# The Influence of Salt Reduction and Replacement on Cottage Cheese Cream Dressing

Rheology, Tribology, and Sensory Behavior

A Thesis Presented in Partial Fulfillment of the Requirements for the Degree of Master of Science with a Major in Food Science in the College of Graduate Studies University of Idaho by Hannah L. Damiano

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## AUTHORIZATION TO SUBMIT THESIS

This thesis of Hannah Damiano, submitted for the degree of Master of Science with a Major in Food Science and titled "The Influence of Salt Reduction and Replacement on Cottage Cheese Cream Dressing Rheology, Tribology, and Sensory Behavior," has been reviewed in final form. Permission, as indicated by the signatures and dates below, is now granted to submit final copies to the College of Graduate Studies for approval.

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#### ABSTRACT

Understanding how salt reduction and replacement affects cottage cheese cream dressing properties is critical for producing such a dressing with acceptable sensory attributes. The objective of this work was to determine how salt reduction and replacement impacted rheological, tribological, and sensory characteristics of cottage cheese cream dressing. All samples were pseudoplastic and exhibited weak gel viscoelastic behavior. The magnitudes of viscosities and viscoelastic moduli varied, although differences were not always significant. pH and specific cation had the greatest impact on behavior. Near the isoelectric point of casein, viscosities were higher with more viscoelastic solid behavior. Consumers found reduced sodium and KCl-substituted formulations to be acceptable as compared to a full salt dressing. CaCl<sub>2</sub>-substituted formulations were not as acceptable. These results indicate that creation of reduced salt cottage cheese dressing is feasible, although more study on the impact on microbiological growth and dressing structure–function–texture relationships matrix is recommended.

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### **DEDICATION**

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# TABLE OF CONTENTS

Authorization to Submit Thesisii
Abstractiii
Acknowledgementsiv
Dedicationv
Table of Contents vi
List of Figures
List of Tables x
Chapter 1: Introduction
References
Chapter 2: Literature Review7
Production of Creamed Cottage Cheese7
Major Components of Dairy Systems
Stabilization of Dairy Products
Sensory Attributes of Cottage Cheese
Mechanical Behaviors of Dairy Systems
Tribology of Dairy Systems
Conclusions
References
Chapter 3: The Impact of Sodium Reduction on Cottage Cheese Cream Dressing Rheology,
Tribology, and Sensory Behaviors
Introduction
Materials and Methods
Results and Discussion
Conclusions
References
Chapter 4: The Impact of Sodium Replacement on Cottage Cheese Cream Dressing
Rheology, Tribology, and Sensory Behavior
Introduction76
Materials and Methods

Results and Discussion	
Conclusions	
References	106
Chapter 5: Conclusions	
Appendix A: Protocol Approval from Institutional Review Board	
Appendix B: Cottage Cheese Cream Dressing Sensory Ballot	119
Appendix C: Sensory Panel Demographics	125

# LIST OF FIGURES

Figure 2.1. General process flow diagram for cottage cheese curd manufacture. Cooking time
and temperature will vary based on method used
<i>Figure 2.2.</i> Cross-section of a proposed model of a casein micelle
Figure 2.3. Example of a potential TPA readout. Different sensory characteristics may be
calculated using a variety of calculations utilizing the different areas shown in the readout32
<i>Figure 2.4.</i> Tribology setup
<i>Figure 2.5.</i> Stribeck curve with lubrication regions delineated by vertical blue lines
Figure 3.1 Viscosity profiles of reduced sodium dressing formulations generated from shear
rate sweep data67
Figure 3.2 Viscoelastic behavior of dressing formulations generated from strain sweep
data
Figure 3.3 Frequency dependent behavior of dressing formulations generated from frequency
sweep data
<i>Figure 3.4</i> Friction curve of 2.2% and 0.73% NaCl cottage cheese dressings70
Figure 3.5 Just about right frequency of response data for salt, acidity, and dressing consistency
<i>Figure 4.1.</i> An ideal Stribeck curve, such as one that might be generated from testing an ideal,
Newtonian fluid
<i>Figure 4.2</i> . Viscosity profiles of sodium substituted cottage cheese cream dressing at 8°C98
<i>Figure 4.3.</i> Viscosity profiles of sodium substituted cottage cheese cream dressing at 25°C99
Figure 4.4. Viscoelastic behavior from strain sweeps of sodium substituted cottage cheese
cream dressing at 8°C100

Figure 4.5. Viscoelastic behavior from strain sweeps of sodium substituted cottage cheese
cream dressing at 25°C101
<i>Figure 4.6.</i> Viscoelastic behavior from frequency sweeps of sodium substituted cottage cheese cream dressing at 8°C
Figure 4.7. Viscoelastic behavior from frequency sweeps of sodium substituted cottage
cheese cream dressing at 25°C103
Figure 4.8. JAR data for salt, acidity, and dressing consistency104
Figure 4.9. Just about right frequency of response data for salt, acidity, and dressing
consistency105

# LIST OF TABLES

Table 2.1. Comparison of starter culture amount, hold time, and hold temperature differences
between different cottage cheese curd making methods
Table 2.2. Summary of the different proteins that make up cow's milk
Table 2.3. Summary of common viscosity profiles    27
Table 3.1. Dressing formulations
Table 3.2. Proximate analysis and pH values for dressing formulations
<i>Table 3.3.</i> Flow model fit and variable values for averaged shear rate sweep data
Table 3.4. Average 9-point hedonic liking scores of overall liking, appearance, texture, flavor,and bitterness of full- and reduced-salt dressing formulations
Table 3.5. Penalty analysis of saltiness, acidity, and consistency of full- and reduced-salt         dressings
Table 4.1.    Formulations for NaCl substituted cottage cheese dressings using KCl and      CaCl2
Table 4.2. Composition of reduced sodium dressing formulations
Table 4.3. Flow model fit and variable values for 1:3 NaCl:KCl/CaCl <sub>2</sub> substituted dressing formulations
Table 4.4. Flow model fit and variable values for 1:1 NaCl:KCl/CaCl <sub>2</sub> substituted dressing
formulations
Table 4.5. Flow model fit and variable values for 3:1 NaCl:KCl/CaCl2 substituted dressing
formulations
Table 4.6. Least squares means for sodium substituted cottage cheese dressing formulations,
with a 2.2% (w/w) NaCl sample acting as the control94
Table 4.7. Mean drops in overall liking scores for samples panelists considered not just-about-
right9

#### **CHAPTER 1: INTRODUCTION**

Cottage cheese is a fresh, acid-coagulated cheese of American origin. Because the curd is made with skim milk and without flavor enhancers such as salt, a dressing composed of cream, milk, salt, and hydrocolloids is commonly added to the curd to provide body and flavor; additional flavor may also be induced by culturing the dressing. Despite being naturally high in protein (approximately 25 g in a 225 g serving), cottage cheese consumption has decreased over the past several decades. Concurrent to this decline, yogurt consumption has increased steadily. There are a number of reasons this formerly popular dairy product has fallen out of favor. Increased marketing for yogurt has likely contributed, although production and sensory issues related to cottage cheese are also contributing factors.

Cottage cheese is not an easy product to manufacture. The final curd is delicate, and manufacturing a high-quality product is more difficult for cottage cheese than other cheese products. Difficulties in production may be one reason that cottage cheese has fallen out of favor. In addition, there are certain flavor and textural characteristics of cottage cheese, typically caused by improper formulation or processing conditions, that consumers find unappealing. Curd that is too firm/rubbery or mushy, combined with dressing that can become slimy if overstabilized, causes unappealing textural sensations. Additionally, many cottage cheese manufacturers add whey to the dressing to increase protein content and provide bulk in reduced-rate formulations; the whey can impart an unpleasant cardboard off-flavor. Whey may also separate from the dressing matrix during storage in a manner similar to that seen in yogurt. While this is not detrimental from a food safety perspective, it is a defect when viewed from a consumer food quality angle.

Despite difficulties in cottage cheese manufacture and some negative sensory characteristics, cottage cheese is still a relatively popular product for health-conscious consumers because it is naturally high in protein and lower in fat than many conventional dairy products. However, it also tends to be high in salt. There has been a consumer-driven push for lower-sodium products in recent years (Kim and others 2012), despite conflicting scientific evidence on the long-term benefits of a reduced sodium diet (Paterna and others 2008; Graudal and others 2011; Krikken and others 2009). To meet consumer demands, successful development of a low-salt, and thus reduced sodium cottage cheese product has the potential to re-energize widespread interest in cottage cheese.

There are a number of difficulties that may arise when attempting a reduced salt cottage cheese formulation. Two avenues exist to reduce salt in cottage cheese: the amount of sodium chloride can be incrementally reduced or sodium chloride can be partially replaced with a sodium substitute such as potassium or calcium chloride. There are several potential issues resulting from this reduction. Salt serves not only as a flavor agent, but it also acts as an antimicrobial. As a result, reducing the amount of salt in a food product could shorten its shelf-life. Salt substitutes also present some difficulties from a sensory perspective. Sensory studies show that other salts can impart a bitter taste to foods when used in high concentrations.

Three separate but related methodologies were used to assess differences among reduced salt cottage cheese cream dressing formulations in this study:

- 1. Rheology the study of flow behavior
- 2. Tribology the study of friction behavior
- 3. Sensory studying consumer acceptance of dressing attributes

Each selected methodology contributes beneficial information for product formulation and behavior. Rheology can provide valuable data regarding viscosity and viscoelastic food characteristics and how these change with material deformation. Tribology can be used to understand food friction and lubrication behaviors; specifically, tribology can estimate how foods interact with the soft palate of the mouth. Finally, sensory studies offer practical and easy-to-understand information on consumer perceptions of cottage cheese, and how product liking changes with formulation. Additionally, other studies have had success correlating texture descriptors such as astringency, creaminess, and thickness of food products with rheological and tribological data (Meyer and others 2011b; Joyner (Melito) and others 2014; Folkenberg and others 2006).

The overall goal of this project was to determine how sodium reduction and replacement impacted the rheological, tribological, and sensory characteristics of cottage cheese cream dressing. This goal was achieved through three objectives:

- Determine the impact of salt concentration and type on cottage cheese cream dressing rheological and tribological behaviors.
- Evaluate sensory attributes and consumer acceptability of selected reduced salt cottage cheese cream dressings.
- 3. Determine the relationships among sensory, rheological, and tribological behaviors of cottage cheese cream dressings.

This research helps fill a current gap in published scientific research. Studies on the rheology and sensory attributes of cottage cheese curd have been conducted, but little data exists on the cream dressing added to the curds. The data provided from this study are not

only pertinent to the study of cottage cheese dressing. Because the dressing used in this study is a relatively simple dairy system, this study provides valuable information on how salt and common hydrocolloids interact with dairy proteins. From a practical standpoint, this information can help cottage cheese manufacturers develop a high-quality, reduced salt cottage cheese product.

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#### **CHAPTER 2: LITERATURE REVIEW**

Creamed cottage cheese is a fresh, acid-coagulated cheese of American origin, to which a cream dressing can be added. A number of hydrocolloids and stabilizers are added to this dressing to improve its stability and mouthfeel over time; hereafter, these attributes will be referred to as the quality of the dressing. The type and amount of stabilizer used, as well as dressing pH, processing and storage temperature, and salt concentration and type all impact the rheological, tribological, and sensory characteristics of the final product.

### PRODUCTION OF CREAMED COTTAGE CHEESE

Cottage cheese is categorized as a fresh, acid curd cheese (Park and Haenlein 2013). Although produced from skim milk, cottage cheese nonetheless possesses a creamy texture (Walstra and others 2006). Cottage cheese is a soft, uncured cheese with at least 4% milkfat by weight and dry curd that is no more than 80% moisture (Code of Federal Regulations, 2013); the creaming mixture, henceforth referred to as dressing, added to the fresh, dry curd provides the necessary milkfat to keep the final product in compliance with federal regulations.

Cottage cheese may be produced using one of three methods: short-set, long-set, or directset, with differing time requirements being the determining factor for manufacturers. In the short set method, starter cultures (5-6% w/w), rennet, and milk are held for 4 to 5 hours at 32°C. In the long-set method, 1% w/w starter cultures are added to the milk and held for 12 to 16 hours at 22°C (Walstra and others 2006). In the direct-set method, the milk is acidified with phosphoric, lactic, citric, or hydrochloric acid; agitated until the pH reaches 4.6, then allowed to set for approximately 12 minutes (Walstra and others 2006). A comparison of the three set methods is outlined in Table 2.1. Following the clotting step, the manufacturing steps to reach the finished product do not differ regardless of set method. The entire process flow diagram is outlined in Figure 2.1.

*Table 2.1. Comparison of starter culture amount, hold time, and hold temperature differences between different cottage cheese curd making methods.* 

	Starter Culture (w/w%)	Hold Time (hr)	Hold Temperature (°C)
Short	5-6	4-5	32
Long	1	12-16	22
Direct	None – acid addition to pH 4.6	0.2	_

In an unaged product such as cottage cheese, the quality of the final curd is particularly important. The point at which gelation occurs during the cheesemaking process has a significant influence on curd quality, as it largely impacts the texture of the curd. If gelation occurs at a pH below 4.6, the resulting curd expels more whey, leading to a cheese that is acidic, crumbly, and dry (Scott and others 1998). Increased whey expulsion occurs above pH 4.9, resulting in a curd that is firm and tough (Walstra and others 2006; Gunasekaran and Ak 2002). Therefore, the pH of the curd is generally between 4.6 and 4.8 at the time of cutting (Walstra and others 2006).

Heating rate during cooking also has a significant impact on the final product. A slower rate of heating during cooking results in more even syneresis and a more uniform curd shape and firmness. This gentler cooking process heats the product evenly and gradually, reducing the likelihood of large temperature gradients in the product. Gently and continuously stirring the curd/whey mixture also helps keep the grains from knitting together (Walstra and others 2006). Because cottage cheese is sold as individual curds suspended in dressing, preserving the integrity of the individual curds is important to product quality. Ideally, cottage cheese

curds are uniform in size and shape, with a "meaty" consistency that is neither rubbery nor tough (Clark and others 2009).

A cream dressing is typically added to the cheese curds to enhance the flavor and texture of the final product. For a full fat product, the dressing has a fat content of 10-20%, depending on the desired fat content of the final product and the dressing to curd ratio. A variety of stabilizers and thickening agents (hydrocolloids) are added to the cream dressing to create the desired texture and prevent phase separation, which manifests as a layer of clear, yellow liquid on top of the dressing (Walstra and others 2006). This phenomenon is also known as "wheying off." The stabilizers interact with the major components of dairy systems (proteins, fats, and carbohydrates) in a variety of ways; these interactions impact dairy component function and stability in the food matrix.

#### MAJOR COMPONENTS OF DAIRY SYSTEMS

The functionality of the major components of dairy systems is greatly impacted by product composition, physicochemical properties, processing conditions, and storage temperature. A basic knowledge of dairy chemistry is critical to understand how changing product and processing conditions impact the functionality of dairy systems.

## Proteins

Protein is an essential macronutrient, required for growth and tissue maintenance (WHO 2007). Proteins and peptides in milk are particularly important, as they provide much of the necessary nutrition to infants, as well as a number of non-nutritive benefits (Fox and Flynn 1994). These milk proteins are divided into two major categories: casein and whey (serum) (Park and Haenlein 2013). The overall protein content of commercial milk averages between

3.4 and 3.5% (Park and Haenlein 2013); however, protein content in bovine milk ranges from 2.3 to 4.4% based on the breed of cow, time of year, and diet.

