# OPTIMIZING EMULSION STABILITY OF HIGH-OIL POURABLE DRESSINGS USING DIFFERENT STABILIZERS

by

#### MIRANDA GRIZIO

B.S., Charter Oak State College, 2003

#### A REPORT

submitted in partial fulfillment of the requirements for the degree

#### MASTER OF SCIENCE

Food Science

KANSAS STATE UNIVERSITY Manhattan, Kansas

2011

Approved by:

Major Professor Dr. Fadi Aramouni

## Abstract

To find an optimal formulation of oil-in-water (O/W) emulsion pourable dressings containing 60% and 70% soybean oil, nine stabilizers common to the dressing industry were tested, each at three concentrations. The stabilizers tested were xanthan gum, propylene glycol alginate (PGA), a xanthan gum/guar gum/sodium alginate blend, a viscosifying modified corn starch, an emulsifying modified corn starch, microcrystalline cellulose (MCC), liquid salted egg yolks, liquid salted whole eggs, and dried egg whites (DEW). The emulsions were made with a laboratory high shear disperser and evaluated over 8 weeks by measurement of creaming in 100-mL graduated cylinders and by change in viscosity as measured by a Brookfield viscometer. The stabilizers and concentrations most effective at preventing separation and maintaining viscosity were MCC at 1%, 2%, and 3% in the 60% oil emulsion and 0.25% xanthan gum in the 70% oil emulsion. Average viscosities ranged from 3300 - 23,400 centipoise (cP). The emulsifying starch and dried egg whites were also effective at preventing creaming, but failed to maintain viscosity. The other ingredients showed some initial emulsion stability followed by either a gradual or sudden decrease in stabilization, as seen by decreasing viscosity and eventual separation.

## **Table of Contents**

List of Figures iv
List of Tables
Acknowledgementsvii
Chapter 1 - Introduction and Literature Review
Chapter 2 - Materials and Methods
Materials6
Ingredients6
Equipment7
Methods7
Sample preparation7
Sample storage
Sample measurement
Chapter 3 - Results and Discussion
Gums9
Starches and Cellulose
Egg Products
Chapter 4 - Conclusions
References

## List of Figures

Figure 1: Effect of % xanthan gum on creaming and viscosity of 60% oil emulsion 12
Figure 2: Effect of % xanthan gum on creaming and viscosity of 70% oil emulsion 13
Figure 3: Effect of % propylene glycol alginate on creaming and viscosity of 60% oil emulsion
Figure 4: Effect of % propylene glycol alginate on creaming and viscosity of 70% oil emulsion
Figure 5: Effect of % gum blend on creaming and viscosity of 60% oil emulsion
Figure 6: Effect of % gum blend on creaming and viscosity of 70% oil emulsion 17
Figure 7: Effect of % emulsifying modified corn starch on creaming and viscosity of 60% oil
emulsion
Figure 8: Effect of % emulsifying modified corn starch on creaming and viscosity of 70% oil
emulsion
Figure 9: Effect of % viscosifying modified corn starch on creaming and viscosity of 60% oil
emulsion
Figure 10: Effect of % viscosifying modified corn starch on creaming and viscosity of 70% oil
emulsion
Figure 11: Effect of % microcrystalline cellulose on creaming and viscosity of 60% oil emulsion
Figure 12: Effect of % microcrystalline cellulose on creaming and viscosity of 70% oil emulsion
Figure 13: Effect of % dried egg whites on creaming and viscosity of 60% emulsion
Figure 14: Effect of % dried egg whites on creaming and viscosity of 70% emulsion
Figure 15: Effect of % liquid salted egg yolks on creaming and viscosity of 60% oil emulsion. 29
Figure 16: Effect of % liquid salted egg yolks on creaming and viscosity of 70% oil emulsion. 30
Figure 17: Effect of % liquid salted whole eggs on creaming and viscosity of 60% oil emulsion
31
Figure 18: Effect of % liquid salted whole eggs on creaming and viscosity of 70% oil emulsion

Figure 19: Effect of stabilizer type and concentration on viscosity of 60% oil emulsi	on including
only stabilizers that prevented creaming	
Figure 20: Effect of stabilizer type and concentration on viscosity of 70% oil emulsi	on including
only stabilizers that prevented creaming	

## List of Tables

Table 1: Stabilizer categories of ingredients used in high-oil emulsion stability test	6
Table 2: Formulations used in high-oil emulsion stability test	7
Table 3: Average viscosity and change in viscosity of 60% oil emulsion by stabilizer type	
including only stabilizers that prevented creaming	33
Table 4: Average viscosity and change in viscosity of 70% oil emulsion by stabilizer type	
including only stabilizers that prevented creaming	34

## Acknowledgements

I would like to thank Dr. J. Scott Smith, Chair of the Food Science Graduate Program at Kansas State University, for the invaluable instruction provided in his Food Science Graduate Seminar course on effectively organizing and presenting scientific research. I also wish to thank Dr. Fadi Aramouni, professor and my advisor at Kansas State University, for showing me how to be steadfast in applying scientific principles to product development through his course on Research and Development of Food Products which provided the initial framework for this project. His guidance and patience have been instrumental in my progress. In addition, I would like to acknowledge Dr. Elizabeth Boyle, professor at Kansas State University and member of my graduate committee, for her thorough and thoughtful evaluation of this report, for encouraging rigorous inquiry into the significance of experimental results, and for reminding me that a scientist never stops asking why.

### **Chapter 1 - Introduction and Literature Review**

Pourable dressings can be described as oil-in-water (O/W) emulsions with fat contents of 30 – 45% (McClements, 2005). This study provides an initial screening of stabilizers to find an optimal formulation of pourable dressings containing 60% - 70% oil. High-oil dressings are a unique concept with wide application. Not only can they answer the desire for rich, decadent dressings, but there is also considerable cross-over application, such as garlic- or butter-flavored sauces for vegetables and pasta or as a lower cost alternative to oil in baking applications that require some surface browning, such as garlic bread or pie crusts. This level of oil is similar to that of mayonnaise, which has a standard of identity of not less than 65% oil (FDA, 2010). While formulas for mayonnaise and pourable dressings abound, as evidenced by their presence in the market, an emulsion product with the oil level of mayonnaise but the flow properties of a pourable dressing is less understood. This study sought to answer the following questions: Can any of the stabilizer types and concentrations chosen effectively stabilize a pourable O/W emulsion of 60% or 70% oil, and if so, what viscosities do they produce?

A wide variety of stabilizers have been investigated for use in stabilizing O/W food emulsions, including some common to the dressing industry, such as xanthan gum (Hemar et al., 2001; Parker et al., 1995; Krstonisic et al., 2009; Paraskevopoulou et al., 2005; Pettitt et al., 1995; Yilmazer et al., 1991; Fonseca et al., 2009), propylene glycol alginate (Parker, 1995; Paraskevopoulou, 2005; Pettitt, 1995; Yilmazer, 1991; Fonseca, 2009), modified celluloses (Fonseca, 2009; Sun et al., 2007); egg whites (Ercelebi and Ibanoglu, 2009), and egg yolks (Riscardo et al., 2003). While many showed promising results in these studies, none were tested at oil levels as high as 60 - 70%. The highest oil level tested was 50% by Krstonisic (2009), Paraskevopoulou (2005), and Sun (2007). Emulsions of 40% oil were tested by Pettitt (1995) and Yilmazer (1991), and several had oil levels that were much lower.

To screen for suitable stabilizer types and concentrations, nine stabilizers common to the dressing industry were tested, each at three concentrations in a 60% oil emulsion and in a 70% oil emulsion. Stabilizers can be categorized as either emulsifiers or texture modifiers and work to

stabilize emulsions through different mechanisms (McClements, 2005). Emulsifiers contain nonpolar regions that bind to the surface of an oil droplet, and polar regions that bind to the water, effectively forming a protective film over the droplet that prevents droplets from aggregating. Texture modifiers prevent droplet aggregation by thickening the aqueous phase which slows the movement of the droplets. Both categories are represented in this study (Table 1).

