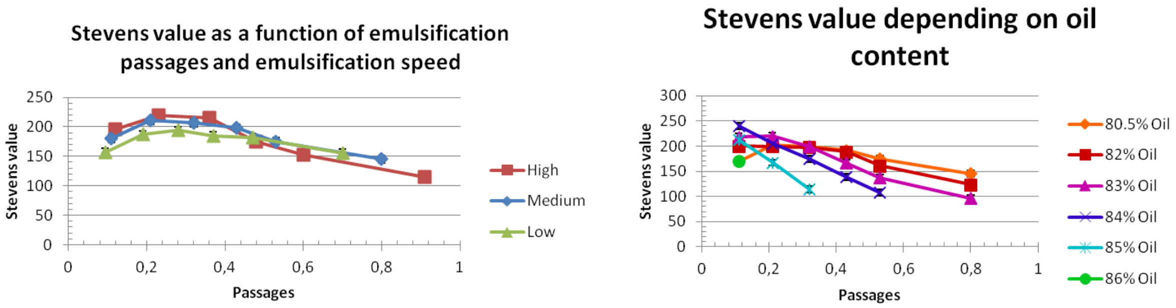

Figure 5.1: Texture curves for mayonnaise. Left: Mayonnaise produced with different emulsification tip speed (low, medium and high). Right: Mayonnaise produced with increased oil content (80.5, 82, 83, 84, 85, 86% oil).

5.2 Phase inversion

5.2.1 Emulsification phase inversion

Emulsification phase inversion is when the phase inversion occurs during the emulsification step. This means that mayonnaise have already been formed before it breaks. This was observed in the experiments with high oil content (84-86% oil), section 4.2.4 *Increased oil content* and in the case of abnormal mayonnaise obtained in section 4.3.2 *Oil inlet flow rate*.

When high oil content was used there was a high initial Stevens value followed by a steep decline in texture, see right side of Figure 5.1. This behavior is abnormal for mayonnaise and is discussed in section 5.1 *Over-shear*. The many junction points in mayonnaise with a high oil content makes the system more viscous and the resulting shear higher. This could make the destruction of egg yolk proteins more rapid compared to mayonnaise with a lower oil content. The abnormal mayonnaise obtained from the experiments with the coarse emulsion tip speed of 24.9 m/s follow the same abnormal texture-curve. This would suggest that this mayonnaise actually have a higher amount of dispersed phase than the 80.5% oil that was included in the recipe. It is possible that the abnormal mayonnaise is a double emulsion, specifically a w/O/W emulsion. This results in an increase in the effective dispersed phase volume fraction and would explain why it behaves like a mayonnaise with increased oil content.

5.2.2 Coarse emulsion phase inversion

Coarse emulsion phase inversion occurs during the coarse emulsion step, which is the step where the oil is added. This type of inversion is characterized by a very early phase inversion resulting in no mayonnaise being formed. By combing the results from the immersion blender and the high shear mixer it is seen that the position of the rotor and the shape of the vessel is of great importance to avoid coarse emulsion phase inversion. The effect of positioning the rotor in the water phase is illustrated in Figure 5.2 below.

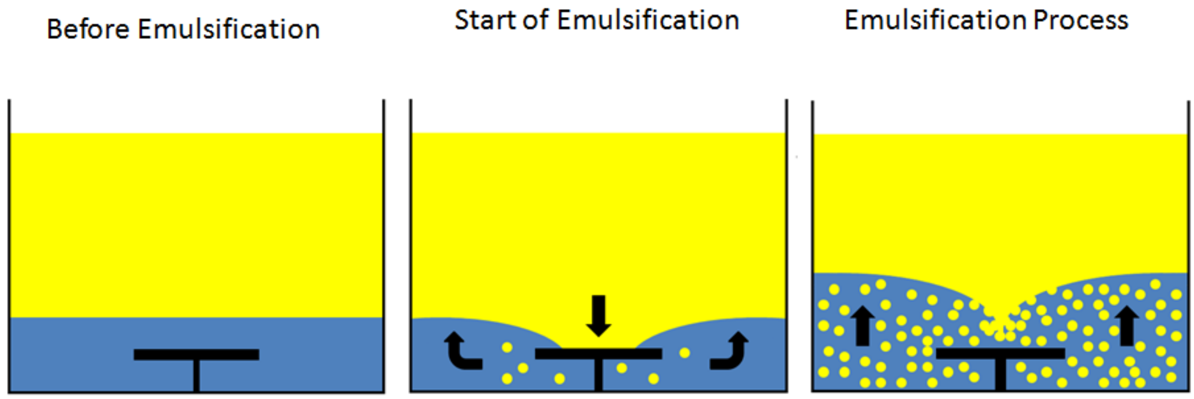

Figure 5.2: Illustration of the effect of the placement of the rotor. The blue on the bottom represents water while the yellow on top represents oil. When the stirring is started the oil will be dragged down to the water phase to form an O/W emulsion (Modified after original by Salager et al., 2001).

That the rotor needs to be positioned in the water phase became clear when the immersion blender was used. When too much oil entered the rotor there was an immediate phase inversion. The same could be seen in several experiments done in the high shear mixer where no mayonnaise could be made using an oil inlet flow rate of 7.4 kg/s. When such a high oil inlet flow rate is used it is probable that at a certain instant in time the amount of oil in the rotor will be too high, resulting in a phase inversion.

The importance of having the rotor in the water phase can also be seen when the highest coarse emulsion tip speed was used. At this speed the flow pattern changed so that the rotor pushed out the liquid faster than it could enter, resulting in a mostly empty rotor. Since the rotor was not placed in the water phase during the addition of oil there was a coarse emulsion phase inversion where no mayonnaise was formed.