_	Protein	% of total protein	Molecular weight (Da)	pI	Amount in skim milk (g/L)
Casein	$\alpha_{s1}$	0.32	22068-23742	4.2-4.76	12-15
	$\alpha_{s2}$	0.09	25230	-	3-4
	β	0.28	23944-24092	4.6-5.1	9-11
	κ	0.12	19007-19039	5.45-5.77	2-4
Whey	α-lactalbumin	0.11	14147-14175	4.2-4.5	0.6-1.7
	β-lactoglobulin	0.05	18205-18363	5.13	2-4

Table 2.2. Summary of the different proteins that make up cow's milk\*

\* Data obtained from Eigel and others (1984)

#### Caseins

Caseins account for the majority of milk protein, approximately 80%. They are a hydrophobic group of proteins that are difficult to denature due to their relative lack of secondary or tertiary structure (Walstra and others 2006). Casein proteins are nutritionally significant due to their high phosphate content, which allows them to bind calcium, and also due to their high lysine content. Four types of casein proteins have been identified:  $\alpha_{s1}$ ,  $\alpha_{s2}$ ,  $\beta$ , and  $\kappa$ .

There are two subclasses of  $\alpha$ -caseins.  $\alpha_{s1}$ -caseins account for 40% of the caseins present in bovine milk (Park and Haenlein 2013) and are highly negatively charged.  $\alpha_{s2}$  caseins account for only 10% of the total casein present in bovine milk. Both  $\alpha_{s1}$  and  $\alpha_{s2}$  caseins serve as molecular chaperones, which play a significant role in protein aggregation (Treweek 2012). Specifically, molecular chaperones suppress protein aggregation during folding and unfolding of the protein, thus influencing protein folding kinetics. The role of  $\alpha_s$ -caseins as molecular chaperones makes them highly beneficial in the food industry. They have proven successful in the prevention of aggregation of heat denatured proteins (Treweek 2012) and are important to the stabilization of dairy product systems.

β-casein is the most hydrophobic type of casein, containing a large number of proline residues (Walstra and others 2006). Despite its hydrophobicity, the charge along the molecule is unevenly distributed. The first 44 residues from the N terminus contain far fewer hydrophobic proline residues, occurring at a frequency of 0.02, as opposed to 0.20 for the remainder of the molecule. The lower frequency of hydrophobic amino acid residues gives βcasein a hydrophilic head and hydrophobic tail (Walstra and others 2006). β-casein increases the viscosity of fluid milk due to its solubility at low temperatures and is a particularly efficient emulsifier in dairy systems due to its relatively small mass, charge distribution, and minimal secondary and tertiary structure, which gives β-casein an open structure that can interact with the food matrix (Walstra and others 2006; Fox and McSweeney 1998)

 $\kappa$ -casein differs significantly from α- and β-casein. Along with α<sub>s2</sub>-casein,  $\kappa$ -casein is the only type of casein to form intermolecular disulfide bonds, due to the presence of two cysteine residues (Walstra and others 2006). Similar to β-casein,  $\kappa$ -casein contains both hydrophobic and hydrophilic regions. Approximately 2/3 of the  $\kappa$ -casein molecules have an esterified carbohydrate group attached somewhere along the molecule, with the tri- or tetrasaccharides accounting for approximately 5% of total  $\kappa$ -casein composition (Fox and McSweeney 1998; Walstra and others 2006). These carbohydrate groups have hydrophilic charges (Walstra and others 2006). These carbohydrate groups have hydrophilic charges (Walstra and others 2006). The presence of hydrophobic and hydrophilic regions on the  $\kappa$ -casein molecule allow it to stabilize casein micelles.

Caseins form micelles in milk that are roughly spherical, with diameters ranging from 150 to 300  $\mu$ m (Muller-Buschbaum and others 2007). Although the exact structure of the

casein micelle is still unknown, Figure 2.2 shows a cross-section of a proposed model. The smaller black circles indicate the hydrophobic core of the casein submicelle. The gray "hairy" layer on the outside of the micelle consists of the hydrophilic C-terminal end of  $\kappa$ -casein. This hairy layer stabilizes the micelle via electrostatic (Walstra and others 2006) and steric stabilization. If the outer layer comes in contact with other polymeric molecules, the free energy increases, marking the stabilization as mainly entropic (de Kruif 1999; Horne 2006). The small black circles indicate nanoparticles composed primarily of calcium phosphate. The nanoparticles also include protein moieties such as organic phosphate and glutaminic acid residues (Walstra and others 2006). Hydrophobic bonds between protein groups and peptide chain crosslinks (not shown in the figure) help maintain the structure of the micelle (Walstra and others 2006).

The presence of these micelles is significant, as they impact the stability of milk. Unfolded proteins have the potential to form toxic amyloid fibers, but the formation of casein micelles in fluid, unhomogenized milk prevents this aggregation (Holt and others 2013). Other functions of casein micelles include trapping calcium, increasing the calcium content in milk beyond what would be soluble in the serum phase, and emulsification of fat. In terms of food processing, the rheological properties of sour cream and other concentrated milk products is largely determined by casein micelles (Walstra and others 2006).

## Serum (Whey) Proteins

Whey proteins account for the remaining 20% of dairy proteins. Because whey is drained from the cottage cheese curd, whey (serum) proteins are of less significance in the curd. However, because wheying off is of concern in cottage cheese cream dressing, an understanding of the primary whey proteins and how they interact with the food system is pertinent to understanding the stability of creamed cottage cheese.

While caseins have little secondary or tertiary structure,  $\alpha$ -lactalbumin and  $\beta$ lactoglobulin exhibit both  $\alpha$ -helix and  $\beta$ -sheet secondary structures and have a generally globular tertiary structure (Walstra and others 2006). They are also highly hydrophobic (Walstra and others 2006).

Whey protein gelation is a complex process that is dependent on multiple factors. The currently accepted model for whey protein gelation is a four-step process (Singh and Havea 2003; Lorenzen and Schrader 2006):

- 1. Protein unfolding
- 2. Unfolded protein aggregation
- 3. String formation of the aggregates
- 4. String linkage to form a three-dimensional network

Whey protein gels at approximately 60°C, with whey protein isolate exhibiting stronger gelation when prepared at temperatures above 80°C. This is due to whey protein denaturation, resulting in a greater number of protein-protein interactions (Lorenzen and Schrader 2006).

α-lactalbumin acts as a coenzyme in the production of lactose. It self-associates only at low ionic strength (Walstra and others 2006). Calcium can bind strongly to α-lactalbumin and help stabilize the tertiary structure of the protein (Walstra and others 2006). β-lactoglobulin characteristics typically dominate whey properties, since it is present in higher concentrations than α-lactalbumin. At high temperatures, the protein dissociates from its normal dimer state. β-lactoglobulin self-associates between pH 3.5 and 5.5 (Walstra and others 2006). While  $\alpha$ -lactalbumin and  $\beta$ -lactoglobulin are the primary whey (serum) proteins, at 1.2 g/L of milk and 3.2 g/L of milk, respectively (de Wit 1998), several other proteins are also present in small quantities, but do not play a significant role in the production of dairy products. These other proteins include serum albumin, immunoglobulins, proteose peptone, and lactoferrin (Walstra and others 2006).

#### Milk Fat

Lipids in milk are important for a variety of reasons. They act as an energy source, serve as a carrier for fat-soluble vitamins, and keep milk flavor compounds in solution. (Park and Haenlein 2013). Triacylglycerols account for over 98% of the lipids present in bovine milk (Park and Haenlein 2013). These triacylglycerols contain an average of 14.4 carbon atoms and 0.35 double bonds (Walstra and others 2006). Because they are the predominant component in milk fat, triacylglycerols largely determine the characteristics of milk fat. Other fats present in milk include phospholipids and conjugated linoleic acids.

Fluid milk is usually homogenized prior to further processing or the production of dairy products. The primary purpose of homogenization is to reduce the size of the fat globules. Fat globule size varies depending on homogenization conditions, with higher homogenization pressures producing smaller fat globules. Unhomogenized milk has fat globules ranging in size from 3.4 to 4.5  $\mu$ m depending on fat content and bovine breed. Once homogenized, the average size of fat globules decreases to 0.6  $\mu$ m (Walstra and others 2006). When milk is homogenized, the fat globules are reduced in size and an emulsion is formed in which the milk proteins, mostly casein, cover a large portion of the fat globules. This emulsification helps to decrease creaming, or the aggregation of fat molecules (Walstra and others 2006).

#### Carbohydrates

Lactose is the major carbohydrate in milk. Lactose is a disaccharide comprising glucose and galactose monomers. The monomers are linked by a  $\beta$ -1,4-glycosidic bond connecting the aldehyde group of galactose to the C4 group of glucose (Walstra and others 2006). Lactose is susceptible to a number of chemical reactions, including Maillard browning, oxidation, reduction, and hydrolysis. These reactions can lead to a number of textural and flavor changes in the milk. Maillard browning can result in development of a cooked or even bitter flavor in the milk. While a mildly cooked flavor is not necessarily detrimental to consumer acceptance, more severe heating can result in aromatics reminiscent of sulfide components, creating undesirable flavors (Clark and others 2009). However, the oxidation or reduction of lactose is generally mild, and hydrolysis of lactose by acid does not easily occur (Walstra and others 2006).

While there are other sugars in milk, including glucose and galactose, bovine milk contains no polysaccharides (Walstra and others 2006).

#### Sodium

In considering sodium reduction in dairy products, it is important to look not only at added salt but the inherent sodium content in milk. Analysis of milk by absorption spectrophotometry has shown the sodium content of milk to be approximately 0.01 g/L (Murthy and Rhea 1967), which is comparable to the information given on the nutrition facts panel on commercially available milk. Analysis of the nutrition labels of heavy whipping cream indicates a higher concentration of sodium, with approximately 0.4 g/L. The higher concentration of sodium in cream is likely due to the reduced amount of water in cream, resulting in a higher concentration of sodium and other minerals.

The sodium in milk provides an important mineral resource to feeding calves, although elevated sodium content in milk can be an indicator of mastitis in cows (El Zubeir and others 2005).

### STABILIZATION OF DAIRY PRODUCTS

#### Factors Contributing to Stability of Dairy Systems

### pН

The pH of dairy systems has the most significant impact on the proteins present in the food system. O'Connell and Fox found that heat stability of milk increased with increasing pH from 6.4 to 7.1 (O'Connell and Fox 2001). Below a certain pH, the quaternary, tertiary, and secondary structure of a protein can be disrupted, and these conformational changes affect the functionality of the protein in the system. At the same time, acidic conditions can be beneficial in food systems by limiting the growth of pathogenic or spoilage bacteria, which can increase the safety, quality and shelf life of the product. Hence, it is important to optimize the pH of food products to maximize protein functionality and inhibit unwanted microbial growth.

 $\beta$ -lactoglobulin has been shown to markedly influence the heat stability of milk at various pH (Elfagm and Wheelock 1978; Sawyer 1969; Tessier and Rose 1964). A widely accepted theory to explain the impact of β-lactoglobulin milk coagulation postulates that at pH of approximately 6.7, β-lactoglobulin reduces the dissociation of κ-casein that occurs at higher temperatures, while at pH 6.9, β-lactoglobulin enhances this dissociation, making the casein micelles more susceptible to precipitation (O'Connell and Fox 2001).

Cottage cheese curds can be produced using acids to reduce the pH to 4.6, inducing curd formation by precipitation of caseins. However, it has been shown that gelation of micellar

casein can occur across a wide range of pH, from 4.8 to greater than 6.0, and that this variation in gelation occurs as a result of varying salt concentration. Auty and others (2005) found that gelation during acidification occurred more rapidly at low salt concentration, while gel formation was slower at higher salt concentrations, with the resulting network also being more homogenous.

#### **Sodium Chloride and Salt Substitutes**

Sodium chloride enhances the flavor and stability of dairy products and limits unwanted microbial growth. However, with growing concerns on the contribution of sodium to hypertension and other chronic illnesses (CDC 2015), manufacturers seek to reduce or replace sodium chloride in foods. Unfortunately, this reduction in sodium chloride impacts both the sensory attributes of the final product and microbial inhibition.

Efforts to reduce sodium chloride concentration in dairy products has been studied more in aged cheeses such as Cheddar, as the sodium reduction can lead to bitter peptide formation during the aging process. Some information exists on sodium reduction in cottage cheese and other fresh cheeses as well. Fresh feta cheese produced using 1% and 2% sodium chloride was not significantly different in terms of body, texture, or taste in comparison to a control containing 4% sodium chloride (Aly 1995). In the same study, a blend of up to 1% KCl and 1% NaCl in feta cheese formulation was found to have acceptable sensory attributes, but the flavor, body, and texture were significantly different from the control, with cheese made with more KCl perceived as less salty and more bitter (Aly 1995). Similar results were achieved in Kefalogravaiera, a Greek cheese produced with ewes' milk and a 1:1 substitution of KCl for NaCl. Panelists in a sensory study found this ratio to be acceptable, although as aging of the cheese progressed, a metallic off-flavor was noted (Katsiari and others 1998).

#### Temperature

As with pH, temperature is of particular concern in dairy products because of the risk of microbial contamination. Spoilage microorganisms and pathogenic bacteria can grow in the temperature range of 4.4 to 60°C; for major foodborne pathogens including *Campylobacter jejuni, Listeria monocytogenes, Salmonella spp.*, and *Staphylococcus aureus*. The minimum temperature required for growth ranges widely, but are all inactivated at temperatures exceeding 55°C (FDA 2011). However, the starter cultures necessary for the production of cultured dairy products such as cottage cheese experience optimal growth in the same temperature range as pathogenic organisms. As a result, milk is typically pasteurized prior to addition of starter cultures to destroy unwanted microorganisms and provide optimal growth conditions for the starter culture.

The primary spoilage microorganisms in milk and other dairy products are psychrotrophic, with certain species of *Pseudomonas* bacteria being of primary concern. In particular, *Pseudomonas fluorescens* is known to contribute greatly to milk spoilage (Clark and others 2009; Sorhaug and Stepaniak 1997). Proper pasteurization is sufficient to destroy these microorganisms; however, mishandling of the product can lead to contamination following pasteurization (Clark and others 2009; Sorhaug and Stepaniak 1997). *Pseudomonas* species are known to be lipolytic, which can cause off-flavors in food products. They can also hydrolyze casein into soluble peptides, which have a bitter taste. Hence, the presence of psychrotrophs in cottage cheese is significantly correlated to a bitter taste in the final product (Sorhaug and Stepaniak 1997).

Although the milk used to produce cottage cheese is pasteurized, a severe heat treatment is not used because this would interfere with the syneresis of whey from the curds necessary to produce the final product (Lucey 2004).

#### **Stabilizing Agents**

Emulsifiers and other stabilizing agents have been used for over 50 years in the dairy industry (Lal and others 2006). Their usage is becoming increasingly critical for enhancing the appearance, texture, flavor, and shelf life of dairy products (Lal and others 2006). Hydrocolloids, a term which refers to polysaccharides and proteins that may be derived from botanical, algal, microbial, or animal sources, are widely used in the food industry in a variety of applications, including thickening, stabilization, and inhibition of ice crystal formation (Phillips and Williams 2009).

## Xanthan Gum

Xanthan gum is produced commercially by a bacterial fermentation process; it is a polysaccharide excreted by *Xanthomonas campestris* (Phillips and Williams 2009). Xanthan gum consists of glucose, mannose, and glucuronic acid in a 2:2:1 ratio. Interactions between milk proteins and xanthan gum have been shown to exhibit pseudoplastic behavior that is typical of xanthan gum solutions (Hemar and others 2001). Milk protein/xanthan gum solutions with 1% xanthan gum and 0.2 to 2% protein exhibited rheological behavior similar to xanthan gum alone at the same concentration, regardless of shear rate. As xanthan concentration increased, aggregate size decreased (Hemar and others 2001).

The efficacy of xanthan gum is also influenced by solution pH. Xanthan gum was found to be effective at reducing syneresis at pH 7 and pH 9 but was less effective in high acid conditions (pH 3); (Phillips and Williams 2009; Sae-kang and Suphantharika 2006). The synergy between xanthan gum and plant galactomannans is exhibited through the formation of thermoreversible gels and is due to interactions among the different polymer chains, which form mixed junction zones (Copetti and others 1997; Cuvelier and Launay 1986). Researchers have also proposed a variety of mechanisms for this synergistic behavior, notably a lock and key model in which the side chains on the xanthan gum molecule are inserted into backbone segments of the galactomannan-containing molecule (Tako and Nakamura 1985). When mixed with locust bean gum, xanthan gum forms a weak gel (Lundin and Hermansson 1995; Mannion and others 1992; Zhan and others 1993; Sanchez and others 2000). Mixtures of xanthan and guar gums yield solutions of increased viscosity (Khouryieh 2006; Tipvarakarnkoon and Senge 2008; Wang 2002). This lack of gelation in comparison to xanthan-locust bean gum mixtures can be explained by side chains on guar gum inhibiting interactions with xanthan gum side chains, preventing formation of a network (Tako and Nakamura 1985).