The stabilizers chosen include two gums, xanthan gum and propylene glycol alginate (PGA), as well as a proprietary blend of xanthan gum, guar gum, and sodium alginate formulated for use in the dressing industry. These gums are all texture modifiers, or more specifically, thickening agents (McClements, 2005). In addition, PGA has been modified to have emulsifying properties. Two starches were chosen, a viscosifying starch which acts as a thickening agent and an emulsifying starch which has been chemically modified to have emulsifying properties and so can be classified as both a thickening agent and an emulsifier. Both are cold swelling starches and do not require heat for functionality. Microcrystalline cellulose (MCC) was also used, and similarly can be classified as both a thickening agent and an emulsifier based on its chemical modifications. Three egg ingredients were also selected, liquid salted egg yolks, liquid salted whole eggs, and dried egg whites (DEW). The liquid salted egg yolks and whole eggs contain 10% salt, as is common in the dressing industry, to help preserve them during shipping and storage. Both the liquid eggs work primarily as emulsifiers due to their content of lipoprotein and phospholipid (McClements, 2005). Liquid whole eggs also contain egg white proteins, which are not very good emulsifiers but can provide some thickening (Gaonkar, 2000). Dried egg whites contain egg white proteins and so work primarily as a texture modifier. These ingredients were chosen to represent the main classes of stabilizers used in the dressing industry (i.e., gums, starch, cellulose and eggs), with the expectation that manufacturers may already have many of these ingredients available in-house, be familiar with their properties and handling, and consider them to be acceptable on a finished product ingredient statement.

Gums, starch, and cellulose are all polysaccharides, which can be defined as large molecular weight carbohydrates that interact with water (Daniel and Weaver, 2000). Both starch and cellulose are composed of repeating D-glucose units, but differ in their glycosidic bonds, with starch possessing primarily  $\alpha$ -1-4 linkages and cellulose possessing  $\beta$ -1-4 linkages (Jane,

2000). Starch acts as energy storage in plants, whereas cellulose imparts structure. Both can be chemically and physically modified for improved functionalities in different applications. Gums are typically derived from plant seeds and exudates, seaweed, and microorganisms (Daniel and Weaver, 2000). Specifically, alginates, are derived from brown algae and consist of repeating D-mannuronic acid and L-guluronic acid units, guar gum contains D-mannose and D-galactose units and is derived from guar seeds, and xanthan gum is synthesized by the bacterium *Xanthomonas* and is composed of D-glucose, D-mannose, and D-glucuronic acid units, closely resembling a cellulose chain (Daniel and Weaver, 2000). Like cellulose, these gums possess  $\beta$ -1-4 linkages. Natural gums can be modified to impart different functionalities as with the conversion of alginate to propylene glycol alginate. Eggs, unlike the polysaccharide stabilizers, are complex systems consisting of many different proteins in both the egg yolk and egg white, as well as lipids, carbohydrates, vitamins, and minerals (Breeding and Beyer, 2000). Lipoproteins and phospholipids in the egg yolk are natural emulsifiers. Eggs can be used whole or separated into various components depending on the desired functionality.

The formula models were made with soybean oil, stabilizer, and water with the expectation that the most promising models, based on their ability to resist creaming and change in viscosity, would be remade with the additional ingredients typical to dressings such as vinegar, salt, sugar, and spices.

Emulsion formation typically requires mechanical agitation, most commonly provided by either a high speed mixer, homogenizer, colloid mill, or high shear disperser (Weiss, 2008). A high speed mixer blends the product with a rotating high speed mixing head, and offers several mixing head styles such as blade, propeller, or turbine (McClements, 2005). Homogenizers and colloid mills provide more shear and smaller droplet size than a high speed mixer. A homogenizer forces product through a narrow valve and reduces droplet size by a combination of turbulence and shear (APV, 2011). A colloid mill has a rotor and stator conformation, with the product entering the chamber between the rotating cone and the stationary disk by centrifugal force, generating high shear (APV, 2011). Emulsions made with homogenizers or colloid mills require initial or primary homogenization with a high speed mixer, since they are more efficient at reducing droplet size of an existing emulsion than at initial homogenization (McClements,

2005). A high shear disperser is a rotor-stator system, similar to a colloid mill (Weiss, 2008), that does not require initial homogenization with a high speed mixer. Of these four methods, the high speed mixer and high shear disperser are standard methods of preparation for pourable dressings. The samples in this study were prepared with a Benchmix OPLB-250 high shear disperser, manufactured by Admix.

Emulsion stability can be measured in different ways. The basic premise of emulsion instability is that over time, since an emulsion is a thermodynamically unstable system, droplets will merge or "coalesce" into larger droplets. In an O/W emulsion, this coalescence will eventually cause creaming or "oiling off" (McClements, 2005). Change in droplet size and size distribution can be measured over time with methods such as microscopy, light scattering (in which a beam of light passed through the emulsion is analyzed based on the angle of refraction), ultrasonic spectrometry (using the same principle as light scattering but with ultrasonic waves), and electrical pulse counting (which measures change in electrical conductivity). Change in rheology can also be used as an indicator of droplet coalescence; as droplet size increases, viscosity decreases (Gaonkar, 2000). It should be noted that unlike Newtonian fluids, which maintain a constant viscosity at various shear rates, emulsions exhibit a decrease in viscosity with increased shear rate, known as "shear-thinning," and are considered to be non-Newtonian fluids (Brookfield Engineering, 2005). To compare emulsion viscosities, it is important to adhere to the same testing parameters. Creaming can also be measured directly, by visual observation. In this study, emulsion stability was evaluated over 8 weeks by measurement of creaming in graduated cylinders and by change in viscosity, measured by a Brookfield RVT dial viscometer. Samples were held and measured at  $68 - 70^{\circ}$  F.

A number of factors affect emulsion stability. Formulation parameters affecting stability include the amount and type of stabilizer used and the amount of oil in the O/W emulsion (Gaonkar, 2000) often expressed as a volume fraction or mass fraction. Process parameters include the shear applied in forming the emulsion (Gaonkar, 2000) because shear determines droplet size and the smaller the droplets, the longer it will take for coalescence and creaming to occur. Storage parameters affecting stability include temperature and time. As temperature increases, oil viscosity decreases which disrupts droplet stability; in addition, higher

temperatures can render some emulsifiers ineffective (McClements, 2005). Moreover, since all emulsions will destabilize eventually, time is a critical consideration in assessing emulsion stability and determining product shelf life.

## **Chapter 2 - Materials and Methods**

#### **Materials**

#### **Ingredients**

Xanthan gum as Keltrol 521 (CP Kelco, Atlanta, GA), propylene glycol alginate as Satialgine PGA 200 (Degussa Texturant Systems, Atlanta, GA), proprietary gum blend as Saladizer 250 Powder (TIC Gums, Belcamp, MD), viscosifying modified corn starch as Ultra-Sperse M (National Starch, Bridgewater, NJ), emulsifying modified corn starch as N-creamer 46 (National Starch), microcrystalline cellulose as Tabulose SC-611 (Blanver USA, Boca Raton, FL), liquid salted egg yolks (Ballas Egg Products, Zanesville, OH), liquid whole eggs (Ballas Egg Products), dried egg whites as Type H-40 (Ballas Egg Products) and soybean oil (ADM, Decatur, IL) were kindly donated by the respective suppliers.

Ingredient	Stabilizer category
Xanthan gum	Texture modifier
Propylene glycol alginate	Texture modifier, emulsifier
Blend of xanthan gum, guar gum, and sodium alginate	Texture modifier
Viscosifying modified corn starch	Texture modifier
Emulsifying modified corn starch	Texture modifier, emulsifier
Microcrystalline cellulose	Texture modifier, emulsifier
Liquid salted egg yolks	Emulsifier
Liquid salted whole eggs	Emulsifier
Dried egg whites	Texture modifier

Table 1: Stabilizer categories of ingredients used in high-oil emulsion stability test

	60% soybean	60% soybean	60% soybean	70% soybean	70% soybean	70% soybean
	oil	oil	oil	oil	oil	oil
Xanthan gum	0.15%	0.20%	0.25%	0.15%	0.20%	0.25%
Propylene glycol alginate	0.15%	0.20%	0.25%	0.15%	0.20%	0.25%
Blend of xanthan gum, guar gum, and sodium alginate	0.15%	0.20%	0.25%	0.15%	0.20%	0.25%
Viscosifying modified corn starch	1%	2%	3%	1%	2%	3%
Emulsifying modified corn starch	1%	2%	3%	1%	2%	3%
Microcrystalline cellulose	1%	2%	3%	1%	2%	3%
Liquid salted egg yolks	0.5%	1%	1.5%	0.5%	1%	1.5%
Liquid salted whole eggs	0.5%	1%	1.5%	0.5%	1%	1.5%
Dried egg whites	0.2%	0.4%	0.6%	0.2%	0.4%	0.6%

Table 2: Formulations used in high-oil emulsion stability test

#### Equipment

Equipment used included an Admix Benchmix OPLB-250 high shear disperser, Brookfield RVT dial viscometer, 100-mL plastic graduated cylinders (108), 8-fl oz glass jars with lids (486), 2000-mL plastic beakers, aluminum foil, and a gram scale.