Mayonnaise is less sensitive to high oil flow rates when an O/W emulsion has been formed. This can be seen in the experiments 4.3.3 *Ratio of oil added at lower flow rate* where different ratios of oil were added to the mixer at a lower flow rate. This agrees with the cookbooks that state that the first half of the oil must be added very slowly. That mayonnaise is less sensitive when it has been formed can be explained by the hysteresis of the system, which is illustrated in Figure 5.3. The site of droplet formation, which in this case is the rotor/stator combination, is sensitive to a high local oil fraction. Therefore, the local composition in the rotor needs to be in the O/W part in Figure 5.3 during the initial addition of oil. When the initial emulsion has been formed it is possible to move to the left of the phase inversion line where the composition of oil is really high but an O/W emulsion is still continually formed, illustrated by the blue arrow in Figure 5.3. If the local oil composition in the rotor goes left of the hysteresis zone there is a coarse emulsion phase inversion.

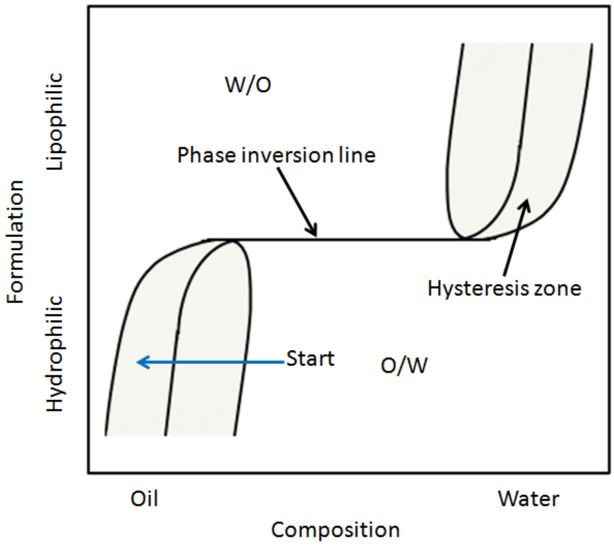

Figure 5.3: A schematic overview of how the hysteresis zone makes it possible pass the phase inversion line while still maintaining an O/W emulsion. The horizontal arrow indicates how the system is changed when oil is added during mayonnaise formation (Modified after original by Salager et al., 2001).

The shape of the vessel can help prevent a coarse emulsion phase inversion by covering the rotor in water phase more efficiently. In order to cover the rotor a narrow vessel is needed but this makes it more difficult to get a mixing pattern that reaches the entire vessel. To circumvent this problem a vessel with a cone shaped bottom can be used. This could be used when the size of the vessel is scaled up.

Scaling up the vessel size and rotor dimensions might allow for a higher oil inlet flow rate to be used. This is, however, assuming that the rotor is always under the water surface during the addition of oil. If the oil/water ratio in the rotor is kept constant and the size of the rotor is increased then the amount of oil in the rotor is increased compared to the smaller scale. This suggests that a higher oil flow might possibly be used.

6 Conclusion

The texture of mayonnaise shows a distinct peak around 0.3 normalized passages, independent of the emulsification tip speed used. The droplets size of the mayonnaise has a minimum that coincide with the texture peak. The higher emulsification speeds gives shorter processing time and slightly higher texture at the peak. Therefore higher emulsification tip speeds can be used to optimize the production time. Over-shear is an irreversible phenomenon that occurs when the emulsification time is too long. Mayonnaise that has been over-sheared is characterized by a lower Stevens value and an increase in droplet size. Mayonnaise made with a higher oil content is more sensitive to over-shear and therefore short emulsification times should be used. Increasing the oil content to 84% increased the maximum Stevens value while 85% and 86% gave a lower Stevens value. All mayonnaise with an oil content of 84% and up broke and underwent emulsification phase inversion due to the over-shear. Higher oil content resulted in a faster phase inversion.

To get a mayonnaise with a high Stevens value cold ingredients and a high egg yolk content should be used. Mayonnaise made with egg yolk powder has a peak in texture around 0.1 normalized passages. The texture is higher than what could be expected from liquid egg yolk but the mayonnaise is very sensitive to over-shear. The recipe containing whole egg does not meet the expected standard of mayonnaise since it had a low texture and large droplet size. Mayonnaise made with egg yolk powder and whole egg were slimy with a long texture.

If a too high oil inlet flow rate is used the mayonnaise will undergo a catastrophic phase inversion during the oil addition step. No apparent correlation between the oil inlet flow rate and the coarse emulsion tip speed was found. Instead the theory that the rotor has to be positioned in the water phase to get an O/W emulsion was supported by the experiments made both in the high shear mixer and with the immersion blender.

The mayonnaise is more sensitive to high oil inlet flow rates at the start of the emulsion formation. To optimize the oil addition time the first part of the oil should be added at a lower rate before a higher oil flow rate can be used.

Cold ingredients are preferred to be able to use a high oil flow rate without causing a coarse emulsion phase inversion.

The recipe with decreased egg yolk content gave mayonnaise that is less sensitive to phase inversion. The cause for this is unknown and more research is recommended. When making mayonnaise with a recipe containing egg yolk powder a very low oil inlet flow rate should be used to avoid coarse emulsion phase inversion. To be able to use a higher oil inlet flow rate it is necessary to optimize the dissolvement of the powder.

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8 Appendix

8.1 Mixer settings

The settings used in the different experiments are presented in Table 8.1

Table 8.1: The setting used for the experiments.