## Guar Gum

Guar gum is derived from the plant *Cyamposis tetragonoloba* (Phillips and Williams 2009). A galactomannan with the backbone units connected by 1-4 linkages, guar gum has the highest viscosity of any naturally produced commercial gum, due in part to its long, relatively rigid mannopyranosyl backbone. There are  $\alpha$ -D-galactopyranosyl branches on O6; this branching can be seen on the upper left portion of the guar gum molecule shown in Figure 5. Approximately half of the backbone units exhibit branching (Fennema 1996), with the other half containing a hydroxyl group on O6. Despite the high viscosity of guar gum solutions, the even spacing of the branching side chains inhibits the gum's ability to form junction zones (Fennema 1996), preventing gelation.

There are many applications of guar gum in the dairy industry. Guar gum is soluble in cold water and is used as a thickening agent to increase product viscosity without negatively impacting palatability (Lal and others 2006). Because its viscosity in solution is not affected by pH (Lal and others 2006), guar gum is often used in acidified milk products (Lal and others 2006) such as yogurt and cottage cheese dressing. Furthermore, addition of guar gum (0.2 and 0.4% w/w) to a 25% w/w sucrose-lactose solution led to a marked decrease in ice crystal propagation (Wang and others 1998).

Partially hydrolyzed guar gum has shown promise in reducing syneresis of low-fat yogurt, as well as increasing its viscosity to levels above that of full-fat controls (Brennan and Tudorica 2008). Researchers studying the effect of guar gum on flavor compounds in acidified milk products found that its addition did not significantly change the distribution of acetaldehyde, ethanol, or diacetyl (Lal and others 2006). These results further indicate that guar gum can provide stabilization in acidic milk products without disrupting other important components. Guar gum also acts synergistically with xanthan gum. In combination, the thickening ability of these two hydrocolloids significantly increases (Phillips and Williams 2009).

### Locust Bean Gum

Also known as carob bean gum, locust bean gum is derived from the seed of the locust bean tree, *Ceratonia siliqua*. Similar to guar gum, locust bean gum is a galactomannan with a molecular weight of 300,000 to 360,000 Daltons (Casas and Garcia-Ochoa 1999). However, the degree of branching in locust bean gum is approximately half that of guar gum. The lesser extent of branching allows for closer polymer associations between the hydrocolloid molecules. Locust bean gum is not charged, so changes in solution pH do not have a

significant change on its properties (Kok 2010). Unlike guar gum, locust bean gum is only slightly soluble in cold water and must be heated in solution to approximately 80°C to achieve full dispersion (Casas and Garcia-Ochoa 1999). Locust bean gum exhibits synergistic behavior with many other hydrocolloids, particularly xanthan gum.

Increasing concentrations of locust bean gum in a yogurt beverage has been found to lead to changes in its viscoelastic behavior, mainly by causing viscous properties to more strongly dominate (Kok 2010). As locust bean gum concentration increased from 0 to 0.1% in whipped cream, whipping time increased. It was suggested this increase in whipping time may have been due to hydrocolloids kinetically hindering cream foaming by increasing viscosity of the liquid cream (Camacho and others 1998).

#### Other Stabilizers Used in Dairy Systems

The hydrocolloids discussed previously are all commonly used in dairy products but are not a comprehensive list of the stabilizers used in dairy systems. Other stabilizers commonly used in commercial cottage cheese formulations include carrageenan and food starches (Joyner (Melito) and Damiano 2015).

Carrageenan is a widely-used stabilizer in dairy products; however, companies recently have been making efforts to reduce its usage in response to negative consumer perceptions of the ingredient (Watson 2009; Kirsch 2002). Carrageenans are plant polysaccharides that may be derived from a variety of red seaweeds, most from the genus *Gigartina*. In dairy products, carrageenans provide texture benefits such as smoothness and body, and their stability to freeze-thaw cycles makes them particularly valuable in frozen dairy products such as ice cream (Tecante 2012). Two different forms of carrageenan, kappa and iota, impart different textural characteristics to dairy products. κ-carrageenan provides different textural properties

depending on the type of salt present. A strong, rigid gel forms upon addition of potassium salts, while brittle gels form in the presence of calcium salts (Imeson 2000; McHugh 2003). 1-carrageenan gives soft and elastic gels that are freeze/thaw stable when combined with calcium salts (McHugh 2003; Imeson 2000).

Starches are also commonly used in dairy products, particularly to serve as a texture modifier in reduced-fat products (Abbas and others 2010). Tapioca starch has been used as a fat replacer in yogurt and cheese (Castilla 2003; Sipahioglu 2000). Various starches are used in combination with other hydrocolloids in the dairy industry for texture modification. Addition of starch in combination with carrageenan can provide various textures depending on type and concentration used (de Vries 2002; Imeson 2000). Physical, enzymatic, or chemical modifications can improve the functional properties of native starches by increasing their water holding capacity and heat resistance, minimizing syneresis, and improving their thickening capabilities (Adzahan 2002; Miyazaki 2006).

## SENSORY ATTRIBUTES OF COTTAGE CHEESE

Texture of dairy products is a significant driver in overall consumer liking, but many studies on cottage cheese focus largely on sensory attributes as they relate to formulation, production procedure, and physiochemical properties. Formal sensory evaluations of creamed cottage cheese and other dairy products encompass visual, textural, and flavor assessments. A large number of sensory defects are possible in cottage cheese. For an in-depth discussion of these defects, as well as their possible causes, *The Sensory Evaluation of Dairy Products* (Clark and others 2009) is a good resource. A number of these defects pertain specifically to the cream dressing. For example, overstabilized creamed cottage cheese has an excess of stabilizers or emulsifiers intended to increase dressing viscosity. This manifests as curd that

is dry or surrounded by dressing that appears thick and pasty (Clark and others 2009). If this defect is severe, off-flavors and a slippery or coated mouthfeel can also occur. Flavor defects to which the cream dressing may contribute are varied and include bitter, high salt, rancid, and sweet. It is important to note that product attributes that are considered defects by official dairy judging criteria do not necessarily represent consumer preferences. Attributes such as high diacetyl or high salt are considered defects, yet consumers may prefer these characteristics in their cottage cheese product (Antinone and others 1994; Demott and others 1984).

Cheese consumption as an overall category has trended upward since 1975, but consumption of cottage cheese specifically has decreased (Davis and others 2010). Despite this decrease in consumption, little data on consumer preferences in cottage cheese exists to aid manufacturers in creating a cottage cheese product that consumers wish to purchase (Drake and others 2009).

One potential way to renew interest in cottage cheese is through a reduction of its sodium content. Cottage cheese has a larger serving size than most cheeses, thus raising concerns over the amount of sodium it can potentially contribute to the diet (Harvard 2015). Sodium chloride usage in cottage cheese can be reduced significantly without negatively impacting consumer liking in comparison to full salt products (Drake and others 2011; Wyatt 1983). Sodium content in cottage cheese can be further reduced by substitution with other salts, such as potassium chloride. Up to a 50% substitution of potassium or calcium chloride for sodium chloride in cottage cheese has been found to result in an acceptable product based on consumer sensory panels (Demott and others 1984; Shelf and Ryan 1988).

In a study comparing cheesecake produced using cottage cheese of different fat levels, Yasin and Shalaby (2013) found that panelists generally preferred a control cheesecake prepared with cream cheese to reduced-fat cheesecakes prepared with cottage cheese. However, cheesecakes made with full and reduced-fat cottage cheeses scored higher for texture and color, and significant differences were not observed between the cottage cheesecontaining cheesecakes. Thus, cottage cheese shows promise not only as a standalone product but also as a substitute for higher-fat dairy ingredients, which could prove valuable to manufacturers seeking to create a healthier product to appeal to a broader range of consumers.

Sensory studies are important indicators of consumer likes and dislikes, but these consumer data are largely qualitative. Studies on the mechanical behaviors of food systems in the fields of rheology and tribology offer quantitative data that can be related to texture data gathered from sensory panels.

## MECHANICAL BEHAVIORS OF DAIRY SYSTEMS

## Rheology of Dairy Systems

Rheology is the study of the deformation and flow of matter (Barnes and others 1989). Per this definition, rheology is particularly concerned with the stress–strain relationships in materials, and how these relationships change with the rate of applied stress or strain. Studying the rheology of food products can provide a valuable link between food structure and its impact on texture.

While the rheology and texture of cottage cheese curd has been studied (Castillo and others 2006a; Castillo and others 2006b), the rheology of cottage cheese cream dressing has received little attention in the literature. The cream dressing provides significant flavor and

texture to the finished product, thus playing a major role in consumer acceptance of cottage cheese. Additionally, different stabilizer blends and sodium concentrations may change the mechanical properties of the dressing. Thus, studying cottage cheese cream dressing rheological behaviors is beneficial.

Uniaxial compression is a popular test for determining the rheological properties of cheese (Gunasekaran and Ak 2002). In uniaxial testing, a constant force is applied to the sample and its resulting deformation is measured, or vice versa. One particular type of uniaxial testing, Texture Profile Analysis (TPA), uses a double compression to estimate parameters such as resilience and cohesiveness.

Figure 2.3 shows a sample TPA graph. TPA was developed by researchers at the General Foods Corporation in the early 1960s to simulate a chewing motion with two "bites" of a small, flat plunger (Gunasekaran and Ak 2002). TPA tests are set to have the plunger move up and down with a set speed and compress the sample to a pre-set percent strain; the strain chosen varies based on the material being tested. The set time between the first and second bites determines the degree to which samples will return to their original form. The TPA readout allows a number of parameters to be calculated; resilience is calculated by dividing Area 4 by Area 3 (Figure 2.3) and is defined by how well a product returns to its original height, while cohesiveness is calculated by dividing Area 2 by Area 1 (Figure 2.3) to measure how well a second deformation is withstood by the product. Young's modulus is another means of assessing product elasticity (Steffe 1996). While the information garnered from TPA analysis can be beneficial, it is important to note that these data are not intended to be used in place of trained sensory panels. Rather, TPA can provide supplemental data that may be used to provide a more complete, in-depth understanding of the food being studied.

Studying viscosity and viscoelastic properties is particularly important in the rheological study of foods. Viscosity is defined as a material's resistance to flow. There are many models used to describe viscosity profiles; Table 2.3 contains descriptions of some of the commonly used models for foods: Newtonian, Power Law, and Herschel-Bulkley (Steffe 1996).

Table 2.3. Summary of common viscosity profiles.

Fluid type	Relationship between shear stress and shear rate	Yield stress?
Newtonian	Linear	No
Power law	Exponential	No
Herschel-Bulkley	Exponential	Yes

Rheology can be beneficial in categorizing not only the viscosity of a solution but also its viscoelastic behavior. When categorizing the viscoelastic behavior of a material, they are often defined as being solid or fluid, with an ideal solid exhibiting a linear relationship between stress and strain and an ideal fluid exhibiting a linear relationship between stress and strain rate. Most complex matrices, such as that of a food system, exhibit both solid (elastic) and fluid (viscous) characteristics to varying degrees. Strain and frequency sweeps can be used to determine whether viscoelastic behavior remains constant or shifts toward fluid or solid flow profiles with increasing strain and frequency, respectively. Two moduli are used to describe viscoelastic behavior; the storage modulus corresponds to solid-like behavior, while the loss modulus corresponds to fluid-like behavior. These changes in viscosity and viscoelastic properties often can be correlated with descriptive sensory data, with rheological data being beneficial in indicating the structural changes that occur in foods with the application of different forms of deformation.

#### TRIBOLOGY OF DAIRY SYSTEMS

Tribology is an interdisciplinary field incorporating physics, chemistry, rheology, and solid and fluid mechanics to study the friction, lubrication, and wear behaviors of materials (Bhushan 2013). Tribology can be used in the study of food products to better quantify oral processing and mouthfeel. By mimicking the friction behavior among food, saliva, and oral surfaces, more insight into food textures can be gained, particularly when rheological data cannot differentiate products having different sensory characteristics (Stokes and others 2013). Tribology currently shows promise in quantifying the effect of oils and fats on food texture (Prakash and others 2013).

A typical tribology setup for tribometry performed on a rheometer is shown in Figure 2.4. The sample is loaded onto the circular plate, which is meant to mimic the mouth's soft palate. The friction coefficient of the sample is evaluated over a range of sliding speeds. A plot of friction coefficient versus sliding speed is termed a Stribeck curve (Figure 2.5), which has three distinct lubrication regimes: boundary, mixed, and hydrodynamic. The boundary regime is characterized by full surface–surface contact between and the friction behavior is driven by surface interactions. In the mixed regime, the surfaces begin to separate, but not yet to the extent seen in the hydrodynamic regime when the surfaces are fully separated by the lubricant and viscous drag dominates the friction response (Chen and Stokes 2012). There are several assumptions made when using the Stribeck curve, notably that the contact surfaces are hard (nondeformable) and the lubricant exhibits Newtonian behavior. These assumptions are not always met in tribological evaluation of food systems, so care must be taken when analyzing the results of tribological measurements on foods.

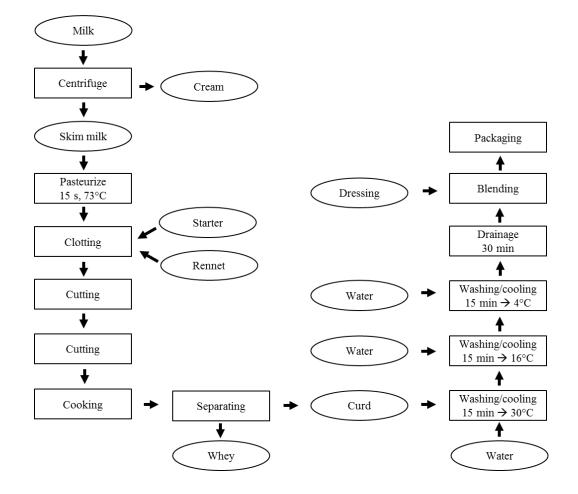
In terms of food applications, tribology is still an emerging field. Nevertheless, studying friction behavior of foods, particularly how the food matrix interacts with human saliva, can provide important information regarding texture and mouthfeel of foods that cannot be estimated using standard rheometry. Tribology is gaining traction as a food science discipline to contribute toward understanding how food behaves in the mouth. Rheometry can provide data regarding texture sensations that can be observed during the initial stages of oral processing. By measuring friction behavior, tribology can be related to other friction-related sensory terms, thus providing information that may be correlated to sensations perceived in the mouth later in oral processing (Chen and Stokes 2012).

Tribological studies have been carried out on a number of food products. The efficacy of inulin as a fat replacer in multiple dairy systems has been studied using tribology; in skim milk, the addition of inulin was found to lessen tribological differences between skim and whole milk (Meyer and others 2011b). Tribology has been used multiple times to study the effect of yogurt formulation on creaminess and in-mouth viscosity (Selway and Stokes 2013; Sonne and others 2014; Krzeminski and others 2014), further demonstrating the importance of tribology studies in the realm of texture studies. Using tribology, researchers have been able to correlate tribological behavior to a number of sensory descriptors, including creaminess (Meyer and others 2011b; Prakash and others 2013), astringency (Upadhyay and others 2016), and roughness (Selway and Stokes 2013).