#### Methods

#### Sample preparation

Each of the nine stabilizers were tested at three concentrations, based on manufacturers' recommendations, both in a 60% soybean oil emulsion and a 70% soybean oil with the balance as water, for a total of 54 treatments. There were no replications.

For each formula in the matrix (Table 2), a 2000-g batch was made. The stabilizer was weighed into a weigh boat. Water and oil were weighed into separate 2000-mL beakers. Then, the beaker of water was placed under the Benchmix high shear disperser, set to 8000 rpm. The mixer was turned on and the stabilizer was immediately added to the water. The mixing continued for 3 minutes to ensure hydration of the stabilizer. Next, the oil was added to the

water-stabilizer blend and mixing continued for an additional 2 minutes. Mixing parameters were based on recommendations from Admix, the Benchmix manufacturer. Finally, the batch was poured into 9 8-fl oz jars at 150 g each and 2 100-mL graduated cylinders at 100 mL each. Lids were put on the jars and aluminum foil was used to cover the graduated cylinders.

#### Sample storage

All samples were stored undisturbed in a cabinet in the dark at  $68 - 70^{\circ}$  F and removed only for sample measurement.

#### Sample measurement

Creaming was measured at 1 day and at weekly intervals thereafter for 8 weeks. Creaming was measured as a percent of total volume by visual observation of the samples in the 100-mL graduated cylinders. For example, if no oil had separated from the sample, then the recorded value would be 0%. If all the oil in a 60% oil sample had separated out, then the recorded value would be 60%. Creaming values were taken in duplicate, from 2 graduated cylinders, and averaged. Viscosity was measured at day 1 and at weekly intervals thereafter for 8 weeks or until creaming reached 10%. A 10% cutoff was determined because at that level of oiling off, viscometer readings can become inaccurate as the spindle is partially submerged in oil. Viscosity was measured in centipoise (cP) with a Brookfield dial viscometer on the 8-fl oz jar samples. After each scheduled measurement, the sample was discarded, so that any agitation caused by the viscometer would not affect the next scheduled viscosity measurement.

### **Chapter 3 - Results and Discussion**

#### Gums

Of the three gum ingredients, only xanthan gum at the highest level (0.25%) in the 70% oil sample prevented creaming at 8 weeks (Figure 2), with an average viscosity of 7900 cP, and a change in viscosity of -3000 cP (Table 4). In the 60% oil samples, creaming reached or exceeded 10% of the total volume at 1 week, 3 weeks, and 8 weeks, respectively, for the 0.15%, 0.20%, and 0.25% concentrations (Figure 1). In the 70% oil samples, creaming reached 10% at 2 weeks for the 0.15% and exceeded 10% at 4 weeks for the 0.20% concentrations (Figure 2). In the samples with the lowest levels of xanthan gum, complete oil separation was seen during the study, i.e., 60% in the 60% oil sample (Figure 1) and 70% in the 70% oil sample (Figure 2). While Hemar et al. (2001) reported that xanthan gum at a critical concentration ( $\leq 0.2\%$  in a 30%) oil emulsion with sodium caseinate) can increase the rate of creaming due to depletion flocculation, all levels of xanthan gum tested were seen to provide increased resistance to creaming in this study. Depletion flocculation occurs when polymer molecules are "excluded from the space between two approaching particles due to their large size" causing osmotic pressure that forces the droplets together (Stechemesser and Dobias, 2005). Dickinson (2009) reports that at very low concentrations, depletion flocculation can be destabilizing, but that at higher concentrations, droplets flocculate into a viscoelastic gel-like network that inhibits creaming. Parker et al. (1995) reported delayed creaming at higher xanthan concentrations, and identified three distinct phases of creaming: initial delay, linear creaming, and then cream compression. Similarly, higher concentrations of xanthan delayed creaming in our study. Additionally, Parker (1995) reported that the length of the delay phase increased as the oil fraction increased, likewise seen here, with 0.25% xanthan gum delaying creaming in the 70% oil emulsion longer than in the 60% oil emulsion (Figures 1 and 2). The stability against creaming seen in our study also agrees with the results of Krstonisic et al. (2009), who evaluated creaming stability of 50% oil emulsions containing Tween 80, and found that xanthan concentrations of 0.08%, 0.12%, and 0.20% prevented creaming over 15 days. In our 8-week study, several of the lower xanthan concentrations failed to prevent creaming for the full duration, but at 15 days, there was good stability against creaming at 0.20% and 0.25%

concentrations in the 60% oil emulsions (Figure 1), and at 0.15%, 0.20%, and 0.25% concentrations in the 70% oil emulsions (Figure 2). Krstonosic's (2009) results also showed no viscosity change in any of the xanthan samples over 15 days. Our results showed a slight decrease in viscosity over that time (Figures 1 and 2), though it should be noted that our study used higher volume fractions of oil and also did not use additional emulsifiers such as Tween 80. Moreover, research by Krstonosic (2009) noted a decrease in average droplet size with increased xanthan concentrations. Paraskevopoulou et al. (2005) found 0.30% - 0.45% xanthan gum used with 0.5% - 1% gum arabic or PGA to be effective at preventing creaming of 50% oil emulsions with low pH, but did not test lower xanthan gum concentrations.

The propylene glycol alginate samples behaved similarly to the xanthan gum samples overall, but provided less stability at the same levels. The propylene glycol samples exceeded 10% creaming at 1 week, 2 weeks, and 5 weeks, respectively, for the 0.15%, 0.20%, and 0.25% concentrations in the 60% oil samples (Figure 3), and at 1 week, 1 week, and 3 weeks in the 70% oil samples (Figure 4). The highest level (0.25%) of propylene glycol alginate was much more effective at stabilizing the 60% and 70% oil emulsions than the 0.20% and 0.15% levels. At the lower concentrations, nearly total oil separation was observed during the study (Figures 3 and 4). Research by Pettitt et al. (1995) investigated using propylene glycol alginate in conjunction with xanthan gum in both aqueous solutions and in 40% oil emulsions. Compared to a xanthan only solution, a blend of xanthan with propylene glycol alginate lessened the pseudoplasticity of the solutions, which could be a texture benefit. However, a reduction in pseudoplasticity of the 40%oil emulsions was not seen although xanthan gum and propylene glycol are commonly used together in salad dressings, presumably for the thickening effect of xanthan and the texture or flow properties of propylene glycol alginate (Pettitt, 1995). Moreover, results showed that the emulsions made with xanthan gum or a blend of xanthan and propylene glycol alginate had much smaller droplets than emulsions made with propylene glycol alginate only. Likewise, Yilmazer et al. (1991) found smaller droplets in 40% oil emulsions stabilized with 0.4% xanthan gum than with 0.4% blends of xanthan gum and propylene glycol alginate. Moreover, when propylene glycol alginate was added, viscosity decreased and some coalescence occurred. Overall, the use of propylene glycol alginate seems to destabilize xanthan emulsions, but its use could still be considered in very stable dressings as a means of modifying texture.

In the proprietary gum blend samples, creaming exceeded 10% at 2 weeks, 3 weeks, and 4 weeks for the 0.15%, 0.20%, and 0.25% levels, respectively, in the 60% oil samples (Figure 5), and at 3 weeks, 1 week, and 2 weeks in the 70% oil samples (Figure 6). In the 60% oil samples, increased gum blend levels provided increased emulsion stability (Figure 5), but in the 70% oil samples, the lowest level (0.15%) of gum blend provided much greater control of creaming, exhibiting only 13% creaming at 8 weeks compared to 60 - 70% at the higher levels (Figure 6). This could indicate that a critical concentration had been reached for the gum blend in the 70% oil emulsion, at which depletion flocculation occurred and increased the rate of creaming. Perhaps this was not seen in the 60% oil emulsion because, while the total gum concentrations are the same, there is a higher gum concentration per aqueous phase in the 70% emulsion as compared to the 60% emulsion. For all levels tested, higher concentrations produced higher viscosities (Figures 5 and 6). This gum blend is a synergistic blend of xanthan gum, guar gum, and sodium alginate, and in work by Pellicer et al. (2000), another gum system shown to have viscous synergism is xanthan gum and locust bean gum. This may be another option to improve the stabilization ability of xanthan gum.