Run nr.	**Date Y/M/D**	**Purpose**	**Coarse emulsion**		**Emulsification**
			Oil inlet flow rate [kg/s]	**Tip speed [m/s]**	**Normalized passages**
R1	17/02/22	Rep. run	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R2	17/02/27	Rep. run	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R3	17/03/01	Rep. run, Center point	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R4	17/02/27	ET and ES	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R5	17/03/01	ET and ES	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R6	17/03/15	1 °C oil, 8 °C ing.	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R7	17/03/13	20 °C oil, 8 °C ing.	3.2	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R8	17/04/25	8 °C oil, 20 °C ing.	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R9	17/04/19	Less egg yolk	4.0	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R10	17/04/19	Egg yolk powder	0.27	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R11	17/05/09	Egg yolk powder, 10 s	0.27	8.3	0.053, 0.11, 0.21, 0.32
R12	17/04/25	Whole egg	1.2	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R13	17/03/15	82% oil	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R14	17/03/15	83% oil	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R15	17/03/06	84% oil	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R16	17/03/08	85% oil	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R17	17/03/08	86% oil	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R18	17/03/08	87% oil	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R19	17/03/06	88% oil	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R20	17/03/06	94% oil	0.61	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R21	17/04/06	CE tip speed phase inv	0	0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80

Run no	Date Y/M/D	Purpose	Coarse emulsion		Emulsification
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Normalized passages
R22	17/04/04	CE tip speed phase inv	0.61	1.4	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R23	17/04/04	CE tip speed phase inv	0.61	2.8	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R24	17/04/06	CE tip speed phase inv	2.5	2.8	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R25	17/03/22	CE tip speed phase inv	2.5	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R26	17/03/22	CE tip speed phase inv	3.6	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R27	17/03/23	CE tip speed phase inv	4.4	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R28	17/03/23	CE tip speed phase inv	4.9	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R29	17/03/13	CE tip speed phase inv	2.5	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R30	17/03/13	CE tip speed phase inv	3.6	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R31	17/03/20	CE tip speed phase inv	4.9	13.0	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R32	17/03/20	CE tip speed phase inv	3.6	19.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R33	17/03/20	CE tip speed phase inv	4.4	19.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R34	17/03/20	CE tip speed phase inv	4.9	19.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R35	17/03/21	CE tip speed phase inv	0.61	24.9	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R36	17/03/21	CE tip speed phase inv	3.6	24.9	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R37	17/03/21	CE tip speed phase inv	4.4	24.9	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R38	17/05/03	New standard	5.1	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R39	17/05/03	New standard	6.7	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R40	17/03/27	Smaller opening	2.5	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R41	17/03/27	Smaller opening	3.5	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R42	17/04/24	Larger opening	4.3	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R43	17/04/24	Larger opening	5.4	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R44	17/05/02	0% oil slow	4.0	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R45	17/05/02	0% oil slow	5.1	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R46	17/04/18	40% oil slow	5.1	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R47	17/05/09	40% oil slow	6.7	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R48	17/04/25	8 °C oil, 20 °C ing. Inv.	5.1	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R49	17/04/25	8 °C oil, 20 °C ing. Inv.	6.7	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R50	17/05/05	20 °C oil, 20 °C ing. Inv	4.0	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80

Run no	Date Y/M/D	Purpose	Coarse emulsion		Emulsification
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Normalized passages
R51	17/05/05	20 °C oil, 20 °C ing. Inv	5.1	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R52	17/05/08	Less egg yolk Inv.	6.7	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R53	17/05/08	Less egg yolk Inv.	7.4	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80
R54	17/05/09	Egg yolk powder Inv.	0.73	8.3	0.11, 0.21, 0.32, 0.43, 0.53, 0.80

8.2 Analysis results

The analysis results obtained are shown in Table 8.2.

Table 8.2: The texture and droplet size results obtained from the analysis.

Run nr.	Date Y/M/D	Purpose	Coarse emulsion		Emulsification	Analysis			
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Norm. passages	Stevens value	Mean Stevens value	Droplet size [µm]	Mean Droplet size [µm]
R1	17/02/22	Rep. run	0.61	13.0	0.11	191	191	2.98	3.00
						-		3.00	
						-		3.03	
					0.21	213	213	2.36	2.44
						-		2.48	
						-		2.51	
					0.32	208	208	2.21	2.24
						-		2.24	
						-		2.27	
					0.43	204	204	2.30	2.33
						-		2.33	
						-		2.36	
					0.53	179	179	2.18	2.22
						-		2.22	
						-		2.26	
					0.80	140	140	2.36	2.40
						-		2.40	
						-		2.44	
R2	17/02/27	Rep. run	0.61	13.0	0.11	158	170	2.79	2.80
						178		2.80	
						172		2.80	
					0.21	206	202	2.27	2.34
						195		2.37	
						206		2.39	
					0.32	192	198	1.95	1.97

Run nr.	Date Y/M/D	Purpose	Coarse emulsion		Emulsification	Analysis			
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Norm. passages	Stevens value	Mean Stevens value	Droplet size [µm]	Mean Droplet size [µm]
R2	17/02/27	Rep. run	0.61	13.0	0.32	191	198	1.97	1.97
						212		1.99	
					0.43	188	192	1.92	1.91
						187		1.91	
						200		1.91	
					0.53	177	175	1.99	2.00
						166		2.01	
						183		2.02	
					0.80	147	145	1.98	1.99
						140		2.00	
						148		2.01	
R3	17/03/01	Rep. run, Center point	0.61	13.0	0.11	181	181	2.89	2.90
						171		2.90	
						190		2.91	
					0.21	215	217	2.17	2.20
						204		2.20	
						231		2.22	
					0.32	217	212	2.02	2.01
						200		2.01	
						219		2.01	
					0.43	194	199	2.03	2.04
						206		2.04	
						196		2.04	
					0.53	169	171	1.97	1.98
						165		1.98	
						180		1.99	
					0.80	142	151	2.07	2.09
						152		2.10	