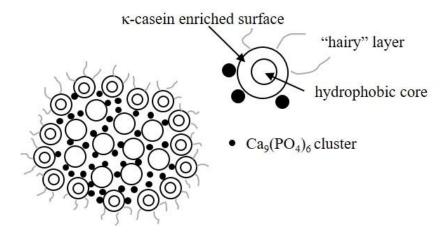
### CONCLUSIONS

Rheological, tribological, and sensory data exists on a variety of dairy products and cottage cheese curd, but there is a dearth of published scientific literature regarding cottage cheese dressing. Existing rheological data on cottage cheese has largely focused on curd firmness, while tribological studies on dairy products have mainly been concerned with studying the effect of fat on the friction behavior of fluid and semisolid dairy products.

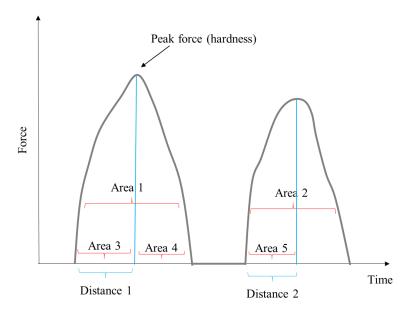
There is a growing consumer interest in reduced-sodium food products. Understanding the impact of sodium reduction and replacement in cottage cheese dressing on the final product will have important applications to the food industry by providing valuable insights for cottage cheese manufacturers. On a fundamental level, the data gathered throughout this study will be beneficial to researchers studying salt reduction in dairy systems in general. This study will help fill in critical knowledge gaps that currently exist in the published literature.



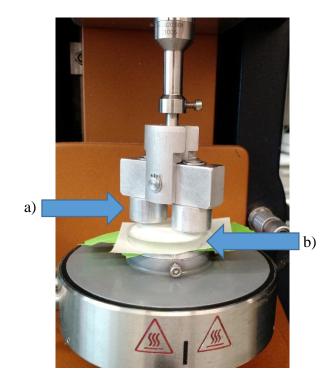
*Figure 2.1.* General process flow diagram for cottage cheese curd manufacture. Cooking time and temperature will vary based on method used. For the shortset method, 5-6% starter culture are added, while only 1% is used during longset. Rennet and starter cultures are not used during the direct set method; they are replaced with acid addition.



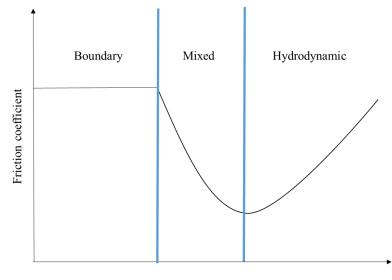
*Figure 2.2.* Cross-section of a proposed model of a casein micelle. Ca<sub>9</sub>(PO<sub>4</sub>)<sub>6</sub> comprises the hydrophobic core of the micelle, with the hydrophilic end of  $\kappa$ -casein making up the hairy layer and stabilizing the micelle.



*Figure 2.3.* Example of a potential Texture Profile Analysis readout, with time acting as the independent variable and force being dependent. Different sensory characteristics may be calculated using a variety of calculations utilizing the different areas shown in the readout.



*Figure 2.4.* Tribology setup. Arrow a) indicates the polypropylene balls acting as one contact surface, with arrow b) indicating the silicone polymer plate acting as the second contact surface. The thin film of dressing in between acts as the lubricating layer.



Sliding speed

*Figure 2.5.* Stribeck curve with lubrication regions delineated by vertical blue lines. The boundary region indicates the lack of a lubricating layer between contact surfaces, with full lubrication occurring in the hydrodynamic region. The gap between contact surfaces begins to increase with increased film thickness in the mixed region.

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# CHAPTER 3: THE IMPACT OF SALT REDUCTION ON COTTAGE CHEESE CREAM DRESSING RHEOLOGY, TRIBOLOGY, AND SENSORY BEHAVIORS

## **INTRODUCTION**

Cottage cheese consists of a soft, unaged curd to which a cream dressing is typically added (Park and Haenlein 2013). This dressing provides both flavor and texture to the curd, which ideally is nearly flavorless. Despite being relatively high in protein, with approximately 25 g protein per 225 g serving, cottage cheese is also high in sodium (approximately 800 mg per 225 g serving), with much of this sodium coming from the dressing. There has been a consumer pushback against high sodium in food products (Kim and others 2012), which may partially explain why cottage cheese consumption has decreased steadily for the past several decades (Drake and others 2009). Currently, there are few reduced sodium cottage cheese options available to consumers, but development of one may help revitalize interest in cottage cheese.

Because salt is not generally added to the cottage cheese curd, the dressing serves as the most likely medium through which to reduce salt, and thus sodium, in the finished product. There are two ways in which this can be done: simply reducing the amount of NaCl added during processing, or by partially substituting NaCl with other salts such as KCl and CaCl<sub>2</sub>. This paper focuses on the previous method of salt reduction, with three methods used to assess the dressing: rheometry, tribometry, and sensory analysis.

Rheology is the study of deformation and flow behavior of materials (Barnes and others 1989). In particular, rheology is concerned with how viscosity and viscoelastic behavior change with applied stress or strain. Because viscosity (in addition to sensory texture

measurements) is a driver of consumer liking of fluid products, understanding mechanisms for rheological behaviors is important for the study of cottage cheese cream dressing. Some rheological studies of cottage cheese curd have been conducted (Castillo and others 2006a; Castillo and others 2006b), although rheological data specifically concerned with the dressing is lacking. Joyner (Melito) and Damiano (2015) studied the impact of different hydrocolloid blends and pH on the stability and viscosity of cottage cheese dressing and found that a 2:1:1 blend of xanthan, guar, and locust bean gums were most stable during storage. The current study is a continuation of their work.

Tribology is the study of friction behavior in foods and how interacting surfaces in relative motion impact lubrication and wear. Still an emerging field in food science, tribology shows promise in correlating more successfully with sensory attributes than rheology (Chen and Stokes 2012). Tribological studies with dairy products often focus on the connection between tribology and in-mouth perception of "creaminess" and other fat-related sensory attributes (Meyer and others 2011a; Prakash and others 2013; de Wijk and Prinz 2005), although researchers have also found correlations between tribology and other textures in food including astringency (Upadhyay and others 2016; Joyner (Melito) and others 2014), roughness (Selway and Stokes 2013; de Wijk and Prinz 2005), and grittiness (van Aken 2010).

While the current study was not concerned with discovering a link between tribology and any particular sensory characteristic of cottage cheese dressing, tribological studies yield other data that can be beneficial to improved understanding of fundamental food behaviors, particularly in the area of oral processing. Some rheological properties, such as creaminess and firmness, have been shown to correlate well with sensory characteristics (Foegeding and others 2003; Janssen and others 2007), but some studies have found tribology correlates more successfully to sensory terms such as astringency and creaminess (Giasson and others 1997; Luengo and others 1997).

Data from tribological studies are generally presented in the form of Stribeck curves, which show relationships between friction coefficient and sliding speed. An ideal Stribeck curve, generated from testing a Newtonian fluid with hard contact surfaces, has three distinct regions: boundary, mixed, and hydrodynamic. However, most foods, especially complex products such as the cottage cheese dressing tested in this study, are not Newtonian. Furthermore, in an attempt to mimic the soft palate of the mouth, hard contact surfaces are not generally used. Nonetheless, it is important to understand the tribological behavior of all three regions of a Stribeck curve, even though all three regions may not be seen in the friction curve generated from a tribology run.

At lower sliding speeds, friction behavior is in the boundary regime: the friction coefficient remains relatively constant as sliding speed changes and is characterized by significant surface contact between sliding surfaces, with little sample in the gap between. The friction behavior in this regime is thus primarily impacted by interactions between sliding surfaces, although the lubricant does play some role in friction behavior. In contrast, in the hydrodynamic regime observed at high sliding speeds, the lubricant completely separates the sliding surfaces, with viscous drag of the lubricant dominating the friction response. The mixed regime marks the transition between the boundary and hydrodynamic regimes, with decreasing friction coefficient as the sliding surfaces become increasingly separated by the lubricant (Chen and Stokes 2012).

Despite the advances in rheology and tribology to understand food behavior, there is still no complete replacement for sensory analysis to fully describe the texture and flavor of various foods. However, studies using rheometry and tribometry have successfully correlated these data to descriptive and consumer sensory data. In this study, a full salt (2.2% w/w) NaCl dressing was tested against reduced NaCl formulations (1.48% w/w and 0.73% w/w) to determine the impact of these reductions on cottage cheese cream dressing rheological, tribological, and sensory behaviors. This was done by observing changes in rheological, tribological, and sensory behaviors with time, temperature, and dressing pH.

# MATERIALS AND METHODS

# Materials

Raw milk and food-grade sodium chloride were obtained from the Washington State University Creamery (Pullman, WA, USA). Ultra-pasteurized heavy cream (40% milkfat) was obtained from Darigold (Seattle, WA, USA). Xanthan gum (Keltrol) and locust bean gum (Genu Gum type RL-200z) were donated by CPKelco (Atlanta, GA, USA). Guar gum (Procol U) was donated by Polypro International Inc. (Minneapolis, MN, USA). Food-grade glucono- $\delta$  -lactone (GDL) was donated by Jungbunzlauer (Marckolsheim, France).

## Cream dressing preparation

3850

650

0.73% NaCl

Raw Cream Xanthan Locust bean Guar Salt Sample milk (g) **(g)** gum (g) gum (g) gum (g) **(g)** 2.2% NaCl 3850 650 9.0 4.5 4.5 101.25 1.48% NaCl 650 9.0 4.5 4.5 67.50 3850

9.0

4.5

4.5

33.75

Table 3.1 Dressing formulations used for this study. Milk, cream, and hydrocolloid amounts stayed consistent, with NaCl being decreased incrementally for reduced salt formulations.

Table 3.1 shows the dressing formulations used throughout this study. The dressing make procedure followed in this study was the same as that used by Joyner (Melito) and Damiano

(2015). A dressing base was creating by mixing raw milk with cream in a 37.5 L metal can. A stabilizer blend of xanthan gum, locust bean gum, and guar gum was mixed with NaCl (0.73, 1.48, and 2.20% w/w) then manually stirred into the dressing base. The highest NaCl concentration dressing was chosen based on the cottage cheese cream dressing formulation used by Joyner (Melito) and Damiano (Joyner (Melito) and Damiano 2015) The dressing was placed in a pre-heated steam chest set to 76.7°C and allowed to come up to 71.1°C in the center of the dressing. Upon reaching this point, the dressing was held for 30 min at this temperature. The pasteurized dressing was immediately homogenized in a two-stage homogenizer (APV-Gaulin model 400/200 M6-3TPS; Charlotte, NC, USA) at a first stage/second stage pressure of 3.45/11.7 MPa. The homogenized dressing was stored in sanitized 1 L plastic storage bottles in a 4°C blast chiller for 24 hr until acidification with GDL. GDL was used because it works slowly within the dressing system, thus not causing protein precipitation as a result of rapid acidification. 96. Dressing was brought to room temperature, then acidified to pH 4.5, 5.0, or 5.5 using 0.32-12.66% w/w GDL. Samples were then stored for an additional 24 hr at 4°C to allow the GDL to dissociate, then the pH of the dressing was measured. Samples were tested within 48 hours after acidification and after storage for 14 days at 4°C. pH was measured again prior to testing. Additionally, 100 mL of each sample was transferred to 50 mL plastic tubes then frozen until proximate analysis.

#### Proximate analysis

Before analysis, frozen samples were placed in a water bath at  $25^{\circ}$ C for ~1 hr until thawed. Moisture content was determined in triplicate using a forced-air oven, according to the method of Horwitz and Latimer (2010). Sample (2.5 g) was weighed into aluminum dishes, then placed in a forced air oven at 100°C for 4 hr. Ash and fat content were determined

in triplicate using standard methods for fluid dairy products (Wehr and Frank 2004). Fat content was determined using the Babcock method for cream. Samples were dry ashed by weighing 10 g of sample into ceramic crucibles, dried at 100°C for 1 hr in a forced air oven, then held overnight at 550°C in a muffle furnace. Crucibles were allowed to cool to room temperature before final weight determination. Protein content was determined using the Dumas method in a LECO FP-528 nitrogen analyzer (Saint Joseph, MI, USA). Samples weighing  $102 \pm 2.5$  mg were weighed into aluminum capsules then combusted at 850°C in the presence of oxygen. Carbon dioxide and water were separated from nitrogen, which was measured using a thermal conductivity detector (Simonne and others 1997).

#### Rheological measurements

All rheological testing was performed on a DHR-3 rheometer (TA Instruments, New Castle, DE, USA) with a 60 mm, 1° cone and temperature-controlled Peltier plate. All samples were conditioned for 10 s at the testing temperature, presheared at 10 rad/s for 20 s, then equilibrated for another 60 s. All tests were run at 8°C and 25°C, with 8°C representing a typical consumption temperature of cottage cheese and 25°C being the standard temperature for rheological testing of food.

Shear rate sweeps were performed from 0.1 to 100 s<sup>-1</sup>. Strain sweeps to determine critical strain were conducted at a frequency of 1 Hz with a ramp from 0.1 to 100% strain. Critical strain was also determined prior to running frequency sweeps. To ensure they were run within the linear viscoelastic region (LVR), critical strain was calculated. Critical strain was determined as the strain beyond which the complex modulus (G\*) varied by more than 2%. G\* was calculated using the following formula, in which G' represents the storage modulus and G" represents the elastic modulus.

$$G^{*} = \sqrt{G^{'2} + G^{''2}}$$
(1)

Frequency sweeps were run at 0.5% strain, which was in the LVR for all samples, from 0.1 to 100 Hz. All rheological tests were run in triplicate within 48 hr of acidification and again after 14 days of storage at 4°C.

#### Tribological measurements

Two representative samples were chosen from the rheology study for further tribological testing: 2.20% NaCl and 0.73% NaCl, both at pH 5.0. Tribological testing was performed in triplicate on a DHR-3 rheometer (TA Instruments, New Castle, DE, USA) using a two-ball tribological attachment. Polydimethylsiloxane (PDMS) plates were used as a testing surface to simulate the soft palate of the mouth. PDMS plates were prepared by thoroughly mixing Sylgard silicone polymer and curing agent (Dow Corning, Midland, MI, USA) in a 10:1 ratio. The mixture was added to an aluminum mold to create plates approximately 4 cm in diameter and 3 mm thick. Molds were placed in a vacuum chamber at -0.08 MPa (gauge) for 20 min to remove any bubbles, then in a 60°C oven for 2 hrs. Plates were cooled overnight at room temperature  $(23\pm2^{\circ}C)$  before removal from the molds and subsequent use.

Tribological testing was performed at 25°C and 1 N applied force. Prior to testing, all samples were equilibrated at the testing temperature for 10 s. Velocity was ramped from 0.15 to 95 mm/s with five points per decade, and the resulting friction coefficients were recorded. All surfaces were cleaned with diethyl ether between runs to limit fat buildup on the PDMS plates and balls; plates and balls were replaced every four runs to avoid confounding effects from excessive wear, with four replicates of each sample being conducted. Samples were tested twice: first within 48 hrs of acidification, and again after 14 days of storage at 4°C.

#### Sensory panel

Sensory evaluation protocols were approved by the Institutional Review Board (IRB) at the University of Idaho (protocol number 16-014).

Two representative samples (2.2% and 0.73% NaCl, both at pH 5.0) from the rheology study were evaluated by a panel of consumers. Dressing was combined with cottage cheese curd prepared in the Washington State University Creamery. To make the curd, 165 lb of pasteurized skim milk (Darigold, Seattle, WA) was combined with 160 g of #980 Chr Hansen culture and heated to 90°C. The milk was allowed to set until it reached pH 4.9. The milk was cut by hand into small curds using wire paddles. After cutting, the curds were allowed to heal for 30 min with no agitation. Curds were then gently stirred by hand and brought up to 130°C over the course of 1.5 hr. Food grade phosphoric acid (8 mL) mixed with 500 mL water was added to the curd to drop the pH to 4.55. The curd was cooled with 60 L of 4.4°C water, then excess whey and water were drained off.