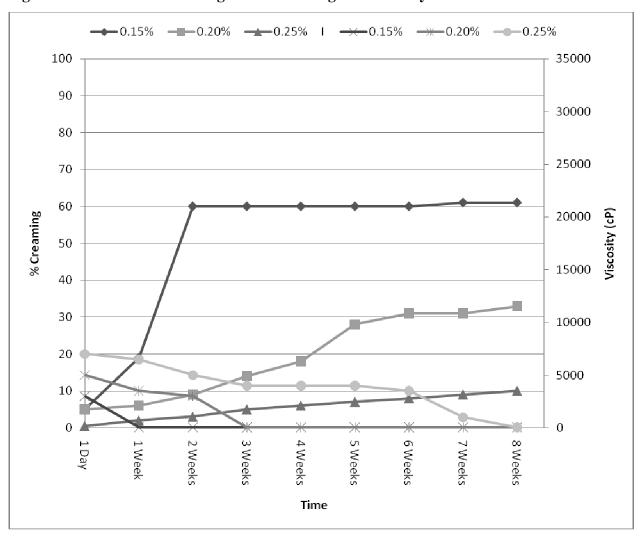


Figure 1: Effect of % xanthan gum on creaming and viscosity of 60% oil emulsion

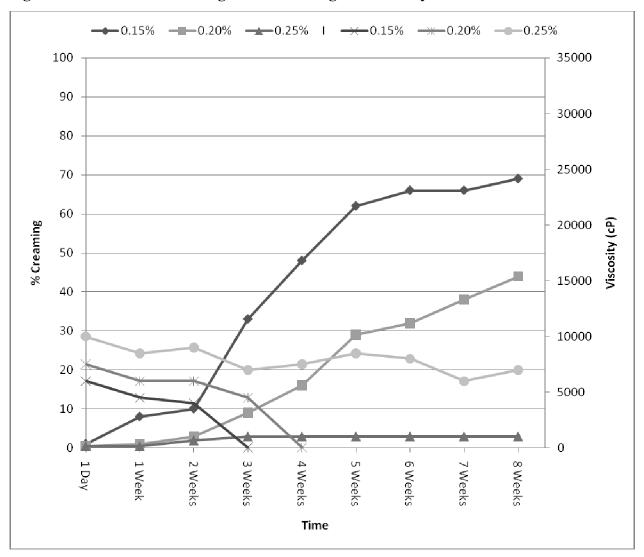


Figure 2: Effect of % xanthan gum on creaming and viscosity of 70% oil emulsion

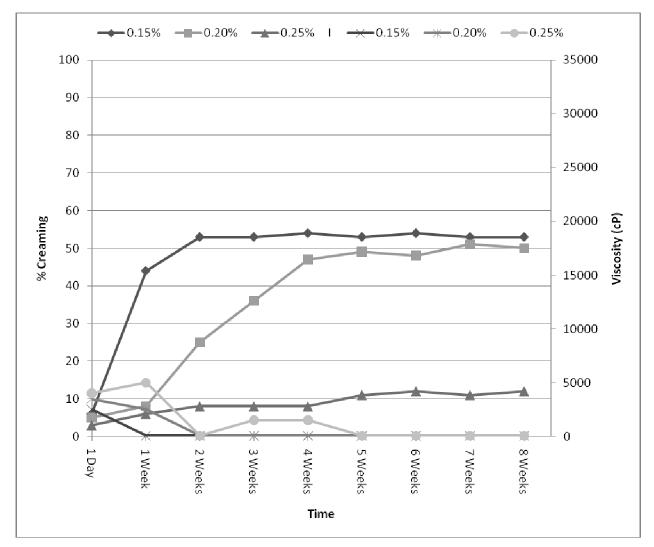


Figure 3: Effect of % propylene glycol alginate on creaming and viscosity of 60% oil emulsion

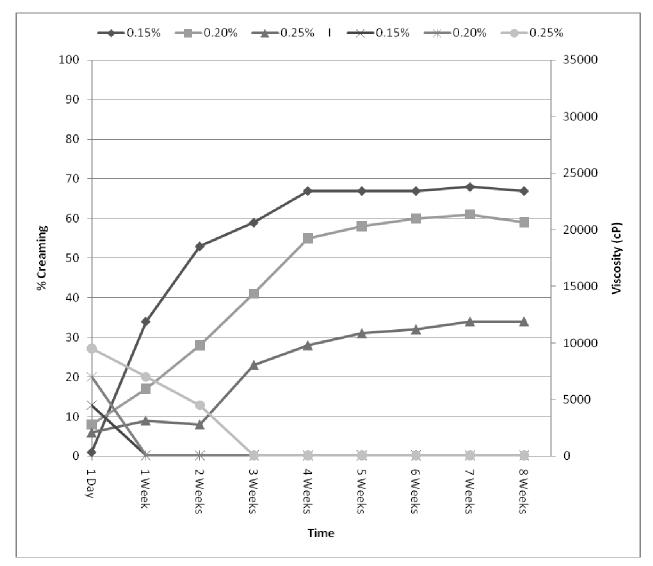


Figure 4: Effect of % propylene glycol alginate on creaming and viscosity of 70% oil emulsion

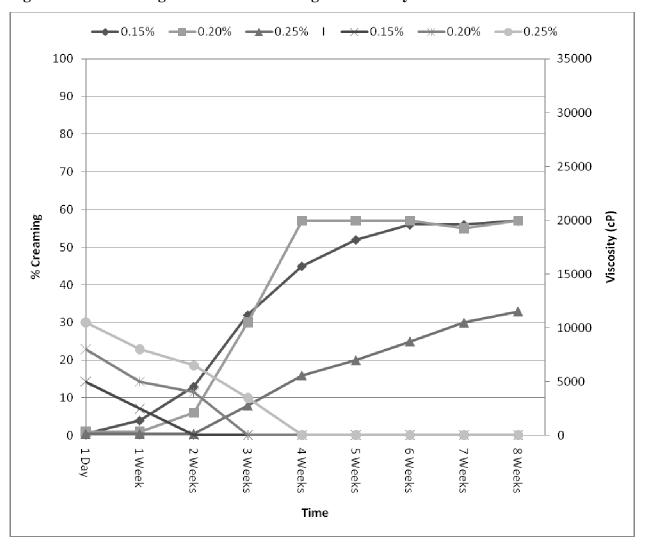


Figure 5: Effect of % gum blend on creaming and viscosity of 60% oil emulsion

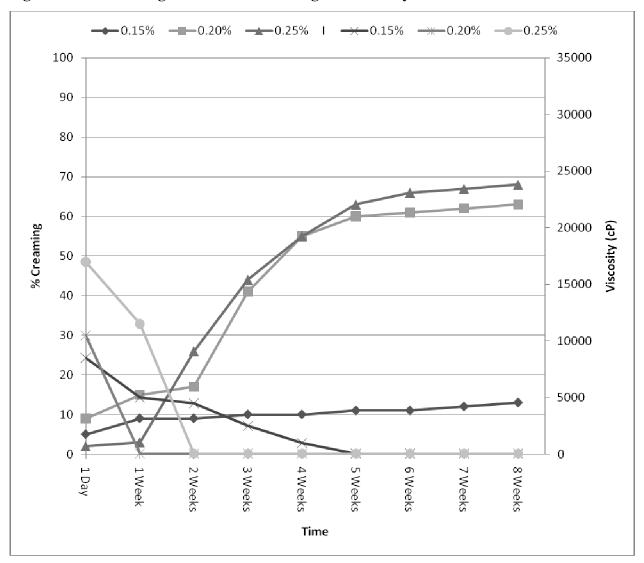


Figure 6: Effect of % gum blend on creaming and viscosity of 70% oil emulsion

#### **Starches and Cellulose**

Of the starch ingredients, only the emulsifying modified corn starch at 2% in the 70% oil sample prevented creaming at 8 weeks (Figure 8), with an average viscosity of 9900 cP, and a steep change in viscosity of -11,000 (Table 4). In the 60% oil samples, creaming exceeded 10% at 1 week for the lowest level (1%), 3 weeks for the highest level (3%), and 8 weeks for the intermediate level (2%) (Figure 7). Similarly, the lowest and highest levels provided the least stabilization in the 70% oil samples with creaming exceeding 10% by 2 weeks and 1 day, respectively, while the intermediate level exhibited no creaming at 8 weeks (Figure 8). In both

the 60% and 70% oil samples, the emulsifying starch at 2% provided the most resistance to creaming, followed by the 3%, which provided some resistance throughout (Figures 7 and 8). This could imply that a critical concentration exists for the emulsifying starch above 2% concentration, at which the rate of creaming is accelerated through depletion flocculation. The samples with emulsifying starch at the lowest level (1%) exhibited nearly complete oil separation during the study (Figures 7 and 8).