Run nr.	Date Y/M/D	Purpose	Coarse emulsion		Emulsification	Analysis			
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Norm. passages	Stevens value	Mean Stevens value	Droplet size [µm]	Mean Droplet size [µm]
R3	17/03/01	R.r, C.p.	0.61	13.0	0.80	157	151	2.12	2.09
R4	17/02/27	ET and ES	0.61	13.0	0.094	147	157	2.92	2.93
						166		2.93	
						159		2.94	
					0.19	190	187	2.45	2.46
						179		2.47	
						192		2.48	
					0.28	197	194	2.01	2.03
						188		2.03	
						197		2.04	
					0.37	181	185	1.93	1.95
						178		1.95	
						195		1.97	
					0.47	181	182	1.96	1.99
						171		1.99	
						192		2.01	
					0.70	152	156	1.94	1.94
						151		1.94	
						164		1.94	
R5	17/03/01	ET and ES	0.61	13.0	0.12	193	196	2.68	2.69
						195		2.70	
						200		2.71	
					0.23	216	220	2.07	2.09
						211		2.09	
						233		2.11	
					0.36	205	215	2.05	2.07
						230		2.07	
						210		2.09	
					0.48	169	175	1.92	1.95

Run nr.	Date Y/M/D	Purpose	Coarse emulsion		Emulsification	Analysis			
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Norm. passages	Stevens value	Mean Stevens value	Droplet size [µm]	Mean Droplet size [µm]
R5	17/03/01	ET and ES	0.61	13.0	0.48	178	175	1.95	1.95
						177		1.98	
					0.60	150	152	2.01	20.4
						150		2.04	
						158		2.07	
					0.91	111	115	2.26	2.31
						108		2.31	
						125		2.36	
R6	17/03/15	1 °C oil, 8 °C ing.	0.61	13.0	0.11	190	186	2.79	2.79
						177		2.79	
						190		2.79	
					0.21	217	222	2.13	2.14
						216		2.15	
						234		2.16	
					0.32	216	216	1.97	1.99
						209		1.99	
						222		2.01	
					0.43	197	209	2.01	2.03
						225		2.03	
						203		2.05	
					0.53	179	183	2.02	2.06
						175		2.06	
						195		2.09	
					0.80	140	144	2.04	2.12
						136		2.15	
						157		2.18	
R7	17/03/13	20 °C oil, 8 °C ing.	0.61	13.0	0.11	166	177	2.96	2.97
						188		2.97	
						178		2.98	

Run nr.	Date Y/M/D	Purpose	Coarse emulsion		Emulsification	Analysis			
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Norm. passages	Stevens value	Mean Stevens value	Droplet size [µm]	Mean Droplet size [µm]
R7	17/03/13	20 °C oil, 8 °C ing.	0.61	13.0	0.21	196	191	2.33	2.35
						190		2.36	
						188		2.37	
					0.32	181	186	2.05	2.07
						180		2.07	
						198		2.09	
					0.43	167	164	1.95	2.01
						161		2.04	
						163		2.06	
					0.53	146	147	2.07	2.09
						152		2.09	
						142		2.11	
					0.80	111	122	2.15	2.24
						126		2.28	
						129		2.32	
R8	17/04/25	8 °C oil, 20 °C ing.	3.2	13.0	0.11	150	150	3.16	3.11
						144		3.08	
						155		3.09	
					0.21	188	189	2.27	2.28
						183		2.28	
						195		2.29	
					0.32	198	195	2.04	2.06
						194		2.06	
						192		2.07	
					0.43	171	175	1.95	2.00
						182		1.97	
						170		2.06	
					0.53	159	159	1.90	1.93
						160		1.93	

Run nr.	Date Y/M/D	Purpose	Coarse emulsion		Emulsification	Analysis			
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Norm. passages	Stevens value	Mean Stevens value	Droplet size [µm]	Mean Droplet size [µm]
R8	17/04/25	8 °C oil, 20 °C ing.	3.2	13.0	0.53	159	159	2.06	1.93
					0.80	131	127	1.99	2.03
						126		2.03	
						125		2.06	
R9	17/04/19	Less egg yolk	2.3	8.3	0.11	109	111	3.65	3.65
						109		3.65	
						116		3.65	
					0.21	153	155	3.01	3.03
						163		3.03	
						149		3.04	
					0.32	143	139	2.84	2.85
						134		2.85	
						139		2.87	
					0.43	119	119	2.78	2.79
						115		2.79	
						124		2.81	
					0.53	104	105	2.78	2.81
						102		2.81	
						109		2.84	
					0.80	79.3	79.9	3.02	3.06
						75.9		3.07	
						84.4		3.11	
R10	17/04/19	Egg yolk powder	0.27	8.3	0.11	173	168	2.70	2.73
						168		2.73	
						162		2.76	
					0.21	136	139	2.81	2.84
						144		2.84	
						136		2.87	
					0.32	114	117	2.89	2.94