The curd was stored in salt-free dressing to prevent it from drying out and to limit the amount of reduced salt dressing absorbed by the curd during the sensory panel. Absorption of the salt in the dressing by the curd prior to the panel would have changed perception of the dressing to curd ratio and perceived saltiness, so it was important to minimize these effects. Prior to each panel, this excess dressing was drained off the curd over a period of 15 min, and the drained curd was stored on ice until sample serving. Dressings were evaluated at two time points: 1) 24 hrs after acidification, and 2) after 14 days of storage at 4°C. Sixty-six panelists participated in the first panel and 64 in the second. Panelists were served curd (30 mL) mixed with dressing (15 mL) (2:1 curd to dressing ratio) in randomized order. The dressing was stored on ice to maintain a consistent serving temperature and prepared as needed per panelist.

Basic demographic information (gender, age, race, marital status, education level, employment status, income, and cottage cheese consumption) was gathered from panelists on a voluntary basis. Panelists evaluated samples for saltiness, acidity, and dressing consistency on a five-point "just about right" (JAR) scale. Overall texture, overall appearance, overall liking, and bitterness of the samples were also evaluated on a 9-point hedonic scale. An example of the Compusense-generated ballot is included in the Appendix.

#### Data analysis

Graphs were generated using Microsoft Excel software. (Redmond, WA, USA). Proximate analysis data were analyzed with XLSTAT (Addinsoft; New York, NY, USA) using one-way analysis of variance (ANOVA) followed by Tukey's HSD.

The average shear rate sweep data for each formulation were fit to flow behavior models using TRIOS software (version 9.13; TA-Instruments). Data were fit to the Herschel-Bulkley model, the equation for which is given below.

$$\sigma = \sigma_0 + K \dot{\gamma}^n \tag{2}$$

In this formula,  $\sigma$  represents shear stress (Pa),  $\sigma_0$  is the yield stress (Pa), K is the consistency coefficient (Pa.s<sup>n</sup>),  $\dot{\gamma}$  is shear rate (s<sup>-1</sup>), and n is the flow behavior index (unitless).

A two-way ANOVA followed by Tukey's HSD was conducted on the hedonic sensory data using XLSTAT (Addinsoft; New York, NY, USA). XLSTAT was also used to run penalty analysis on JAR results.

#### **RESULTS AND DISCUSSION**

#### Proximate analysis

Proximate compositions of all formulations are shown in Table 3.2. The difference in ash content was expected, as reducing the amount of salt in the dressing also decreased the mineral content. The difference in fat contents may have occurred because samples were prepared over the course of several months (August–March). Fat content in milk can vary across seasons due to dairy cattle dietary changes (Larsen and others 2014; Versteeg and others 2016; Heck and others 2009; Auldist and others 1998; Ozrenk and Inci 2008). However, because the dressings were otherwise similar, most of the differences observed in the rheological, tribological, and sensory behaviors of the dressing formulations could reasonably be attributed to the varying salt content.

#### Rheological behavior: viscosity

All formulations displayed shear-thinning behavior, as demonstrated in Figure 3.1.

Sample	Target pH	Actual pH	Moisture (%)	Protein (%)	Fat (%)	Ash (%)
	4.5	4.53	80.68 <sup>a</sup> (0.11)	2.23 <sup>a</sup> (0.04)	8.10 <sup>e</sup> (0.14)	2.63 <sup>a</sup> (0.11)
2.20% NaCl	5.0	5.02	80.52 <sup>a</sup> (0.03)	2.40 <sup>a</sup> (0.06)	8.18 <sup>de</sup> (0.11)	2.61 <sup>a</sup> (0.03)
	5.5	5.54	80.54 <sup>a</sup> (0.08)	2.56 <sup>a</sup> (0.21)	8.45 <sup>bcd</sup> (0.07)	$2.74^{a}(0.12)$
	4.5	4.49	80.50 <sup>a</sup> (0.15)	2.47 <sup>a</sup> (0.04)	8.60 <sup>abc</sup> (0.14)	2.18 <sup>b</sup> (0.07)
1.48% NaCl	5.0	5.01	80.63 <sup>a</sup> (0.11)	2.26 <sup>a</sup> (0.29)	8.58 <sup>abc</sup> (0.06)	2.14 <sup>bc</sup> (0.19)
	5.5	5.53	80.73 <sup>a</sup> (0.11)	2.27 <sup>a</sup> (0.10)	8.78 <sup>a</sup> (0.04)	2.18 <sup>b</sup> (0.09)
	4.5	4.50	80.48 <sup>a</sup> (0.07)	2.43 <sup>a</sup> (0.07)	8.75 <sup>ab</sup> (0.14)	1.96 <sup>bc</sup> (0.05)
0.73% NaCl	5.0	4.98	80.74 <sup>a</sup> (0.10)	2.26 <sup>a</sup> (0.09)	8.37 <sup>cde</sup> (0.13)	1.93° (0.06)
	5.5	5.54	80.63 <sup>a</sup> (0.06)	2.45 <sup>a</sup> (0.27)	8.17 <sup>de</sup> (0.18)	1.97 <sup>bc</sup> (0.12)

Table 3.2. Proximate analysis and pH values for dressing formulations

Values in each column are mean (standard deviation). Numbers followed by the same letter within a column indicate there were no significant differences between values.

increased regardless of sodium concentration. This viscosity variation was attributed to aggregation of the casein micelles. The isoelectric point (pI) of casein is pH 4.6; near the pI, disruption of the colloidal calcium phosphate (CCP) that helps stabilize the casein micelles occurs, causing casein aggregation and formation of a three-dimensional network (Brule and others 2000; Lucey 2002; Phadungath 2005). Viscosity differences were not attributed to pH sensitivity of the hydrocolloids used in the dressing formulations: all hydrocolloids used in this study are relatively pH stable, with significant changes in functionality not occurring until acidic conditions near pH 3.0 are reached (Phillips and Williams 2009; Sae-kang and Suphantharika 2006; Lal and others 2006; Kok 2010).

Storage time also had an impact on dressing viscosity, with samples exhibiting higher viscosities after 14 days of storage regardless of formulation, although this increase was not always significant, particularly at pH 5.0 (both temperatures) and pH 5.5 at 8°C. Increases in viscosity were expected: increased interaction time between the hydrocolloid blend and the dressing matrix allowed for increased structure buildup. Viscosities were also generally higher at 8°C versus 25°C. Again, this result was expected, as materials generally exhibit increased viscosity at lower temperatures.

In general, the 1.48% NaCl formulation exhibited the highest viscosity, regardless of pH, temperature, or test time. However, it is important to note that it was not always significantly higher than the viscosities of the other samples, as evidenced by the overlap in the standard error bars (Figure 3.1). Other studies have found that increasing NaCl concentration in acid milk gels and other acidified dairy systems caused a decrease in viscosity

Sample (% NaCl)	Day	Temperature (°C)	рН	σ0 (Pa)	K (Pa <sup>n</sup> )	n	R <sup>2</sup>
2.20 -			4.5	-	160.81	0.06	0.932
		8	5.0	28.82	5.76	0.68	0.997
	1 —		5.5	6.16	10.99	0.31	0.972
		25	4.5	-	49.82	0.18	0.985
			5.0	17.19	5.07	0.63	0.999
			5.5	12.91	1.70	0.65	0.985
	14 —		4.5	-	168.33	0.20	0.984
		8	5.0	25.53	13.87	0.59	0.998
			5.5	1.92	17.46	0.25	0.969
		25	4.5	-	42.36	0.28	0.989
			5.0	26.72	11.74	0.46	0.976
			5.5	17.56	1.38	0.64	0.965
			4.5	-	79.03	0.29	0.995
		8	5.0	-	69.48	0.30	0.996
			5.5	7.91	11.97	0.50	0.991
	1 —		4.5	14.90	18.95	0.36	0.996
		25	5.0	-	36.39	0.27	0.994
1 40			5.5	19.11	4.03	0.64	0.998
1.48			4.5	-	118.14	0.27	0.994
		8	5.0	-	94.06	0.23	0.995
	14 —		5.5	2.417	29.70	0.32	0.997
12			4.5	-	56.27	0.21	0.994
		25	5.0	3.05	37.60	0.21	0.986
			5.5	12.98	15.05	0.42	0.995
			4.5	-	63.57	0.15	0.980
		8	5.0	-	72.18	0.31	0.995
	1		5.5	9.446	33.72	0.25	0.984
	1 -		4.5	4.843	19.43	0.25	0.993
		25	5.0	-	32.86	0.18	0.982
0.73 -			5.5	6.13	10.25	0.43	0.987
			4.5	-	356.91	0.19	0.994
		8	5.0	9.74	32.29	0.33	0.990
	1.4		5.5	9.45	33.720	0.25	0.646
	14 —		4.5	-	145.44	0.43	0.981
		25	5.0	18.15	16.83	0.44	0.996
			5.5	3.73	14.18	0.48	0.999

Table 3.3. Flow model fit and variable values for averaged shear rate sweep data. Other variables are indicated by consistency coefficient (K) and flow behavior index (n). Correlation coefficients are represented by  $R^2$ .

(Köksoy and Kılıç 2003; Schkoda and others 1999). This inverse relationship between NaCl concentration and viscosity was attributed to an increase in repulsive forces from sodium ions at the casein micelle surface, which reduced casein micelle aggregation (Köksoy and Kılıç 2003; Schkoda and others 1999). This may also explain why the 2.20% NaCl formulation had decreased viscosity in comparison to the other NaCl concentrations. However, at low concentrations, salts can increase viscosity due to charge screening (salting in) (Arakawa and Timasheff 1984), which may also explain why the 0.73% formulation had greater viscosities, particularly at pH 5.0

As seen in Table 3.3, shear rate sweep data were fit to the Herschel-Bulkley and Power Law models. The relationship between stress and strain for both models is the same, but Herschel-Bulkley fluids have a yield stress and Power Law fluids do not.

Yield stress of the different formulations varied widely. Storage time did not appear to have a consistent impact on yield stress. An increase in yield stress would have seemed likely, given the viscosity increase during storage was likely indicative of increased hydrocolloidprotein interactions. However, yield stress did not show definable trends with storage time. There were a number of potential explanations for this. Structural buildup may have increased both viscosity and yield stress, but it was possible that this increase was still weak enough to be immediately disturbed with shear application. The exception was for the lower pH samples, which had larger increases in yield stress between time points, which was also reflected in the strain sweeps.

The lower pH samples were less likely to have a yield stress. Consistency coefficients for samples without a yield stress also tended to be noticeably larger. These results support the previous discussion of viscosity values. Despite greater viscosity values at lower pH, it is likely that the increased internal network formed in the dressings at pH near the isoelectric point of casein was easily broken with applied shear. In contrast, at pH further from the pI, overall viscosity was lower, but the internal network was stronger. The larger critical strain values for the higher pH samples, which will be discussed later, also support this argument.

Consistency coefficient (K) is strongly related to viscosity, with larger K values indicating higher viscosities. Trends for K among formulations, temperature, and time were similar to those seen in the shear rate sweeps. The lower pH formulations had larger values of K, and K increased with time. The increase in K with time was also reflected in the shear rate sweep data (Figure 3.1) and were again likely a result of increased internal structure in the dressing as the hydrocolloids had longer to interact with the dressing proteins.

Flow behavior index (n) was less than 1 for all formulations, indicating pseudoplastic behavior. Pseudoplasticity of these samples was expected because the xanthan, locust bean, and guar gum hydrocolloid blend used in all formulations has been shown to impart pseudoplastic behavior in solution (García-Ochoa and others 2000; Doublier and Launay 1981). Values of n increased with pH, indicating decreased pseudoplastic behavior. As noted previously, this can be attributed to decreased protein aggregation as samples were further from the isoelectric point of casein (Brule and others 2000; Phadungath 2005).

#### Rheological behavior – oscillatory testing

Strain sweep data showed that all samples behaved as viscoelastic solids regardless of formulation, storage time, or temperature (Figure 3.2). The dominance of the storage modulus (G') over the loss modulus (G'') at lower strains indicated the presence of a weak gel, likely due to the protein-polysaccharide interactions in the dressing matrix. As strain increased, the

protein-polysaccharide network began to break down, causing permanent deformation and the eventual dominance of viscous behavior.

The general trends among the strain sweep data were in keeping with the shear rate sweep data. After 14 days of storage, the 0.73% NaCl formulation at pH 4.5 showed a higher degree of solid-like behavior compared to other salt concentrations at the same pH. At pH 5.0 and 5.5, the 1.48% NaCl formulation showed consistently higher G' values. Increases in G' with time were not always significant, which is in keeping with the flow models in Table 3.2. Formulations at a lower pH had higher values of G' and G", which were attributed to the behavior of casein at its isoelectric point. Near the isoelectric point, decreased electrostatic repulsion increases casein-casein interactions, allowing for more numerous and stronger bonds (Lucey 2002; Brule and others 2000). As a result, the protein-polysaccharide network in the samples was stiffer, showing increased G' values (Lee and Lucey 2004). In addition, the moduli values at 8°C were higher than at 25°C. Other studies have shown that acidified milk systems, which were comparable to those in this study, had higher storage moduli at lower temperatures (Koutina and others 2014; Roefs and others 1990).

Determining a strain that was within the linear viscoelastic region (LVR) for all samples was particularly important for the subsequent frequency sweep. Within the LVR, the structure of the material being tested is not permanently deformed by stresses and strains imparted during testing. If testing occurs outside the LVR, the results become difficult to interpret, as there are too many confounding variables affecting the data. The critical strain, which marks the end of the LVR, was calculated by determining the strain after which the complex modulus, G\*, changes by more than 2% between data points. Critical strain values ranged from 0.6% to 6% strain (data not shown). Thus, 0.5% strain was well within the LVR for all

samples; any changes observed during the frequency sweeps were attributed to formulation differences and testing parameters, not material damage induced by the test itself.

As noted above, critical strain varied greatly across formulations, time point, and test temperature. At higher temperature, critical strains tended to be larger, and critical strain also increased with pH. Samples acidified to higher pH levels had larger critical strains regardless of temperature, indicating more elastic gels at this pH. Critical strains at 25°C were generally higher than at 8°C. At lower temperature, the protein-polysaccharide network in the samples was hypothesized to be more rigid, breaking down more easily with increased strain application while simultaneously showing more elastic behavior at low strain due to their more rigid structure.

The observed trends for frequency sweep data (Figure 3.3) were in alignment with other rheological data. In general, G' and G" values were higher at lower pH and at 8°C. Increases in viscoelastic moduli with time was observed across all dressing formulations and testing conditions. As with the strain sweep data, G' values were greater than G" values, indicating viscoelastic solid behavior. The frequency dependency was indicative of weak gel behavior, meaning the internal network of the dressing was sensitive to the rate of applied strain.

#### Tribological behavior

The two samples chosen for tribological evaluation (2.20% NaCl and 0.73% NaCl, both at pH 5.0) were selected to represent the rheological variations in formulations. Similar to the rheological results, in which viscosity and viscoelasticity increased with time, friction coefficients also increased after storage (Figure 3.4). At day 1, the tribological behaviors for both formulas were not significantly different. This was in contrast to the rheological data, where the 0.73% NaCl dressing at pH 5.0 had consistently higher viscosity and viscoelastic

moduli values for both strain and frequency sweeps. By day 14, friction coefficient values for the 2.20% NaCl dressing at low sliding speeds were significantly larger than those for the 0.73% NaCl dressing.

Both samples used in the tribology study had relatively small boundary regions. For viscous products such as the samples used in this study, it is not unusual to have a small or nonexistent boundary regime when soft surfaces are used for testing. Increased resistance to flow makes it more difficult to displace the film that exists between the interacting tribological surfaces (Chen and Stokes 2012). Other tribological studies using acidified dairy systems have also observed a minimal boundary region, which was attributed to PDMS plate deformation at the lower sliding speeds (Joyner (Melito) and others 2014; Selway and Stokes 2013).