Of the viscosifying modified corn starch samples, nearly total oil separation was observed at 1 day at all concentrations with the exception of the intermediate (2%) concentration in 60% oil, which reached 10% creaming at 2 weeks (Figures 9 and 10). Viscosity readings were only taken on this sample.

The microcrystalline cellulose samples with 60% oil showed excellent emulsion stability at all concentrations, with no creaming at 8 weeks (Figure 11). The higher concentrations gave higher viscosities, with average viscosities of 23,400 cP at 3%, 10,500 cP at 2%, and 3300 cP at 1% (Table 3). These same concentrations in the 70% oil samples, however, failed to prevent creaming, with the highest and intermediate levels creaming by 1 week, but the lowest level remaining stable until week 6 (Figure 12), again implying a critical concentration had been reached. The average viscosities produced by the cellulose were relatively high compared to those produced by the other ingredients in the study, a finding also noted by Fonseca et al. (2009) who tested mixtures of xanthan gum, PGA, and carboxymethylcellulose in salad dressings containing whey at levels of 1% stabilizer blend and 7% oil. The samples remained stable for the 4-month study, and were noted to have viscosities most highly influenced by the carboxymethylcellulose. Moreover, Akiyama et al. (2007) showed that viscosity produced by some cellulose derivatives can be further increased by increasing the hydrophobic/hydrophilic ratio through chemical modification. It should be noted that in our samples, an increase in viscosity over 8 weeks was observed (+1500 cP for 3%, +5500 for 2%, and +4000 for 1% microcrystalline cellulose in the 60% oil samples) (Table 3). Sun et al. (2007) observed, regarding the effect of a modified cellulose on 50% oil emulsions, that not only will viscosity increase with increased cellulose concentration, but at higher concentrations an elastic gel is formed, which was described as "a transient three-dimensional network."

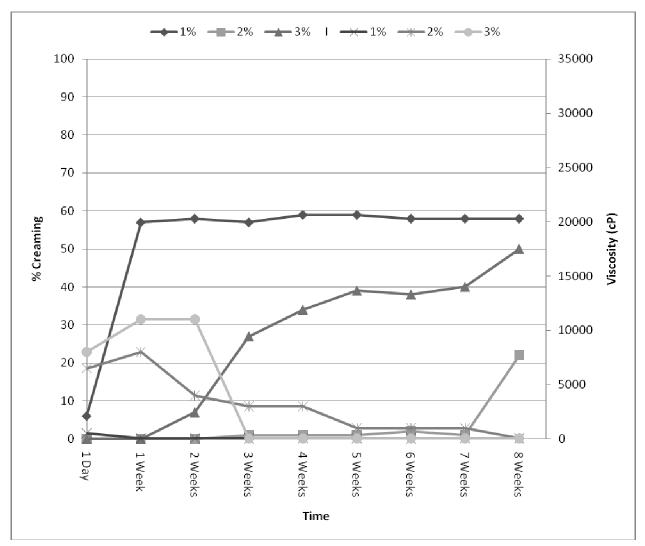


Figure 7: Effect of % emulsifying modified corn starch on creaming and viscosity of 60% oil emulsion

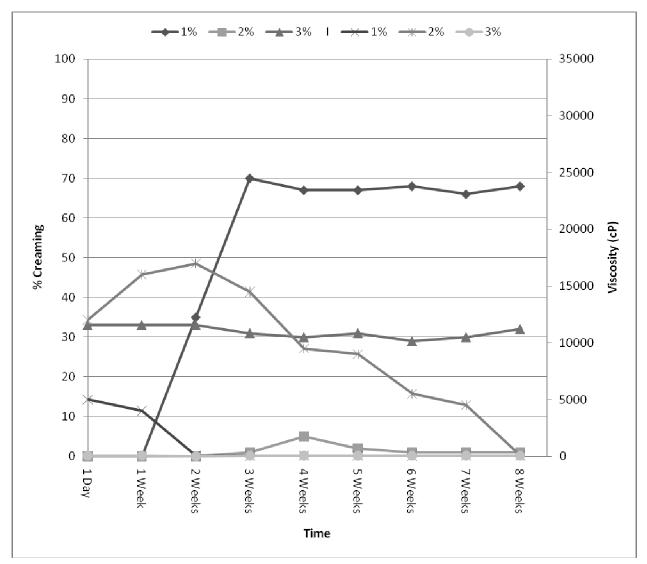


Figure 8: Effect of % emulsifying modified corn starch on creaming and viscosity of 70% oil emulsion

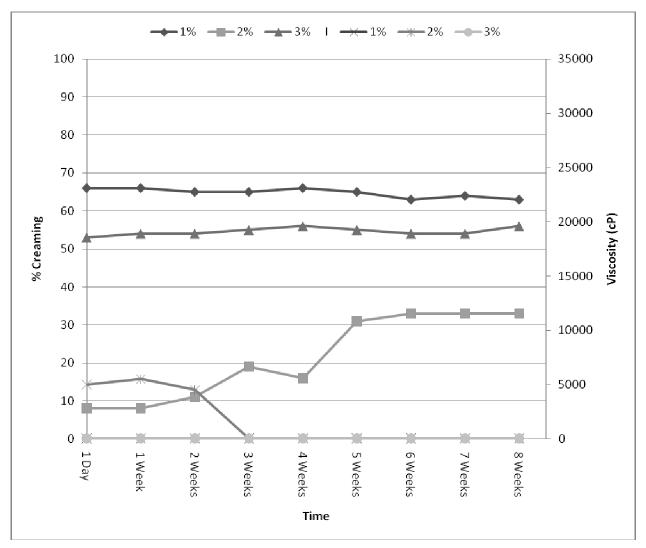


Figure 9: Effect of % viscosifying modified corn starch on creaming and viscosity of 60% oil emulsion

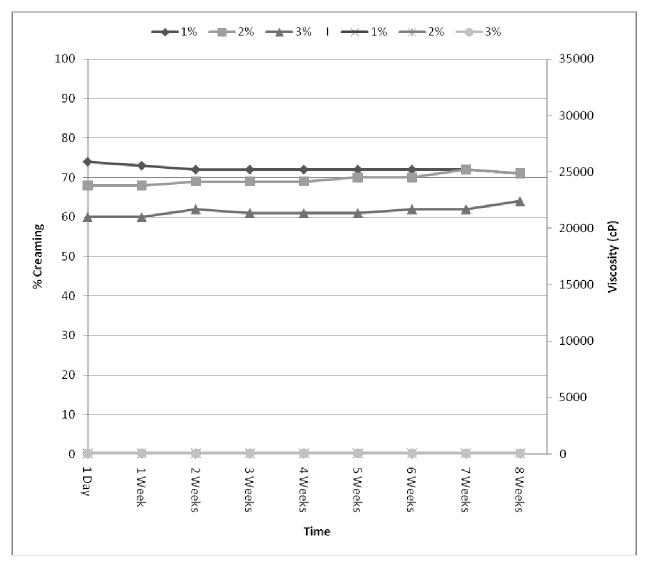


Figure 10: Effect of % viscosifying modified corn starch on creaming and viscosity of 70% oil emulsion

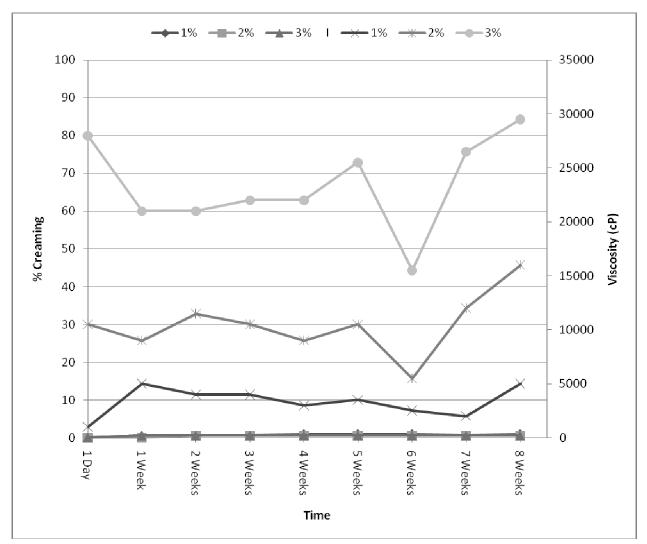


Figure 11: Effect of % microcrystalline cellulose on creaming and viscosity of 60% oil emulsion