Run nr.	Date Y/M/D	Purpose	Coarse emulsion		Emulsification	Analysis			
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Norm. passages	Stevens value	Mean Stevens value	Droplet size [µm]	Mean Droplet size [µm]
R10	17/04/19	Egg yolk powder	0.27	8.3	0.32	116	117	2.94	2.94
						120		2.98	
					0.43	95.1	93.9	3.04	3.10
						90.0		3.10	
						96.7		3.16	
					0.53	75.2	75.3	3.08	3.20
						76.2		3.24	
						74.4		3.29	
					0.80	58.8	61.6	3.39	3.54
						63.9		3.53	
						62.0		3.77	
R11	17/05/09	Egg yolk powder, 10s	0.27	8.3	0.053	135	146	3.10	3.13
						145		3.13	
						160		3.16	
					0.11	142	142	2.87	2.90
						-		2.91	
						-		2.94	
					0.21	127	127	2.90	2.95
						-		2.95	
						-		3.01	
					0.32	104	104	3.16	3.20
						-		3.21	
						-		3.25	
R12	17/04/25	Whole egg	1.2	8.3	0.11	81.3	80.5	4.63	4.66
						78.6		4.67	
						81.5		4.69	
					0.21	75.7	77.1	4.27	4.30
						80.0		4.31	
						75.7		4.33	

Run nr.	Date Y/M/D	Purpose	Coarse emulsion		Emulsification	Analysis			
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Norm. passages	Stevens value	Mean Stevens value	Droplet size [µm]	Mean Droplet size [µm]
R12	17/04/25	Whole egg	1.2	8.3	0.32	54.8	54.8	4.71	4.77
						52.6		4.78	
						57.0		4.82	
					0.43	43.8	42.0	5.23	5.32
						40.6		5.34	
						41.6		5.44	
					0.53	34.4	33.9	6.11	6.10
						33.2		6.10	
						34.0		6.10	
					0.80	30.0	29.4	7.33	7.30
						29.0		7.24	
						29.2		7.32	
R13	17/03/15	82% oil	0.61	13.0	0.11	209	200	2.78	2.79
						191		2.80	
						200		2.81	
					0.21	195	199	2.57	2.59
						205		2.59	
						197		2.60	
					0.32	190	198	2.26	2.29
						195		2.29	
						209		2.31	
					0.43	185	189	2.35	2.37
						178		2.37	
						202		2.39	
					0.53	163	161	2.18	2.27
						159		2.30	
						160		2.34	
					0.80	120	123	2.40	2.49
						117		2.53	

Run nr.	Date Y/M/D	Purpose	Coarse emulsion		Emulsification	Analysis			
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Norm. passages	Stevens value	Mean Stevens value	Droplet size [µm]	Mean Droplet size [µm]
R13	17/03/15	82% oil	0.61	13.0	0.80	134	123	2.57	2.49
R14	17/03/15	83% oil	0.61	13.0	0.11	218	218	2.70	2.71
						218		2.71	
						218		2.73	
					0.21	218	219	2.46	2.46
						214		2.45	
						227		2.45	
					0.32	197	199	2.35	2.35
						189		2.35	
						211		2.36	
					0.43	163	166	2.41	2.43
						172		2.44	
						163		2.45	
					0.53	135	137	2.66	2.68
						142		2.69	
						135		2.71	
					0.80	96	96	3.05	3.18
						94		3.22	
						100		3.29	
R15	17/03/06	84% oil	0.61	13.0	0.11	236	240	2.58	2.59
						234		2.59	
						250		2.61	
					0.21	199	205	2.45	2.47
						201		2.47	
						215		2.49	
					0.32	172	174	2.54	2.57
						167		2.57	
						184		2.60	
					0.43	134	139	2.75	2.84

Run nr.	Date Y/M/D	Purpose	Coarse emulsion		Emulsification	Analysis			
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Norm. passages	Stevens value	Mean Stevens value	Droplet size [µm]	Mean Droplet size [µm]
R15	17/03/06	84% oil	0.61	13.0	0.43	135	139	2.91	2.84
						148		2.85	
					0.53	110	108	3.16	3.25
						108		3.26	
						107		3.36	
					0.80	-	Broken	-	Broken
						-		-	
						-		-	
R16	17/03/08	85% oil	0.61	13.0	0.11	218	213	2.62	2.64
						210		2.64	
						211		2.65	
					0.21	167	167	2.62	2.64
						167		2.64	
						168		2.66	
					0.32	112	115	3.15	3.22
						112		3.23	
						120		3.30	
					0.43	-	Broken	-	Broken
						-		-	
						-		-	
R17	17/03/08	86% oil	0.61	13.0	0.11	173	170	2.72	2.73
						168		2.73	
						170		2.75	
					0.21	-	Broken	-	Broken
						-		-	
						-		-	
R18	17/03/08	87% oil	0.61	13.0	0.11	-	Broken	-	Broken
						-		-	
						-		-	

Run nr.	Date Y/M/D	Purpose	Coarse emulsion		Emulsification	Analysis			
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	Norm. passages	Stevens value	Mean Stevens value	Droplet size [µm]	Mean Droplet size [µm]
R19	17/03/06	88% oil	0.61	13.0	0.11	-	Broken	-	Broken
						-		-	
						-		-	
R20	17/03/06	94% oil	0.61	13.0	0.11	-	Broken	-	Broken
						-		-	
						-		-	

8.3 Phase inversion results

The experiments investigating phase inversion can be seen in Table 8.3. Runs from 55 to 67 are not presented in the report.

Table 8.3: The experiments about phase inversion and what kind of mayonnaise they resulted in.