The mixed region of the friction curve for both samples at day 1 and day 14 were of approximately equal length. During the mixed region, the 2.20% NaCl formulation still had significantly higher friction coefficient values, although they were not significantly different by its end. The more pronounced mixed region observed in this study may be as a result of the presence of hydrocolloids rather than salt concentration. It has been suggested that the presence of polymers, such as guar gum, physically separate the testing surfaces. Additionally, the increased viscosity imparted by hydrocolloid use limits turbulent flow and drag between the surfaces, thus lowering the friction coefficient (Malone and others 2003). The rheological portion of this study showed that the samples used were viscous as a result of hydrocolloid use (Joyner (Melito) and Damiano 2015), and the friction curves reflect this. However, viscous fluids would also be expected to enter the hydrodynamic regime at lower sliding speed than observed in this study. The shear thinning nature of the samples accounted for this. At lower sliding speed, the gap between contact surfaces was smaller; at this point, the shear rate was

also higher. The shear thinning nature of the dressing resulted in their viscosities being reduced, keeping them in the mixed region for a longer period of time. As the sliding speed increased, the viscosity and thickness of the lubricating layer did as well, increasing friction coefficient and leading into the hydrodynamic region (Dresselhuis 2008).

As sliding speed increased to approximately 9.5 mm/s, the samples entered the hydrodynamic region. The differences between samples, regardless of formulation and testing time point, were minimized in this region. As seen in Figure 3.1, there were no significant differences in viscosity among any dressing formulations at pH 5.0 and 25°C, regardless of time point. Thus, the tribological behaviors observed in the hydrodynamic region (Figure 3.4) aligned with previously demonstrated rheological behaviors.

## Sensory study

In general, demographic makeup between the two panels (day 1 and day 14) was similar. The main difference was in the numbers of male to female participants in the day 1 versus day 14 panels. On day 1, 41 of the 66 panelists were female, while on day 14 only 28 of the 64 panelists were female. The average age of participants was 35-40 yr, with most being white or Asian. The majority of participants had at least some college education and an income below \$50,000. Most panelists were not frequent consumers of cottage cheese, eating it once a month or less. A detailed breakdown of the demographic information collected at both panels is included in the Appendix.

Table 3.4 shows the average liking scores for the hedonic liking data on both days of testing. In the day 1 panel, significant differences were found between the 2.2% and 0.73% NaCl dressing formulations in every category except appearance and texture. Overall liking of the 0.73% NaCl formulation was significantly lower than the 2.20% NaCl formulation.

Table 3.4. Average 9-point hedonic liking scores of overall liking, appearance, texture, flavor, and bitterness of full- and reduced-salt dressing formulations. Liking scores were taken 24 hours and 14 days after dressing acidification to pH 5.0.

	Attribute										
Commla	Overall liking		Appearance		Texture		Flavor		Bitterness		
Sample	Day										
	1	14	1	14	1	14	1	14	1	14	
2.20% NaCl	6.35 <sup>a</sup>	6.50 <sup>a</sup>	6.17 <sup>a</sup>	6.14 <sup>a</sup>	6.42 <sup>a</sup>	6.30 <sup>a</sup>	6.35 <sup>a</sup>	6.45 <sup>a</sup>	6.23 <sup>a</sup>	6.25 <sup>a</sup>	
0.73% NaCl	5.68 <sup>b</sup>	5.91ª	5.86 <sup>a</sup>	6.22 <sup>a</sup>	6.05 <sup>a</sup>	6.06 <sup>a</sup>	5.55 <sup>b</sup>	5.88 <sup>a</sup>	5.62 <sup>b</sup>	5.77 <sup>a</sup>	

Sample means followed by the same letter within each column are not significantly different.

Because of the significant differences in liking of bitterness and flavor between the two formulations, it seems that flavor, rather than texture, had a more significant impact on liking.

Multiple panelists commented that the 0.73% NaCl dressing was bland or flat, which is not surprising given its relatively low salt content (66% below the amount used according to the method used by Joyner (Melito) and Damiano (2015). The salt added to cottage cheese dressing not only helps limit microbial growth during storage, but also imparts flavor to the curd, which is manufactured to be relatively flavorless. A straight reduction in NaCl without the use other salts or flavor enhancers is likely to give a rather bland final product. However, it is surprising that panelists found the 0.73% NaCl formulation to have a more objectionable level of bitterness. Sodium substitutes such as CaCl<sub>2</sub> and KCl have been shown to impart a bitter flavor in foods, particularly at high usage rates (de Almeida and others 2016; Charlton and others 2007), but no substitutions were used in this study. It is possible that panelists were experiencing bitter carryover, especially because other samples tested during the same panel did contain sodium substitutes, which will be discussed in Chapter 4. Microbial growth which produced bitter peptides is another possibility, although this was less likely given that bitterness did not increase during storage, which would be expected as bacterial growth increased over time (Doyle and Glass 2009).

Statistical analysis after 14 days of storage revealed that hedonic liking scores on the second day of testing were not significantly different than for freshly prepared dressing. While scores were generally lower for the 0.73% NaCl dressing compared to the 2.20% NaCl dressing at day 14, these differences were not significant. The lack of significance between hedonic liking scores at day 14 may have been due to different panel makeup, with panelists at day 14 being less discerning about cottage cheese preferences.

Penalty analysis data are summarized in Figure 3.5 and Table 3.4. From Figure 3.5, it can be seen that a higher proportion of panelists found the salt level in the 2.2% formulation to be more acceptable than in the 0.73% formulation. Given that the NaCl was so significantly reduced in the latter sample, it is not surprising that over half the panelists found there to be too little salt in that formulation. As time passed, the proportion of panelists in each JAR category for salt, acidity, and dressing consistency did not appreciably change for the 0.73% formulation.

From Table 3.5, it appeared that dressing acidity had the most significant impact on overall liking of the dressing samples, particularly when the acid level was deemed too high. Panelists also more strongly penalized the 2.2% NaCl dressing that was considered too thick after day 14. Mean drops in liking scores also increased from day 1 to day 14 for both

Table 3.5. Penalty analysis of saltiness, acidity, and consistency of full- and reduced-salt dressings. Overall liking scores of samples that were marked as having too little or too much for each category were compared to samples that were just about right. The mean drop in liking scores are represented for each sample at day 1 and day 14 after acidification.

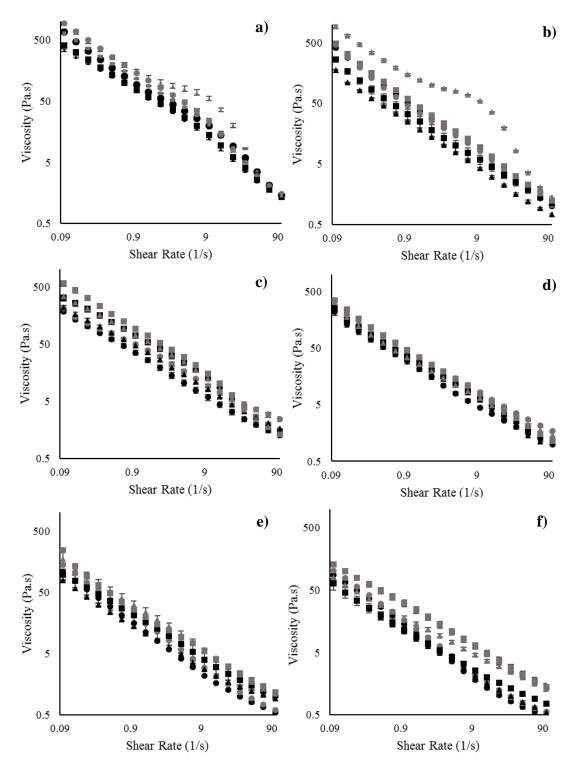
		Salt		Acid		Dressing Consistency	
		Day 1	Day 1 Day 14		Day 14	Day 1	Day 14
2.20/ N. Cl	Too little	0.43	1.46	1.04	1.16	0.04	0.62
2.2% NaCl	Too much	0.52	1.06	1.27	2.96	-0.52	2.71
0.73% NaCl	Too little	1.36	1.34	1.31	1.31	0.74	0.28
0.75% NaCI	Too much	-0.04	2.23	0.98	1.91	0.15	0.36

formulations and all categories, which is interesting given that overall liking scores between both days were revealed through ANOVA to not be significantly different. Because this was a consumer panel and there was variation in participants, it is possible that day 14 panelists simply penalized dressing that differed from their ideal more than day 1 panelists.

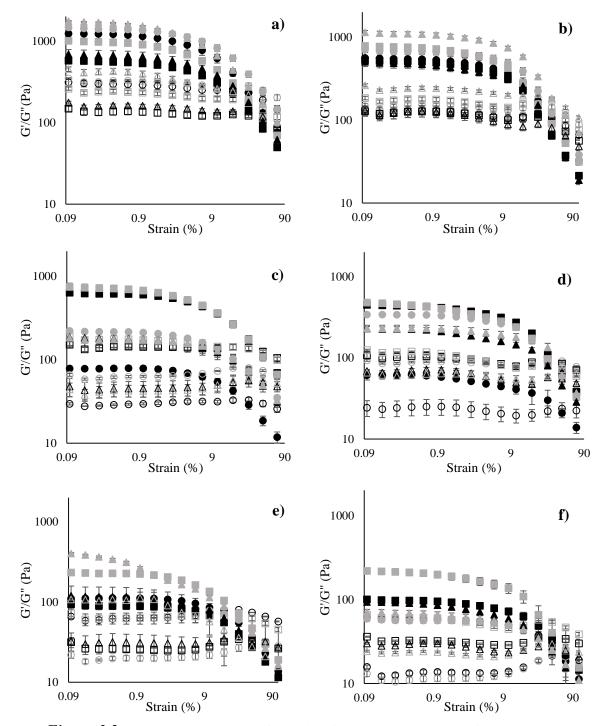
Given the sensory performance of the reduced NaCl formulation in comparison to the full salt formulation, it appears that creation of a reduced salt cottage cheese dressing formulation that consumers find acceptable is certainly feasible. Although hedonic liking scores of the 0.73% NaCl formulation tended to be lower than that of the 2.2% NaCl formulation, they were not significantly different, especially after 14 days of storage. However, because a large proportion of panelists found the reduced salt formulation to be lacking in salt, exploration of sodium substitute performance, such as KCl and CaCl<sub>2</sub>, which still possess some salty flavor, would be advisable. Other flavors to mask the lack of saltiness is another potential means of reducing sodium without compromising flavor.

## CONCLUSIONS

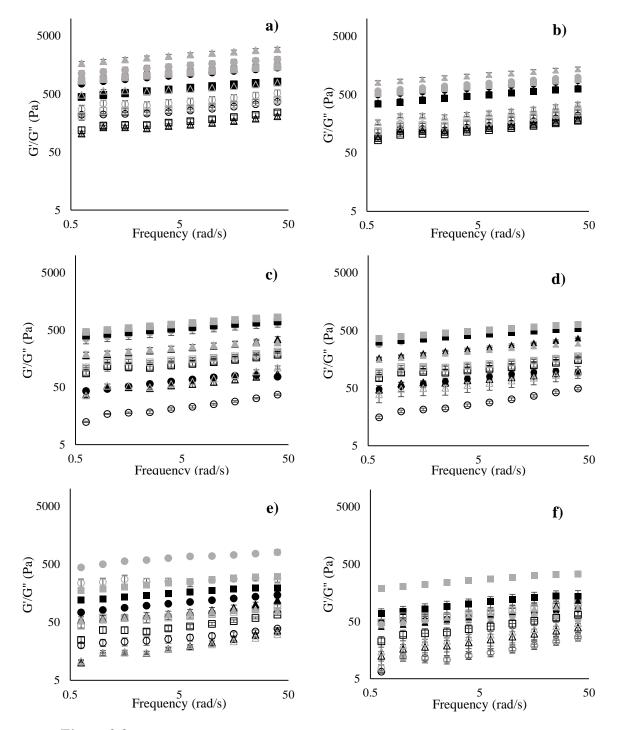
Rheological, tribological, and sensory behavior of cottage cheese cream dressing was dependent not only on NaCl concentration, but also pH, test temperature, and storage. All samples were shear-thinning and showed increased viscosity and viscoelastic solid behavior over a storage period of two weeks. pH tended to have the most significant impact on rheological behavior, although differences were also observed as NaCl concentration changed. Tribological behavior changed more with time, and the lower NaCl concentration was deemed less acceptable by consumers. These results indicate that reduced NaCl cottage cheese dressing formulations may not be accepted by consumers without additional reformulation. Examining the impacts of other components such as fruit or savory flavor enhancers is a potential followup to this study. However, given these results, reduction of NaCl in cottage cheese seems feasible from a rheological, tribological, and sensory perspective. The information gained from this study not only fills a knowledge gap about the mechanical behaviors of cottage cheese cream dressing but also offers valuable data for other researchers studying sodium reduction in acidified dairy systems.



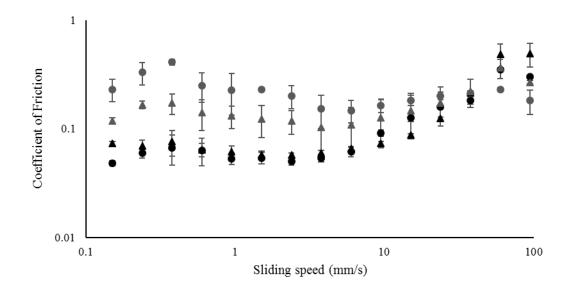
*Figure 3.1* Viscosity profiles of reduced sodium dressing formulations generated from shear rate sweep data. a) pH 4.5 at 8°C b) pH 4.5 at 25°C c) pH 5.0 at 8°C d) pH 5.0 at 25°C e) pH 5.5 at 8°C f) pH 5.5 at 25°C. Black symbols indicate day 1 samples; gray symbols indicate day 14 samples. Circles represent 2.20% NaCl, squares 1.48% NaCl, and triangles 0.73% NaCl. Error bars indicate standard error.



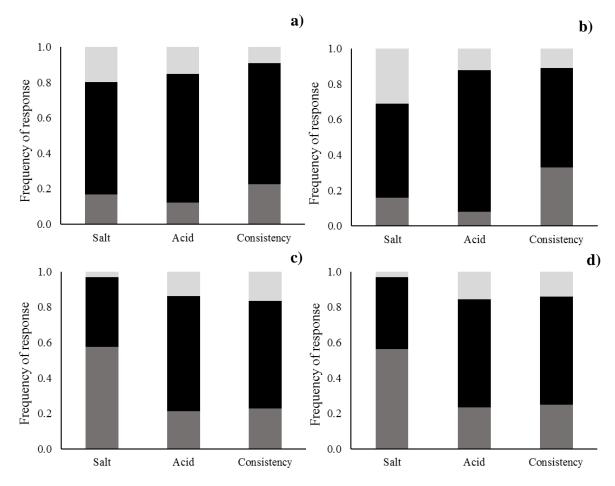
*Figure 3.2* Viscoelastic behavior of dressing formulations generated from strain sweep data. a) pH 4.5 at 8°C b) pH 4.5 at 25°C c) pH 5.0 at 8°C d) pH 5.0 at 25°C e) pH 5.5 at 8°C f) pH 5.5 at 25°C. Black symbols indicate day 1 samples; gray symbols indicate day 14 samples. Open symbols represent G" values, and filled symbols represent G'. Circles represent 2.20% NaCl, squares 1.48% NaCl, and triangles 0.73% NaCl. Error bars represent standard error.



*Figure 3.3* Viscoelastic behavior of dressing formulations generated from frequency sweep data. a) pH 4.5 at  $8^{\circ}$ C b) pH 4.5 at  $25^{\circ}$ C c) pH 5.0 at  $8^{\circ}$ C d) pH 5.0 at  $25^{\circ}$ C e) pH 5.5 at  $8^{\circ}$ C f) pH 5.5 at  $25^{\circ}$ C. Black symbols indicate day 1 samples; gray symbols indicate day 14 samples. Open symbols represent G" values, and filled symbols represent G'. Circles represent 2.20% NaCl, squares 1.48% NaCl, and triangles 0.73% NaCl. Error bars indicate standard error.



*Figure 3.4* Friction curve of 2.2% and 0.73% NaCl cottage cheese dressings. Circles indicate the 2.2% formulation and triangles the 0.73% NaCl formulation. Black symbols are from day 1 of testing, and gray from day 14. Error bars indicate standard error.