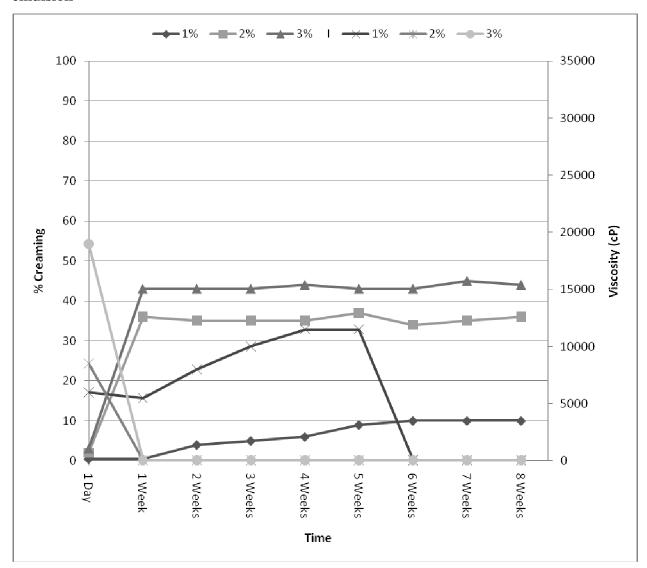


Figure 12: Effect of % microcrystalline cellulose on creaming and viscosity of 70% oil emulsion

### **Egg Products**

Of the egg ingredients, only the dried egg whites prevented creaming at 8 weeks. The effective concentrations were 0.4% and 0.6% in the 60% oil samples (Figure 13) and 0.2%, 0.4%, and 0.6% in the 70% oil samples (Figure 14). In the 60% oil samples, the 0.2% dried egg white concentration exceeded 10% creaming at 2 weeks (Figure 13). While several concentrations were effective at preventing creaming, there was nonetheless a large drop in viscosity over time in both the 60% and 70% oil samples, with a greater drop seen in the 70% oil

samples (Figures 13 and 14). In the 60% oil samples, change in viscosity was -6500 cP at 0.6% dried egg whites and -5000 cP at 0.4% dried egg whites (Table 3). The 70% samples showed viscosity changes of -7000 cP, -9000 cP, and -7500 cP, respectively, for 0.6%, 0.4%, and 0.2% dried egg white concentrations (Table 4). Increased concentrations reliably produced increased sample viscosities. Average viscosities were 2300 cP, 2800 cP, and 3100 cP for 0.2%, 0.4%, and 0.6% concentrations, respectively, in the 70% oil samples (Table 4). In the 60% oil samples, average viscosities were 2100 cP and 2300 cP for 0.4% and 0.6%, respectively (Table 3). Low viscosities for egg-white stabilized emulsions were also seen by Ercelebi and Ibanoglu (2009), who investigated the stabilizing effects of pectin and guar gum on 25% oil emulsions containing egg white. The addition of pectin and guar gum not only increased viscosities but also produced emulsions with smaller droplets than the emulsions stabilized by egg white only. Dried egg whites may be most suitable as a dressing stabilizer as one component of a stabilizer blend.

For the liquid salted egg yolks, creaming reached 10% at 3 weeks, 4 weeks, and 3 weeks in the 60% oil samples for 0.5%, 1.0%, and 1.5% concentrations, respectively (Figure 15). In the 70% oil samples, creaming was noted at 4 weeks, 6 weeks, and 7 weeks for 0.5%, 1.0%, and 1.5% concentrations, respectively (Figure 16). In a study by Riscardo et al. (2003), who measured droplet size distribution of egg yolk-stabilized emulsions, droplet size was found to decrease with increased egg yolk concentrations. Riscardo (2003) used egg yolk concentrations as high as 8% in 35% oil emulsions containing salt, so perhaps the levels used in this study were too low for maximum stabilization.

The liquid salted whole eggs also failed to stabilize the emulsions. In the 60% oil samples, creaming exceeded 10% of the total volume at 1 week, 2 weeks, and 1 week, respectively, for the 0.5%, 1.0%, and 1.5% concentrations (Figure 17). In the 70% oil samples, creaming also exceeded 10% quickly: 1 week for the 0.5% concentration, 1 day for the 1.0% concentration, and 1 week for the 1.5% concentration (Figure 18). As with the liquid egg yolks, perhaps the levels used in this study were too low to effectively stabilize the emulsions. Additionally, there is mounting evidence of emulsion stability derived from protein-polysaccharide interactions at the emulsion interface (Dickinson, 2009) that could be applied in further emulsion studies with egg ingredients. Diffis et al. (2005) suggests that a protein-

polysaccharide conjugate is well-suited for salad dressing emulsions. Use of egg ingredients in conjunction with a starch or gum, for example, may provide effective stabilization of a high-oil dressing. A protein-polysaccharide emulsion can be prepared in one of two ways. In a mixed emulsion system, the protein and polysaccharide are first mixed together to form a complex; in a bilayer emulsion, the emulsion is initially made using protein as the emulsifer, and the polysaccharide is added afterward so it can adsorb onto the protein layer to form a second layer (Jourdain et al., 2008). Heating of a protein-polysaccharide conjugate can also affect its ability to stabilize emulsions (Diftis, 2005). Results showed that increased heating of soy protein isolatedextran conjugates, which caused increased protein-protein interactions, produced a 50% oil emulsion exhibiting depletion flocculation, i.e., a thicker emulsion than was produced at shorter heating times, but one that is possibly less stable over time. While protein-polysaccharides provide a possible solution to dressing stability, it should be noted that oil droplets coated by polysaccharides provide much better stability to environmental stress such as change in temperature, salt, and pH than droplets coated by globular proteins (Charoen et al., 2011). Since proteins stabilize oil droplets primarily by electrostatic repulsion, change in electrostatic interactions can weaken the emulsion, whereas polysaccharide emulsions are more robust because the primary mode of action is steric repulsion.

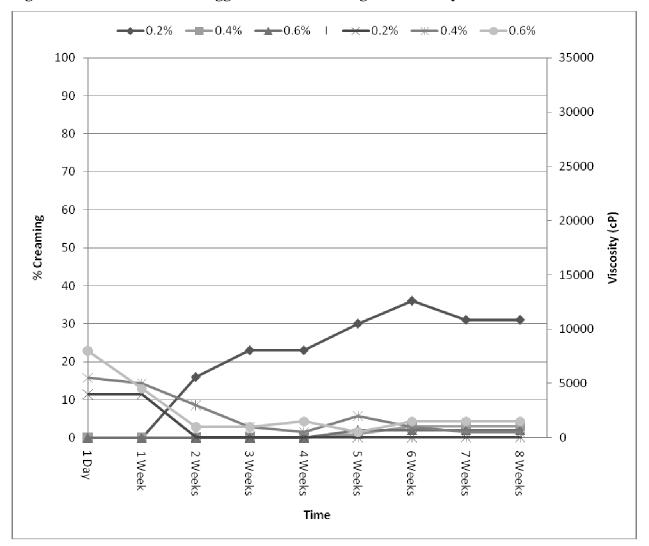


Figure 13: Effect of % dried egg whites on creaming and viscosity of 60% emulsion

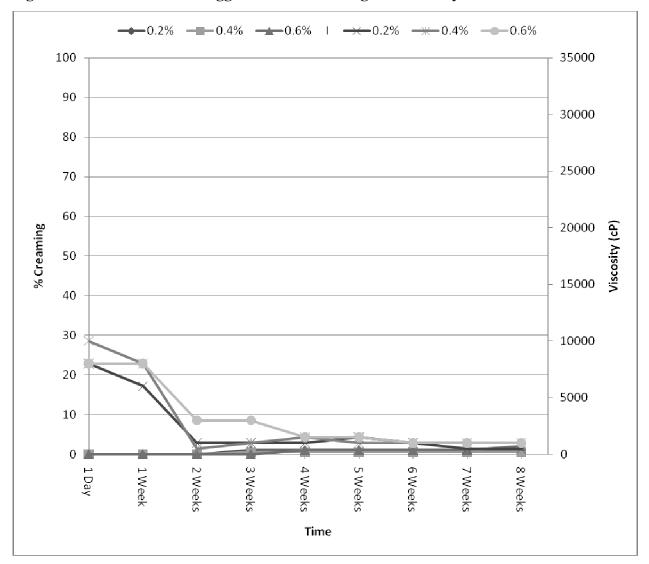


Figure 14: Effect of % dried egg whites on creaming and viscosity of 70% emulsion