Run nr.	Date Y/M/D	Purpose	Coarse emulsion		Type of Mayonnaise
			Oil inlet flow rate [kg/s]	Tip speed [m/s]	
R21	17/04/06	CE tip speed phase inv.	0	0	Broken
R22	17/04/04		0.61	1.4	Proper
R23	17/04/04		0.61	2.8	Proper
R24	17/04/06		2.5	2.8	Proper
R25	17/03/22		2.5	8.3	Proper
R26	17/03/22		3.6	8.3	Proper
R27	17/03/23		4.4	8.3	Broken
R28	17/03/23		4.9	8.3	Broken
R29	17/03/13		2.5	13.0	Proper
R30	17/03/13		3.6	13.0	Broken
R31	17/03/20		4.9	13.0	Broken
R32	17/03/20		3.6	19.3	Proper
R33	17/03/20		4.4	19.3	Broken
R34	17/03/20		4.9	19.3	Broken
R35	17/03/21		0.61	24.9	Abnormal
R36	17/03/21		3.6	24.9	Broken
R37	17/03/21		4.4	24.9	Broken
R38	17/05/03	New standard	5.1	8.3	Proper
R39	17/05/03		6.7	8.3	Broken
R40	17/03/27	Smaller opening	1.8	8.3	Proper
R56	17/03/27		2.5	8.3	Proper
R41	17/03/27		3.5	8.3	Broken
R42	17/03/27		4.5	8.3	Broken
R57	17/04/24	Larger opening	4.3	8.3	Proper
R43	17/04/24		5.4	8.3	Broken
R44	17/04/24		6.8	8.3	Broken
R58	17/05/02	0% oil slow	3.2	8.3	Proper
R45	17/05/02		4.0	8.3	Proper
R46	17/05/02		5.1	8.3	Broken
R59	17/04/18	40% oil slow	3.2	8.3	Proper
R47	17/04/18		5.1	8.3	Proper
R48	17/05/09		6.7	8.3	Broken
R60	17/04/18		7.4	8.3	Broken
R49	17/04/25	8 °C oil, 20 °C ing. Inv.	4.0	8.3	Proper
R50	17/04/25		5.1	8.3	Broken
R61	17/04/25		6.7	8.3	Broken
R62	17/05/05	20 °C oil, 20 °C ing. Inv.	3.2	8.3	Proper
R51	17/05/05		4.0	8.3	Proper
R52	17/05/05		5.1	8.3	Broken

Run nr.	**Date Y/M/D**	**Purpose**	**Coarse emulsion**		**Type of Mayonnaise**
			Oil inlet flow rate [kg/s]	**Tip speed [m/s]**	
R63	17/04/19	Less egg yolk Inv.	4.0	8.3	Proper
R64	17/04/19		5.1	8.3	Proper
R53	17/05/08	Less egg yolk Inv.	6.7	8.3	Proper
R54	17/05/08		7.4	8.3	Broken
R55	17/05/09	Egg yolk powder Inv.	0.73	8.3	Broken
R65	17/04/27		1.2	8.3	Broken
R66	17/04/27		2.3	8.3	Broken
R67	17/04/27		4.4	8.3	Broken

8.4 Recipes for increased oil content

Recipes for experiments with 82-94% oil content can be seen in Table 8.4-8.11.

Table 8.4: Fractions and amount of ingredients for a batch with 82% oil.

Ingredient	**Fraction (w/w%)**	**Amount (kg)**
Water	5.30	1.75
Salt	0.275	0.0908
Sugar	0.275	0.0908
Mustard	2.29	0.756
Vinegar	1.92	0.635
Egg yolk, liquid (8% salt)	7.79	2.57
Rapeseed oil	82.0	27.1
Total	**100**	**33**

Table 8.5: Fractions and amount of ingredients for a batch with 83% oil.

Ingredient	**Fraction (w/w%)**	**Amount (kg)**
Water	5.06	1.67
Salt	0.261	0.0862
Sugar	0.261	0.0862
Mustard	2.18	0.718
Vinegar	1.83	0.603
Egg yolk, liquid (8% salt)	7.39	2.44
Rapeseed oil	83.0	27.4
Total	**100**	**33**

Table 8.6: Fractions and amount of ingredients for a batch with 84% oil.

Ingredient	**Fraction (w/w%)**	**Amount (kg)**
Water	4.79	1.58
Salt	0.247	0.0815
Sugar	0.247	0.0815
Mustard	2.06	0.679
Vinegar	1.73	0.571
Egg yolk, liquid (8% salt)	7.00	2.31
Rapeseed oil	84.0	27.7
Total	**100**	**33**

Table 8.7: Fractions and amount of ingredients for a batch with 85% oil.

Ingredient	**Fraction (w/w%)**	**Amount (kg)**
Water	4.42	1.46
Salt	0.228	0.0754
Sugar	0.228	0.0754
Mustard	1.90	0.628
Vinegar	1.60	0.528
Egg yolk, liquid (8% salt)	6.48	2.14
Rapeseed oil	85.0	28.1
Total	**100**	**33**

Table 8.8: Fractions and amount of ingredients for a batch with 86% oil.

Ingredient	**Fraction (w/w%)**	**Amount (kg)**
Water	4.15	1.37
Salt	0.215	0.0708
Sugar	0.215	0.0708
Mustard	1.79	0.590
Vinegar	1.50	0.495
Egg yolk, liquid (8% salt)	6.06	2.00
Rapeseed oil	86.0	28.4
Total	**100**	**33**

Table 8.9: Fractions and amount of ingredients for a batch with 87% oil.

Ingredient	**Fraction (w/w%)**	**Amount (kg)**
Water	3.88	1.28
Salt	0.200	0.0662
Sugar	0.200	0.0662
Mustard	1.67	0.551
Vinegar	1.40	0.463
Egg yolk, liquid (8% salt)	5.67	1.87
Rapeseed oil	87.0	28.7
Total	**100**	**33**

Table 8.10: Fractions and amount of ingredients for a batch with 88% oil.