*Figure 3.5* Just About Right frequency of responses for salt, acidity, and dressing consistency. Panelist responses were grouped into three categories: too low, just about right, and too high, with the relative frequencies of each adding up to 1. The light gray portions of each bar indicate the too high category, black just-about-right, and dark gray too low. In order, the graphs represent the following: a) 2.2% NaCl dressing at day 1 b) 2.2% NaCl dressing at day 14 c) 0.73% NaCl dressing at day 1 d) 0.73% NaCl dressing at day 14.

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# CHAPTER 4: THE IMPACT OF SALT REPLACEMENT ON COTTAGE CHEESE CREAM DRESSING RHEOLOGY, TRIBOLOGY, AND SENSORY BEHAVIOR

## **INTRODUCTION**

Sodium reduction in processed foods has become an increasingly important issue to consumers in recent years (Kim and others 2012), yet there are few reduced sodium cottage cheese formulations currently in commercial production. As consumption of cottage cheese has declined during the past several decades, successful creation of a reduced sodium cottage cheese may help re-invigorate sales of this once popular dairy product. In cottage cheese, the salt (sodium) in the cream dressing helps provide much-needed flavor to the relatively bland curd. As such, it is especially important that any formulations that are lower in salt provide adequate flavor to the finished product. There are two main methods for salt reduction in food: 1) a simple reduction of the concentration of NaCl added to the product and 2) substitution with other salts. Simple reduction of NaCl was discussed in Chapter 3, and this chapter will focus on substitution of NaCl with two common sodium replacers: KCl and CaCl<sub>2</sub>.

Studies on cottage cheese have focused primarily on curd properties and sensory attributes (Castillo and others 2006a; Castillo and others 2006b; Antinone and others 1994; Drake and others 2009), with several studies also focusing on salt reduction in cottage cheese (Demott and others 1984; Shelf and Ryan 1988; Wyatt 1983). Specific investigation of the cream dressing added for flavor and texture is lacking. Joyner (Melito) and Damiano (2015) recently examined how hydrocolloid blends and pH affected the stability of cottage cheese cream dressing; this study is a continuation of that work.

Both of the sodium substitutes used in this study (KCl and CaCl<sub>2</sub>) present several potential difficulties in food formulations. As a divalent cation, calcium may exhibit different charge screening capabilities than the monovalent potassium or traditionally used sodium. The differences in charge screening may affect the dressing microstructure, which would impact viscosity and viscoelastic behavior. Additionally, researchers have found that both KCl and CaCl<sub>2</sub> can cause a bitter off-flavor, particularly when used above a 50% substitution ratio for NaCl (de Almeida and others 2016; Katsiari and others 1998; Fitzgerald and Buckley 1985; Park and others 2009).

Rheology and tribology were used in tandem in this study to provide a deeper understanding of salt interactions with other components in the dressing matrix. Rheology and tribology provide valuable insights into food mechanical and friction behaviors, and have the potential to improve fundamental understanding of oral processing and texture attributes. Figure 4.1 shows an ideal Stribeck curve that could theoretically be generated in a tribological test. Friction behavior in the boundary and mixed regimes is controlled primarily by surfacesurface interactions, with the lubricant properties having an increasing impact on friction behavior as the lubrication regime moves from boundary (significant surface-surface contact) to mixed (reduced contact and a thicker lubricant layer between the surfaces). Friction behavior in the hydrodynamic regime, where the sliding surfaces are completely separated, is dominated by rheological properties (Prakash and others 2013). As a result, rheological data can be used concurrently with tribological data to characterize food behavior. Correlations between tribology and sensory data have also been found for various fluid and semisolid foods (Meyer and others 2011a; Prakash and others 2013; Sonne and others 2014; Joyner (Melito) and others 2014; Selway and Stokes 2013). This study was not concerned specifically with finding correlations between sensory and tribological behavior, but sensory data was still important to collect to assess consumer liking of sodium-substituted dressing samples.

In combining the three areas discussed above into one study, a well-rounded view of cottage cheese cream dressing behavior can be determined. Thus, the primary objective of this study was to determine whether NaCl-substituted dressing could be created that did not differ significantly in terms of rheological, tribological, and sensory behaviors from dressing formulated with only NaCl.

## MATERIALS AND METHODS

## Materials

Raw milk and food-grade sodium chloride were obtained from the Washington State University Creamery (Pullman, WA, USA). Pasteurized cream (40% milkfat) was obtained from Darigold (Seattle, WA, USA). Xanthan gum (Keltrol) and locust bean gum (Genu Gum type RL-200z) were donated by CPKelco (Atlanta, GA, USA). Guar gum (Procol U) was donated by Polypro International Inc. (Minneapolis, MN, USA). Food-grade glucono-δlactone (GDL) was donated by Jungbunzlauer (Marckolsheim, France). KCl was donated by Chemtrade (Midlothian, TX, USA) and CaCl<sub>2</sub> was purchased from Modernist Pantry (York, ME, USA).

#### Cream dressing preparation

*Table 4.1. Formulations for NaCl-substituted cottage cheese dressings using KCl and CaCl<sub>2</sub>.* 

Sample	Milk (g)	Cream (g)	XG (g)	LBG (g)	GG (g)	NaCl (g)	KCl (g)	CaCl <sub>2</sub> (g)
1:3 NaCl:KCl	3850	650	9.0	4.5	4.5	25.3	75.9	_
1:1 NaCl:KCl	3850	650	9.0	4.5	4.5	50.6	50.6	_
3:1 NaCl:KCl	3850	650	9.0	4.5	4.5	75.9	25.3	_
1:3 NaCl:CaCl <sub>2</sub>	3850	650	9.0	4.5	4.5	25.3	_	75.9
1:1 NaCl:CaCl <sub>2</sub>	3850	650	9.0	4.5	4.5	50.6	_	50.6
3:1 NaCl:CaCl <sub>2</sub>	3850	650	9.0	4.5	4.5	75.9	_	25.3

XG: xanthan gum; LBG: locust bean gum; GG: guar gum

Table 4.1 shows the dressing formulations used in this study. Dressing was prepared according to the method used by Joyner (Melito) and Damiano (2015). Raw milk (3.85 kg) was mixed with cream (0.65 kg) in a 37.5 L metal can to create a dressing base. Xanthan gum (9 g), locust bean gum (4.5 g) and guar gum (4.5 g) were mixed with NaCl and KCl or CaCl<sub>2</sub> before addition into the dressing base. The total salt content of all samples remained constant at 2.2% (w/w). Partial substitution of NaCl using KCl and CaCl<sub>2</sub> occurred at 1:3, 1:1, and 3:1 NaCl:KCl/CaCl<sub>2</sub> ratios. A steam chest was used to pasteurize the dressing at 71.1°C for 30 min. Immediately following pasteurization, the warm dressing was run through a two-stage homogenizer (APV-Gaulin model 400/200 M6-3TPS; Charlotte, NC, USA) at 3.45/11.7 MPa first/second stage. Prior to acidification with GDL, the dressing was stored in sanitized plastic storage bottles for 24 hr in a 4°C blast chiller. GDL was stirred into room temperature dressing (25°C) using 0.12-1.34% (w/w) GDL to acidify dressing samples to pH 4.5, 5.0, and 5.5. Samples were then held at 4°C for another 24 hr prior to further testing to allow adequate time for acidification. Excess sample (~100 mL) was frozen to be used later for proximate analysis.

## Proximate analysis

Frozen samples were thawed in a water bath at 25°C prior to analysis. A forced-air oven was used to determine moisture content in triplicate according to the method of Horwitz and Latimer (2010). Samples weighing 2.5 g were placed in aluminum dishes and dried 4 hr at 100°C. Fat content and ash were also determined in triplicate using the Babcock method for cream (Wehr and Frank 2004). For ashing, 10 g of sample were weighed into ceramic crucibles then dried 1 hr at 100°C in a forced air oven. Samples were held overnight in a muffle furnace at 550°C then allowed to cool to room temperature in a dessicator before final weight determination. A LECO FP-528 nitrogen analyzer (Saint Joseph, MI, USA) was used

to determine protein content using the Dumas method. Aluminum capsules containing  $102 \pm 2.5$  mg of sample each were combusted at 850°C in the presence of oxygen. Nitrogen was separated from carbon dioxide and water, with the nitrogen content then being measured using a thermal conductivity detector (Simonne and others 1997). Nitrogen content was converted to protein content using the conversion factor for dairy products of 6.38.

#### Rheological measurements

A DHR-3 rheometer (TA Instruments, New Castle, DE, USA) with a 60 mm diameter, 1° cone and temperature controlled Peltier plate was used for all rheological testing. All tests were run at 8°C and 25°C. Samples were equilibrated at the testing temperature for 10 s followed by a 20 s pre-shear at 10 rad/s before equilibration for a final 60 s. Testing was immediately started after the final equilibration.

Viscosity profiles were generated using a shear rate sweep run from 0.1 to 100 s<sup>-1</sup>. Strain sweeps were run at frequency 1 Hz from 0.1 to 100% strain. This data was used to calculate the critical strain and ensure the frequency sweep was run within the linear viscoelastic region (LVR). The complex modulus (G\*) was calculated using the following formula:

$$G^* = \sqrt{(G')^2 + (G'')^2} \tag{3}$$

The critical strain was determined to be the point at which G\* changed by more than 2% between strain values. For the frequency sweep, a strain of 0.5% was used with a ramp from 0.1 to 100 Hz. All samples were run in triplicate within 48 hrs of acidification, stored at 4°C for 14 days, then tested again.

## Tribological measurements

After analysis of the rheological data, four representative samples were chosen for tribological testing: 1:3 and 1:1 NaCl:KCl/CaCl<sub>2</sub>, all at pH 5.0. The same DHR-3 rheometer

(TA Instruments, New Castle, DE, USA) from the rheology study was used for tribological testing, this time using a two-ball tribological attachment and polydimethylsiloxane (PDMS) plates. PDMS plates were used due to their similarities in rigidity to the soft palate of the mouth. Sylgard silicone polymer and curing agent (Dow Corning, Midland, MI, USA) were thoroughly mixed in a 10:1 ratio. An aluminum mold was used to create plates 4 cm in diameter and 4 mm thick. A vacuum chamber at -0.08 MPa pressure for 20 min was used to eliminate air bubbles present in the mixture after they were poured into the mold. Plates were then cured in a 60°C oven for 2 hrs, then cooled overnight at room temperature before removal from the molds.

All tribological tests were performed with four replicates at 25°C and 1 N of applied force. Equilibration at 25°C for 10 s was conducted on all samples prior to testing. A velocity ramp from 0.15 to 95 mm/s with five points per decade was applied to each sample, and the resulting friction coefficient was recorded. In between runs, diethyl ether was used to limit fat buildup on the PDMS plates and balls. To avoid confounding effects from excess wear, PDMS plates and balls were replaced every four runs. As in the rheological study, samples were tested at two time points: within 48 hr of GDL acidification and again after 14 days of storage at 4°C.

#### Sensory analysis

Two consumer sensory panels using the same dressing formulations as in the tribology study was also conducted, with a control sample containing 2.2% NaCl and no sodium substitutes also being evaluated. The first panel was held immediately after the 24 hr acidification period passed, with a second panel following after 14 days of dressing storage. Cottage cheese curd was prepared in the Washington State University Creamery. Skim milk (Darigold, Seattle, WA) (165 lb) was mixed with 160 g of #980 Chr Hansen culture. The cultured milk was then heated to 90°C and allowed to set until a pH of 4.9 was reached. Small curds were cut by hand using wire paddles then left to heal for 30 min. Over the course of 1.5 hr, the curds were stirred slowly and constantly by hand until a temperature of 130°C was reached. Phosphoric acid (8 mL) and water (500 mL) were added to the curd to decrease the pH to 4.55. The curd was then cooled with 60 L of 4.4°C water before draining the excess whey and water. The finished curd was stored in a salt free dressing. This was done for two reasons: 1) to keep the curd from drying out during storage and 2) to limit the amount of dressing absorbed by the curd during the panel. Dressing absorption by the curd would have changed the curd:dressing ratio, potentially to the point where the perceived texture would have been affected, providing a confounding effect to the final results.

Before each panel, the curd was drained of the salt-free dressing in a colander, then dressing samples and curd were stored separately on ice until needed. Each panelist received a sample cup containing a 2:1 curd to dressing ratio (30 mL of curd and 15 mL of dressing). All samples received a randomly generated three-digit code, and samples were presented in a randomized order to each panelist. Each panelist was asked a series of voluntary demographic questions (gender, age, race, marital status, level of education, employment status, annual income, and frequency of cottage cheese consumption). Each sample was evaluated on a 9-point hedonic scale for overall liking, overall texture, sample appearance, and bitterness. Samples were also evaluated for saltiness, acidity, and dressing consistency on a 5-point "just about right" (JAR) scale. The Compusense-generated ballot is included in the Appendix.

### Data analysis

Microsoft Excel (Redmond, WA, USA) was used to generate all graphs. Proximate analysis data and hedonic sensory data were analyzed with XLSTAT (Addinsoft; New York, NY, USA) using two-way analysis of variance (ANOVA) followed by Tukey's HSD to determine whether significant differences in means existed. XLSTAT was also used to conduct penalty analysis on sensory JAR data.

Shear rate sweep data from the rheological study were fit to flow behavior models using TRIOS software (version 9.13; TA Instruments). Analysis revealed shear rate sweep data were best fit to the Herschel-Bulkley fluid model. The equation for the Herschel-Bulkley model is given below.

$$\sigma = \sigma_0 + K \dot{\gamma}^n \tag{4}$$

In this formula,  $\sigma$  represents shear stress (Pa),  $\sigma_0$  is the yield stress (Pa), K is the consistency coefficient (Pa.s<sup>n</sup>),  $\dot{\gamma}$  is shear rate (s<sup>-1</sup>), and n is the unitless flow behavior index.

#### **RESULTS AND DISCUSSION**

## Proximate analysis

Dressing composition of all formulations are shown in Table 4.2. No significant differences were found among the means for each component. Ash content did not vary significantly among samples, which was to be expected given that total salt content remained consistent, unlike in Chapter 3. Because of these minimal differences, it is reasonable to conclude that any variation among samples was a result of different ingredient ratios rather than an artifact of formulation differences.