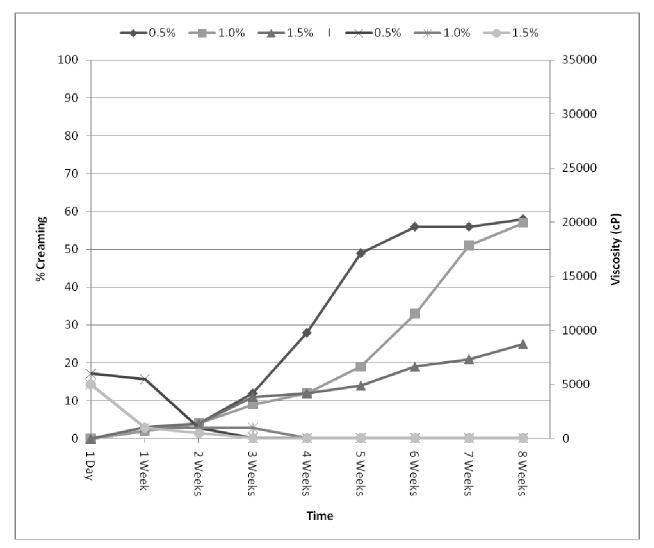


Figure 15: Effect of % liquid salted egg yolks on creaming and viscosity of 60% oil emulsion

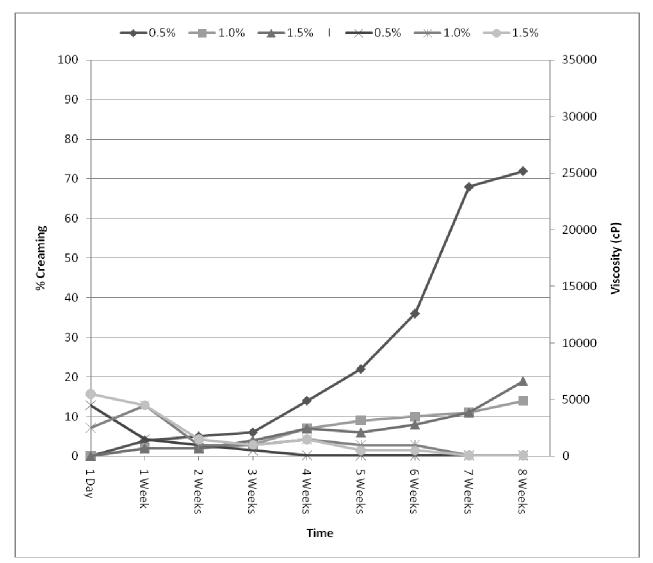


Figure 16: Effect of % liquid salted egg yolks on creaming and viscosity of 70% oil emulsion

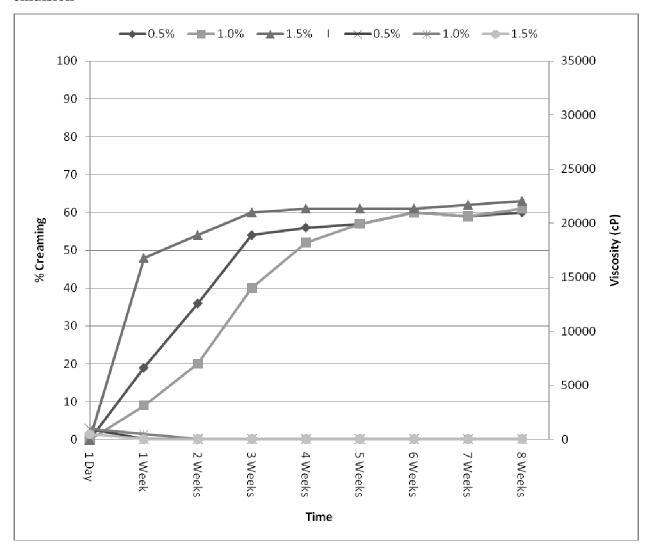


Figure 17: Effect of % liquid salted whole eggs on creaming and viscosity of 60% oil emulsion

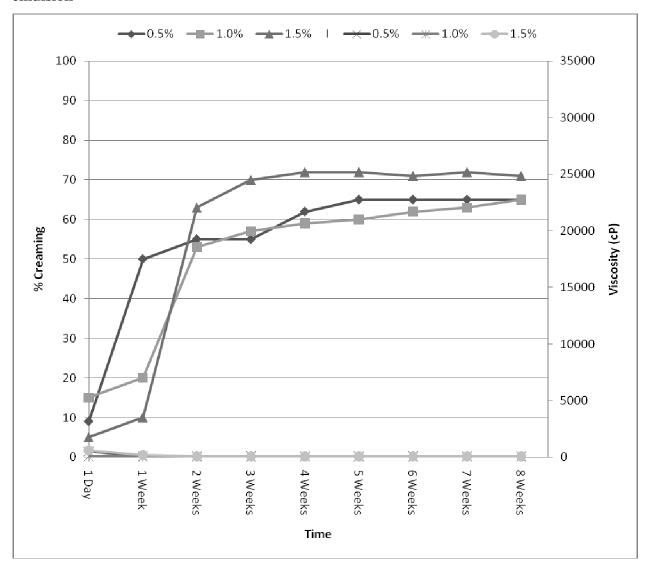


Figure 18: Effect of % liquid salted whole eggs on creaming and viscosity of 70% oil emulsion

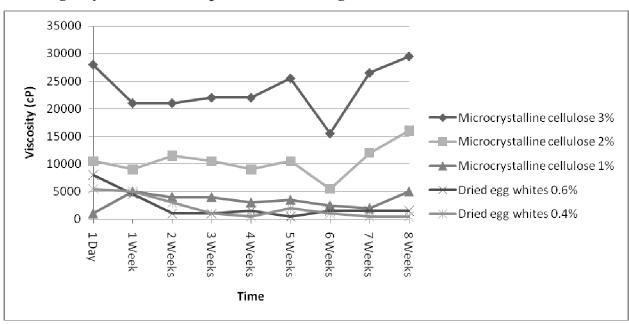


Figure 19: Effect of stabilizer type and concentration on viscosity of 60% oil emulsion including only stabilizers that prevented creaming

 Table 3: Average viscosity and change in viscosity of 60% oil emulsion by stabilizer type

 including only stabilizers that prevented creaming

	Average viscosity (cP)	Change in viscosity (cP)
Microcrystalline cellulose 3%	23,400	1500
Microcrystalline cellulose 2%	10,500	5500
Microcrystalline cellulose 1%	3300	4000
Dried egg whites 0.6%	2300	-6500
Dried egg whites 0.4%	2100	-5000

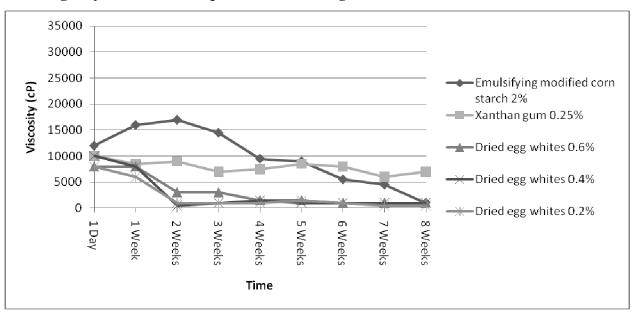


Figure 20: Effect of stabilizer type and concentration on viscosity of 70% oil emulsion including only stabilizers that prevented creaming

Table 4: Average viscosity and change in viscosity of 70% oil emulsion by stabilizer type including only stabilizers that prevented creaming

	Average viscosity (cP)	Change in viscosity (cP)
Emulsifying modified corn starch 2%	9900	-11,000
Xanthan gum 0.25%	7900	-3000
Dried egg whites 0.6%	3100	-7000
Dried egg whites 0.4%	2800	-9000
Dried egg whites 0.2%	2300	-7500

## **Chapter 4 - Conclusions**

The ingredients most effective at preventing creaming of an O/W emulsion of 60% oil for 8 weeks were microcrystalline cellulose at 3%, 2%, and 1% and dried egg whites at 0.6% and 0.4%. The microcrystalline cellulose produced a wide viscosity range, which would allow for a wide array of textural options for this dressing. These samples maintained their viscosity over time, even showing an increase. Viscosity should be monitored beyond 8 weeks to see if the viscosity levels off or continues to increase. Additionally, any texture change such as gelling

should be monitored. The dried egg white samples, on the other hand, showed a sharp drop in viscosity which appeared within the first 2 weeks, and so did not effectively stabilize this formula. However, dried egg whites could be tested at higher concentrations. Additional stabilizer options may include xanthan gum at a concentration higher than 0.25%, since the highest level tested (0.25%) did not exhibit creaming until the final week.