Ingredient	**Fraction (w/w%)**	**Amount (kg)**
Water	3.61	1.19
Salt	0.186	0.0615
Sugar	0.186	0.0615
Mustard	1.55	0.513
Vinegar	1.31	0.431
Egg yolk, liquid (8% salt)	5.27	1.74
Rapeseed oil	88.0	29.0
Total	**100**	**33**

Table 8.11: Fractions and amount of ingredients for a batch with 94% oil.

Ingredient	**Fraction (w/w%)**	**Amount (kg)**
Water	1.80	0.595
Salt	0.0933	0.0308
Sugar	0.0933	0.0308
Mustard	0.776	0.256
Vinegar	0.652	0.215
Egg yolk, liquid (8% salt)	2.64	0.872
Rapeseed oil	94.0	31.0
Total	**100**	**33**

8.5 Pipes used for oil addition

In the beginning of the thesis a standard pipe was used to transport the oil to the mixer. This pipe did not have a connection on it and looked exactly like the pipe in Figure 8.1 but without the connection. This pipe is referred to as the *standard pipe* in all experiments.

The connection was added in the middle of the practical work to be able to connect the pipe to a larger opening in the mixer. By adding the connection to the pipe the oil inlet flow rate was affected and mayonnaise could somehow be made with a higher oil inlet flow rate than before. This pipe is referred to as the *standard pipe after flow changed* in all experiments. The *standard pipe after flow changed* mounted on the mixer and the dimensions of the pipe can be seen in Figure 8.1 and 8.2 respectively. After the pipe there is an additional 4 cm of pipe to the butterfly valve into the mixer.

In section 4.3.4 *Design of oil opening* the valve marked as *valve into mixer* in Figure 8.1 was used to control the oil inlet flow rate when a smaller opening was used. This is referred to as the *smaller opening* in the experiments. In all other experiments the valve marked as *valve* in Figure 8.1 and 8.3 was used to control the oil inlet flow rate.

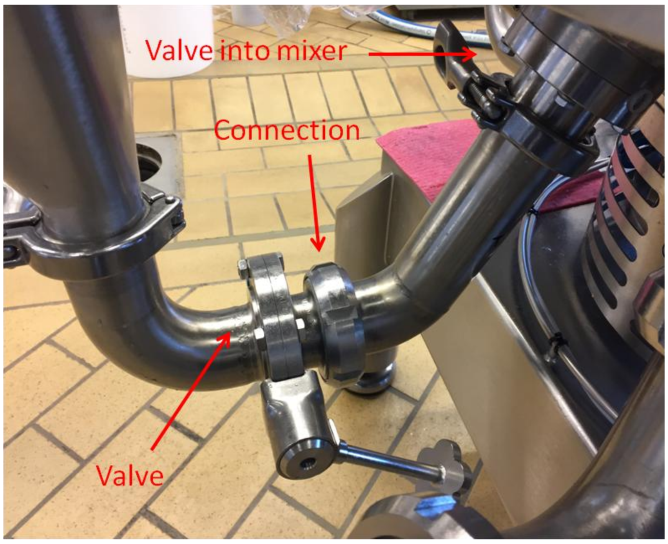

Figure 8.1: The standard pipe after flow changed *attached to the mixer. The* standard pipe *is exactly the same but without the connection.*

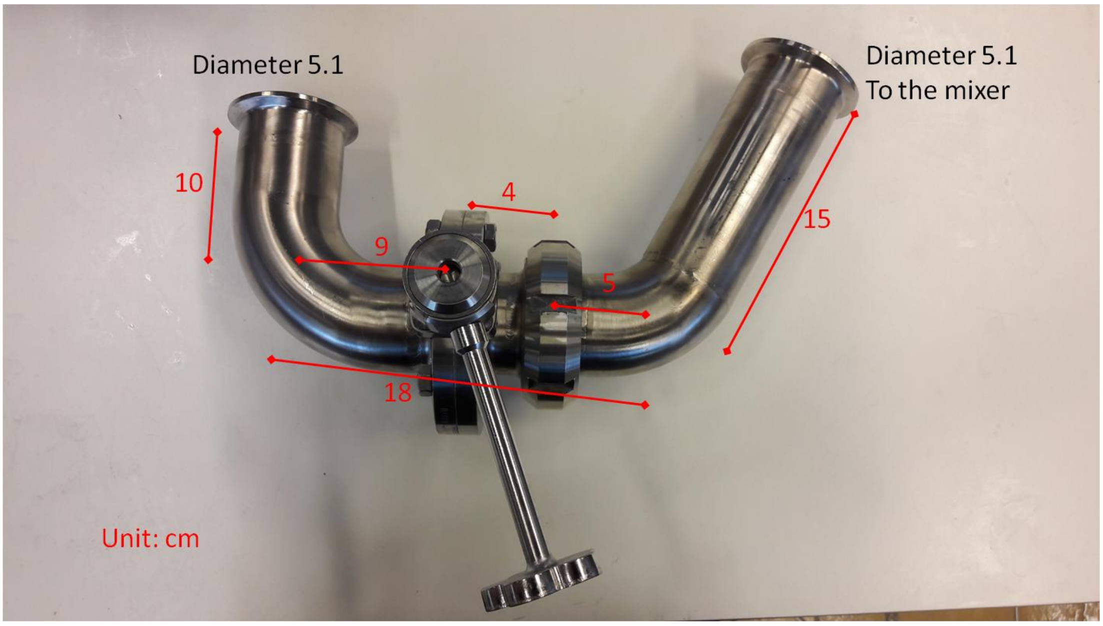

Figure 8.2: The standard pipe after flow changed *used in the experiments. A butterfly valve and a connection are positioned in the middle. The* standard pipe *is exactly the same but without the connection*

To be able to add the oil through a valve with a larger diameter the oil pipe was connected to a larger pipe. This is referred to as the *larger pipe* in all experiments. The *larger pipe* mounted on the mixer can be seen Figure 8.3 and the dimensions of the *larger pipe* can be seen in Figure 8.4. After the *larger pipe* there is an additional 5 cm of pipe to the butterfly valve into the mixer.

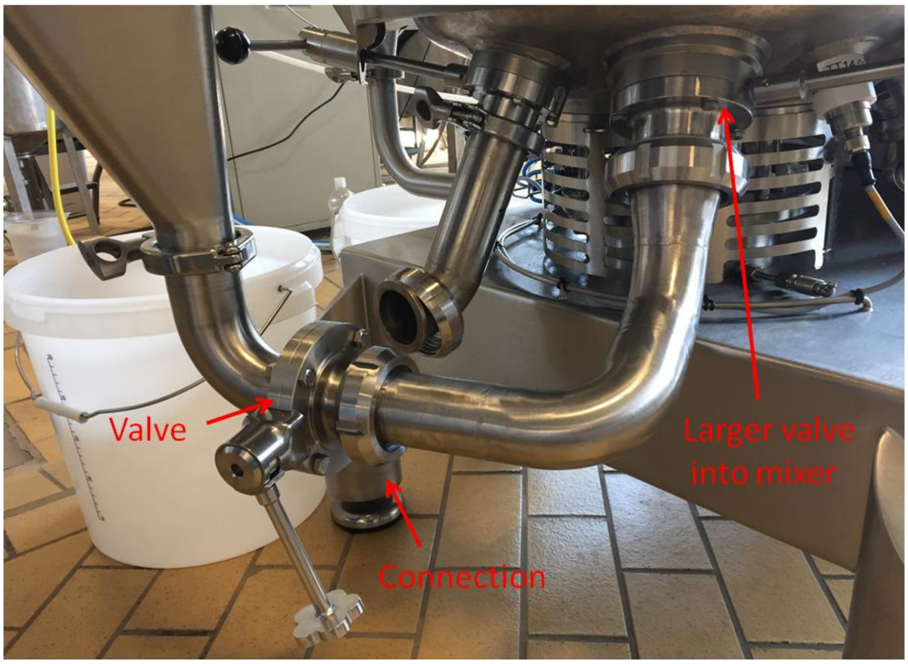

Figure 8.3: The larger pipe *connected to a larger valve.*

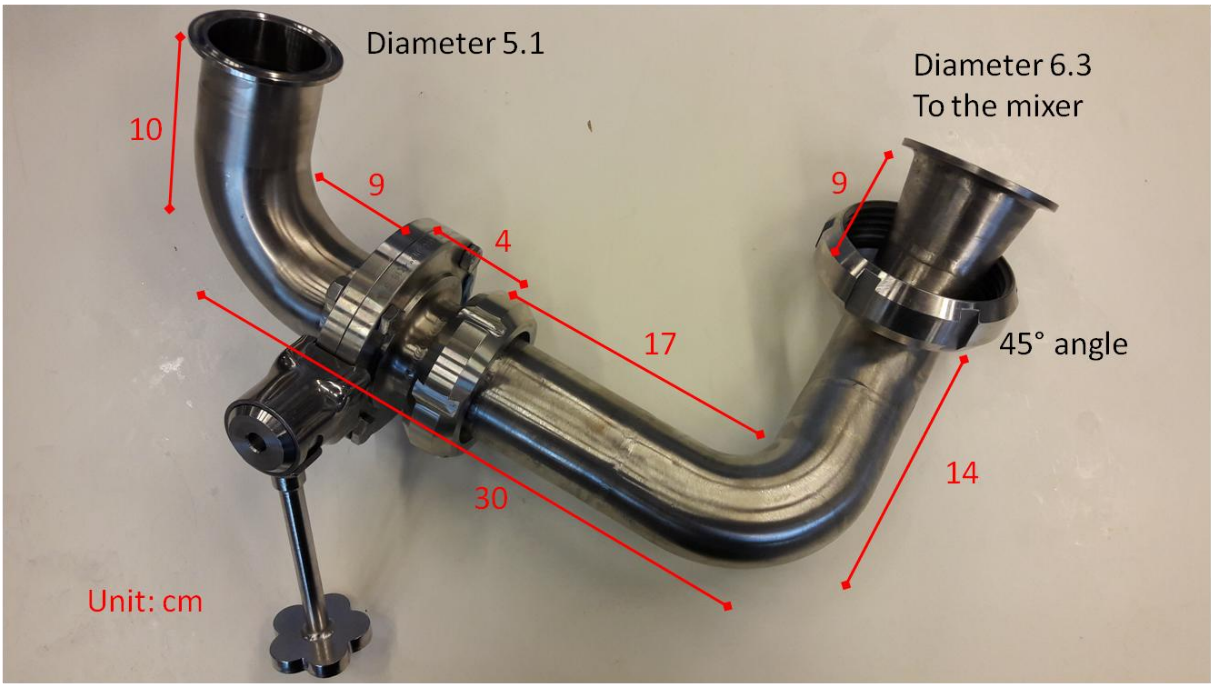

Figure 8.4: The larger pipe used in the experiments. A butterfly valve and a connection are positioned in the middle.