The ingredients most effective at preventing creaming of an O/W emulsion of 70% oil for 8 weeks were the emulsifying modified corn starch at 2%, xanthan gum at 0.25%, and dried egg whites at 0.6%, 0.4%, and 0.2%. While the emulsifying starch at 2% was highly effective in preventing creaming, the sample exhibited a steady decrease in viscosity over 8 weeks, and so cannot be considered to be an effective stabilizer in this formula. Xanthan gum at 0.25% was able to both prevent creaming and maintain a relatively constant viscosity over 8 weeks. While this concentration is promising, xanthan gum at higher concentrations could also be tested. The dried egg whites did prevent creaming, but again the samples showed a sharp drop in viscosity within 2 weeks and so did not effectively stabilize this formula.

The next steps include making real formulas and bench top samples for 60% oil versions with microcrystalline cellulose (at 1%, 2%, and 3%), dried egg whites (at >0.6%), and xanthan gum (at >0.25%), and for 70% oil versions with xanthan gum (at 0.25% and >0.25%) and dried egg whites (at >0.6%). These formulas will include vinegar, salt, sugar, spices, colorants, and flavors as needed, so emulsion stability can be reevaluated in this more complex system, especially as both acid and salt can play a role in destabilizing emulsions. These samples will then be put into shelf life testing at 70°F and also at 90°F to establish an accelerated shelf life program for this unique product. The most promising of these high-oil dressings will then be evaluated in dressing, sauce, and baking applications to ensure excellent performance across the spectrum and to identify the optimal target viscosity and oil level, especially as emulsions with higher fat contents can be rated as thicker and creamier independently of viscosity (Van Aken, et al., 2011). When the dressing has been shown to meet consumer expectations, and an appropriate shelf life has been determined, it will then be commercialized.

## References

- Akiyama, Eri, Takamasa Yamamoto, Yuuko Yago, Hajime Hotta, Takeshi Ihara, and Tomohito Kitsuki. "Thickening properties and emulsification mechanisms of new derivatives of polysaccharide in aqueous solution 2. The effect of the substitution ratio of hydrophobic/hydrophilic moieties." *Journal of Colloid and Interface Science*, 2007: 438-446.
- APV. Homogenizer Handbook: Processing of Emulsions and Dispersions. 2011. http://www.apv.com/pdf/catalogs/Process\_E\_D\_By\_Homog\_3005\_01\_06\_2008\_US.pdf (accessed 2011).
- Breeding, Carol and R. Scott Beyer. "Eggs." In *Food Chemistry: Principles and Applications*, by Genevieve Christen and J. Scott Smith, 421-431. West Sacramento:Science Technology System, 2000.
- Brookfield Engineering. *What is Viscosity*. 2005. http://www.brookfieldengineering.com/education/what-is-viscosity.asp (accessed 2011).
- Charoen, Ratchanee, Anuvat Jangchud, Kamolwan Jangchud, Thepkunya Harnsilawat, Onanong Naivikul, and David Julian McClements. "Influence of Biopolymer Emulsifer Type on Formation and Stability of Rice Bran Oil-in-Water Emulsions: Whey Protein, Gum Arabic, and Modified Starch." *Journal of Food Science - Vol. 76, No. 1*, 2011: 165-172.
- Daniel, James and Connie Weaver. "Carbohydrates: Functional Properties." In *Food Chemistry: Principles and Applications*, by Genevieve Christen and J. Scott Smith, 55-78. West Sacramento:Science Technology System, 2000.
- Dickinson, Eric. "Hydrocolloids as emsulsifiers and emulsion stabilizers." *Food Hydrocolloids* 23, 2009: 1473-1482.
- Diftis, N.G., C.G. Biliaderis, and V.D. Kiosseoglou. "Rheological properties and stability of model salad dressing emulsions prepared with a dry-heated soybean protein isolate-dextran mixture." *Food Hydrocolloids 19*, 2005: 1025-1031.
- Ercelebi, E. Alben, and E. Ibanoglu. "Characterization of Phase Separation Behavior, Emulsion Stability, Rheology, and Microstructure of Egg White-Polysaccharide Mixtures." *Journal of Food Science - Vol. 74, No. 6*, 2009: 506-512.
- FDA. "Sec. 169.140 Mayonnaise." *Code of Federal Regulations Title 21*. 2010. http://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcfr/CFRSearch.cfm?fr=169.140&Se archTerm=mayonnaise (accessed 2011).
- Fonseca, Vania, Charles Haminiuk, Dayane Izydoro, Nina Waszczynskyj, Agnes Scheer, and Maria-Rita Sierakowski. "Stability and rheological behaviour of salad dressings obtained with whey and different combinations of stabilizers." *International Journal of Food Science and Technology* 44, 2009: 777-783.
- Gaonkar, Anil. "Emulsion Basics." *Salad Dressings & Sauces 101: A Short Course*. Baltimore, MD: Association for Dressings & Sauces, 2000.
- Hemar, Y., M. Tamehana, P.A. Munro, and H. Singh. "Influence of xanthan gum on the formation and stability of sodium caseinate oil-in-water emulsions." *Food Hydrocolloids* 15, 2001: 513-519.

- Jane, Jay-lin. "Carbohydrates: Basic Concepts." In Food Chemistry: Principles and Applications, by Genevieve Christen and J. Scott Smith, 35-53. West Sacramento:Science Technology System, 2000.
- Jourdain, J., M.E. Leser, C. Schmitt, M. Michel, and E. Dickinson. "Stability of emulsions containing sodium caseinate and dextran sulfate: relationship to complexation in solution." *Food Hydrocolloids* 22, 2008: 647-659.
- Krstonosic, Veljko, Ljubica Dokic, Petar Dokic, and Tamara Dapcevic. "Effects of xanthan gum on physicochemical properties and stability of corn oil-in-water emulsions stabilized by polyoxyethylene (20) sorbitan monooleate." *Food Hydrocolloids 23*, 2009: 2212-2218.
- McClements, David Julian. *Food Emulsions: Principles, Practices, and Techniques, 2nd Ed.* Boca Raton, FL: CRC Press, 2005.
- Paraskevopoulou, Adamantini, Dimitrios Boskou, and Vassilis Kiosseoglou. "Stabilization of olive oil - lemon juice emulsion with polysaccharides." *Food Chemistry* 90, 2005: 627-634.
- Parker, Alan., Paul. Gunning, Kim. Ng, and Margaret Robins. "How does xanthan stabilize salad dressing?" *Food Hydrocolloids* 9, 1995: 333-342.
- Pellicer, J., J. Delegido, J. Dolz, M. Dolz, M.J. Hernandez, and M. Herraez. "Influence of shear rate and concentration ratio on viscous synergism. Application to xanthan-locust bean gum-NaCMC mixtures." *Food Science Technology International* 6(5), 2000: 415-423.
- Pettitt, David, Jo Ellen Wayne, Jody Nantz, and Charles Shoemaker. "Rheological Properties of Solutions and Emulsions Stabilized with Xanthan Gum and Propylene Glycol Alginate." *Journal of Food Science - Vol. 60, No. 3*, 1995: 528-531.
- Riscardo, M.A., J.M. Franco, and C. Gallegos. "Influence of Composition of Emulsifier Blends on the Rheological Properties of Salad Dressing-type Emulsions." *Food Science and Technology International 9(1)*, 2003: 53-63.
- Stechemesser, Hansjoachim, and Bohuslav Dobias. *Coagulation and Flocculation, Second Ed.* Boca Raton, FL: CRC Press, 2005.
- Sun, Wenbin, Dejun Sun, Yunping Wei, Shangyin Liu, and Shuiyan Zhang. "Oil-in-water emulsions stabilized by hydrophobically modified hydroxyethyl cellulose: Adsorption and thickening effect." *Journal of Colloid and Interface Science 311*, 2007: 228-236.
- Van Aken, George, Monique Vingerhoeds, and Rene de Wijk. "Textural perception of liquid emulsions: Role of oil content, oil viscosity and emulsion viscosity." *Food Hydrocolloids* 25, 2011: 789-796.
- Weiss, Jochen. "Emulsion Processing Homogenization." *Emulsion Workshop*. Amherst, MA: University of Massachusetts, 2008.
- Yilmazer, Gulcin, Ana Carrillo, and Jozef Kokini. "Effect of Propylene Glycol Alginate and Xanthan Gum on Stability of O/W Emulsions." *Journal of Food Science - Vol. 56, No. 2*, 1991: 513-517.