Sample	Target pH	Actual pH	Moisture (%)	Protein (%)	Fat (%)	Ash (%)
1:3 NaCl:KCl	4.5	4.50	80.99 <sup>a</sup> (0.35)	2.05 <sup>a</sup> (0.04)	8.10 <sup>a</sup> (0.14)	2.63 <sup>a</sup> (0.07)
1:3 NaCl:KCl	5.0	4.98	80.91 <sup>a</sup> (0.41)	2.39 <sup>a</sup> (0.27)	8.23 <sup>a</sup> (0.04)	2.65 <sup>a</sup> (0.09)
1:3 NaCl:KCl	5.5	5.53	81.10 <sup>a</sup> (0.54)	2.29 <sup>a</sup> (0.10)	8.35 <sup>a</sup> (0.07)	2.54 <sup>a</sup> (0.07)
1:1 NaCl:KCl	4.5	4.47	80.59 <sup>a</sup> (0.19)	2.11 <sup>a</sup> (0.52)	8.55 <sup>a</sup> (0.07)	$2.58^{a}(0.06)$
1:1 NaCl:KCl	5.0	4.96	80.90 <sup>a</sup> (0.32)	2.26 <sup>a</sup> (0.31)	8.15 <sup>a</sup> (0.07)	$2.65^{a}(0.03)$
1:1 NaCl:KCl	5.5	5.48	80.41ª (0.22)	$2.66^{a}(0.25)$	8.15 <sup>a</sup> (0.07)	$2.62^{a}(0.09)$
3:1 NaCl:KCl	4.5	4.54	80.42 <sup>a</sup> (0.19)	2.44 <sup>a</sup> (0.08)	8.58 <sup>a</sup> (0.04)	2.61 <sup>a</sup> (0.08)
3:1 NaCl:KCl	5.0	5.03	$80.77^{a}(0.07)$	2.41 <sup>a</sup> (0.07)	$8.50^{a}(0.14)$	$2.50^{a}(0.06)$
3:1 NaCl:KCl	5.5	5.50	80.61 <sup>a</sup> (0.33)	$2.82^{a}(0.06)$	8.38 <sup>a</sup> (0.11)	$2.48^{a}(0.08)$
1:3 NaCl:CaCl <sub>2</sub>	4.5	4.52	80.38 <sup>a</sup> (0.09)	2.86 <sup>a</sup> (0.16)	8.25 <sup>a</sup> (0.07)	$2.56^{a}(0.06)$
1:3 NaCl:CaCl <sub>2</sub>	5.0	4.97	80.99 <sup>a</sup> (0.06)	2.62 <sup>a</sup> (0.25)	8.50 <sup>a</sup> (0.14)	$2.53^{a}(0.08)$
1:3 NaCl:CaCl <sub>2</sub>	5.5	5.49	80.40 <sup>a</sup> (0.25)	$2.47^{a}(0.30)$	8.30 <sup>a</sup> (0.14)	$2.56^{a}(0.06)$
1:1 NaCl:CaCl <sub>2</sub>	4.5	4.46	80.60 <sup>a</sup> (0.38)	2.16 <sup>a</sup> (0.09)	8.20 <sup>a</sup> (0.14)	$2.62^{a}(0.03)$
1:1 NaCl:CaCl <sub>2</sub>	5.0	5.04	80.62 <sup>a</sup> (0.29)	2.73 <sup>a</sup> (0.01)	8.08 <sup>a</sup> (0.11)	$2.48^{a}(0.05)$
1:1 NaCl:CaCl <sub>2</sub>	5.5	5.51	80.49 <sup>a</sup> (0.07)	2.29 <sup>a</sup> (0.35)	8.50 <sup>a</sup> (0.14)	2.55 <sup>a</sup> (0.13)
3:1 NaCl:CaCl <sub>2</sub>	4.5	4.49	80.45 <sup>a</sup> (0.12)	2.38 <sup>a</sup> (0.08)	8.43 <sup>a</sup> (0.18)	$2.62^{a}(0.06)$
3:1 NaCl:CaCl <sub>2</sub>	5.0	5.00	80.45 <sup>a</sup> (0.22)	2.39 <sup>a</sup> (0.21)	8.50 <sup>a</sup> (0.14)	2.59 <sup>a</sup> (0.09)
3:1 NaCl:CaCl <sub>2</sub>	5.5	5.54	80.33 <sup>a</sup> (0.12)	2.50 <sup>a</sup> (0.17)	8.50 <sup>a</sup> (0.14)	2.61 <sup>a</sup> (0.13)

Table 4.2. Composition of NaCl substituted cottage cheese dressings using KCl and CaCl<sub>2</sub>

Values in each column are mean (standard deviations). Means in a column followed by the same letter are not significantly different.

Rheological behavior: viscosity

All formulations (KCl- and CaCl<sub>2</sub>-substituted dressings) exhibited shear thinning behavior (Figures 4.2 and 4.3). Viscosity values at 8°C were slightly higher across some formulations and after storage, although the difference was not significant for every formulation. This result was not surprising, given that materials typically have higher viscosities at lower temperatures. Also predictably, viscosity values after 14 days of storage were higher, likely due to increased time for strengthening the hydrocolloid-protein interactions in the dressing matrix. However, increases in viscosity over time and at lower temperature were not significant.

pH had a bigger impact on viscosity trends than temperature or salt used. At lower pH, near the isoelectric point of casein (pI=4.6), the stabilizing effect of colloidal calcium

Sample	Day	Temperature (°C)	рН	σ0	K	n	R <sup>2</sup>
			4.5	-	97.45	0.30	0.996
		8	5.0	-	43.49	0.25	0.990
	1		5.5	9.02	7.16	0.54	0.991
	1		4.5	-	54.74	0.18	0.990
		25	5.0	9.96	7.90	0.51	0.989
KC1			5.5	9.24	2.99	0.61	0.991
KCl -			4.5	-	134.43	0.30	0.990
		8	5.0	17.72	57.21	0.28	0.986
	1.4		5.5	11.06	7.02	0.48	0.988
	14		4.5	-	50.46	0.30	0.990
		25	5.0	26.70	18.14	0.34	0.986
			5.5	12.85	13.82	0.55	0.998
			4.5	19.20	39.03	0.20	0.957
		8	5.0	15.02	12.15	0.47	0.995
	1		5.5	11.65	12.76	0.52	0.996
	1		4.5	16.90	5.84	0.55	0.991
		25	5.0	8.89	6.89	0.58	0.996
			5.5	7.43	7.33	0.57	0.996
CaCl <sub>2</sub>			4.5	36.04	8.89	0.54	0.976
		8	5.0	21.76	11.51	0.43	0.982
	1.4		5.5	24.44	12.14	0.48	0.988
	14		4.5	21.76	3.89	0.62	0.991
		25	5.0	14.75	5.14	0.59	0.996
			5.5	13.10	6.81	0.58	0.995

*Table 4.3. Flow model fit and variable values for 1:3 NaCl:KCl/CaCl<sub>2</sub> substituted dressing formulations* 

phosphate (CCP) in the casein micelle is disrupted, which causes aggregation of casein and the formation of a three-dimensional network (Brule and others 2000; Lucey 2002; Phadungath 2005). Differences for KCl were more pronounced than for CaCl<sub>2</sub>. The difference in viscosity values between samples at pH 4.5 versus pH 5.0 was less pronounced than that observed when comparing them with dressing at pH 5.5. At pH 5.0, the sample pH was close enough to the isoelectric point of casein that there was still a significant amount of charge neutralization. With pH operating on a log scale, pH 5.5 was sufficiently removed from the isoelectric point so that the difference in viscosity was greater. Because the hydrocolloids used

Sample	Day	Temperature (°C)	рН	<b>σ</b> 0	K	n	R <sup>2</sup>
			4.5	-	123.41	0.25	0.996
		8	5.0	-	94.88	0.19	0.994
	1		5.5	3.52	31.16	0.26	0.946
	1		4.5	-	41.42	0.25	0.994
		25	5.0	20.06	21.61	0.31	0.992
V.CI			5.5	17.26	18.52	0.29	0.926
KCl -			4.5	-	164.39	0.21	0.989
		8	5.0	-	129.11	0.16	0.989
	14		5.5	-	73.08	0.20	0.992
			4.5	-	93.83	0.23	0.992
		25	5.0	-	58.94	0.21	0.992
			5.5	13.41	6.20	0.58	0.991
			4.5	-	55.47	0.34	0.991
		8	5.0	16.20	16.35	0.45	0.993
			5.5	16.82	17.37	0.47	0.993
	1		4.5	7.77	19.29	0.34	0.999
		25	5.0	13.00	12.08	0.48	0.996
G (1			5.5	11.01	10.28	0.53	0.996
CaCl <sub>2</sub>			4.5	-	75.54	0.34	0.988
		8	5.0	18.38	24.42	0.32	0.985
	1.4		5.5	20.33	17.26	0.39	0.986
	14		4.5	16.61	19.74	0.35	0.998
		25	5.0	15.82	9.07	0.47	0.991
			5.5	11.54	8.96	0.49	0.994

*Table 4.4. Flow model fit and variable values for 1:1 NaCl:KCl/CaCl<sub>2</sub> substituted dressing formulations.* 

in this study are stable in the range of pH values tested, it is unlikely that viscosity differences with changing pH were as a result of changing hydrocolloid efficacy (Phillips and Williams 2009; Lal and others 2006; Kok 2010).

Viscosity differences between samples were larger at low shear for all formulations. As shear rate increased beyond 9 s<sup>-1</sup>, differences were minimized. Above this shear rate, it is possible that sufficient deformation had been applied to dressing microstructure, breaking down interchain interactions and causing the polymers to align rather than tangle, which would decrease the viscosity. Greater differences between formulations were seen at 25°C, particularly in the KCl-substituted dressing samples.

No significant differences were observed in viscosity profiles for KCl- and CaCl<sub>2</sub>substituted dressing formulations, although dressings made with CaCl<sub>2</sub> had slightly lower viscosities than those made with KCl. In general, dressing made with a higher ratio of either salt substitute (1:3 NaCl:KCl/CaCl<sub>2</sub>) had lower viscosities, compared to the 1:1 and 3:1 NaCl:salt substitute dressings. Charge screening offers a potential explanation. As a divalent cation, calcium was able to screen more negative charges present in the dressing polymers. The increased salt-polymer interactions likely promoted increased polymer-polymer interactions, which would have resulted in the polymers taking up less overall space in the

Sample	Day	Temperature (°C)	рН	<b>σ</b> 0	K	n	<b>R</b> <sup>2</sup>
			4.5	-	108.81	0.25	0.996
		8	5.0	-	84.89	0.23	0.994
	1		5.5	13.06	1.20	0.73	0.977
	1		4.5	-	46.08	0.22	0.996
		25	5.0	-	44.12	0.24	0.992
VO			5.5	12.02	1.21	0.72	0.979
KCl -			4.5	-	217.63	0.26	0.994
		8	5.0	-	128.40	0.21	0.997
	14		5.5	18.22	10.78	0.42	0.962
	14		4.5	-	91.66	0.22	0.994
		25	5.0	-	65.44	0.22	0.969
			5.5	19.21	1.95	0.79	0.985
			4.5	-	76.39	0.25	0.994
		8	5.0	18.89	10.46	0.52	0.991
			5.5	16.52	11.16	0.50	0.990
	1		4.5	18.97	9.07	0.49	0.985
		25	5.0	12.78	5.51	0.62	0.977
			5.5	8.95	6.95	0.55	0.995
CaCl <sub>2</sub>			4.5	-	176.26	0.41	0.990
		8	5.0	-	101.72	0.27	0.996
	14		5.5	13.27	27.34	0.29	0.967
	14		4.5	-	67.62	0.27	0.989
		25	5.0	-	51.24	0.22	0.990
			5.5	20.44	5.02	0.57	0.982

*Table 4.5. Flow model fit and variable values for 3:1 NaCl:KCl/CaCl<sub>2</sub> substituted dressing formulations* 

dressing matrix and thus a decrease in viscosity.

Tables 4.3-4.5 show the flow models for the KCl- and CaCl<sub>2</sub>-substituted dressing formulations. All formulations were fit to the Herschel-Bulkley models. At a yield stress value of zero, which was the case for several samples, the Herschel-Bulkley model reduces to the Power Law model. High correlation coefficient values indicate these models were a good fit. Samples that did have a yield stress generally showed a higher yield stress at 8°C and after 14 days of storage. Given the shear rate sweep trends, this result was not surprising, since comparable viscosity changes were seen with these conditions as well. The consistency coefficient, K, is strongly correlated with viscosity, and the general trends seen with these values among formulations also followed those seen in the shear rate sweeps. Consistency coefficients increased with decreasing pH, which again was likely due to colloidal calcium phosphate disruption and casein aggregation near the isoelectric point. Values of K were also higher at 8°C than at 25°C, which again was because viscosity tends to decrease with increasing temperature. After 14 days of storage, values of K were also higher, indicating again that there may have been increased interaction and structural buildup among hydrocolloids and proteins.

The flow behavior index, n, was less than 1 under all testing conditions, which indicated the pseudoplastic nature of these samples. Pseudoplastic behavior was also expected, as viscosity decreased with increasing shear rate during the shear rate sweeps. Additionally, hydrocolloids such as those used in these formulations have been shown to impart pseudoplastic behavior to solutions (Doublier and Launay 1981; García-Ochoa and others 2000). Values of n also tended to be higher at pH 5.5, regardless of substitution ratio, salt substitute, or testing parameters, indicating decreased pseudoplastic behavior. This may have been as a result of increased distance from the isoelectric point of casein, resulting in less protein aggregation.

#### *Rheological behavior: oscillatory testing*

Overall, differences between samples were more pronounced for strain sweeps (Figures 4.4 and 4.5) and frequency sweeps (Figures 4.6 and 4.7) in comparison to shear rate sweep data. All sodium substitutions showed viscoelastic solid behavior, regardless of pH, temperature, or storage time, as evidenced by G'>G'' (Figures 4.4-4.7). The dominance of G' over G'' indicated weak gel formation due to hydrocolloid interactions with the proteins present in the dressing matrix. The formation of a weak gel as a result of hydrocolloids in the dressing matrix is in alignment with other studies that have observed gelation of solutions when using xanthan, locust bean, and guar gums (Copetti and others 1997; Sanchez and others 2000; Phillips and Williams 2009).

In keeping with the shear rate sweep data, differences between formulations were less pronounced when comparing pH 4.5 and pH 5.0, while values for G' and G" at pH 5.5 were significantly smaller. Samples containing a greater percentage of NaCl tended to have higher values of G' and G" overall, indicating formation of stronger internal networks inside the dressing. Values for both moduli (G' and G") increased with time, although, as for the shear rate sweeps, the differences in G' and G" immediately after acidification versus 14 days of storage were not always significant. Significant increases in G' and G" did occur for some samples, particularly at lower pH (4.5) in the KCl-substituted formulations. CaCl<sub>2</sub>-substituted dressing also had significant increases in G' for some formulations, most notably in the 1:1 substitution and at pH 4.5. Overall, the impact of varying salt type and substitution amount had a more pronounced impact on viscoelastic behavior than flow behavior.

Determination of critical strain values from strain sweep data was important in conducting valid frequency sweeps. Within the LVR, the deformation applied to the material over the course of testing does not permanently damage its structure. Any differences in rheological behavior can be attributed to formulation and test parameters such as temperature, rather than a permanently altered material structure. While there are rheological tests designed to observe material behavior outside of the LVR, these tests were not within the scope of this study.

Critical strain, which marks the end of the LVR, was defined as the point beyond which the complex modulus G\* varied by more than 2% with changing percent strain application to the material (Joyner (Melito) and Meldrum 2016). Although the critical strain varied from 1-6% among formulations, most samples had a critical strain between 1 and 1.5%. Thus, a strain of 0.5% was chosen for the frequency sweeps, as it was within the LVR for all samples. Storage time did not have a significant effect on critical strain values, but pH and testing temperature did. The LVR tended to be larger at 25°C and shorter at lower pH. The impact of pH on the LVR was especially pronounced for the KCl-substituted samples at pH 4.5 and 5.0.

Beyond the LVR and at high strain, G" was greater than G', indicating that at these high strains, the internal network of the dressing matrix broke down, rendering differences among formulations negligible. The dressing samples became more fluid-like as the weak interactions between the hydrocolloids and proteins were disrupted.

The increases in critical strain with increasing pH and temperature (Figures 4.4 and 4.5) indicated a stronger internal network, but the frequency sweep data provided a better idea of the resistance of the microstructure to permanent deformation. All samples showed weakly frequency-dependent behavior. G' and G" both increased with increased frequency, although

G' was greater than G'' for the entirety of the test (Figures 4.6 and 4.7). It is not uncommon for G' of materials to increase with frequency (Steffe 1996), with a stronger microstructure resulting in less frequency-dependent behavior.

As with the other rheological tests, G' and G" increased with storage time, with these increases being nonsignificant for CaCl<sub>2</sub>-substituted dressing formulations at pH 5.0 and 5.5. Testing at 8°C as opposed to 25°C also resulted in slightly higher overall values of G' and G", which is in agreement with the other rheological tests (Figures 4.2-4.5). These results may be related to the increased viscosity at 8°C, which was observed in this study and others (Vandresen and others 2009). More variations were seen among pH levels for the KCl-substituted dressings compared to the strain sweep results, but the most pronounced difference still occurred as pH increased from 5.0 to 5.5.

## Tribological behavior

Four representative samples spanning the range of the rheological results obtained were chosen for tribological evaluation: 1:1 and 1:3 NaCl:KCl and 1:1 and 1:3 NaCl:CaCl<sub>2</sub>, all at pH 5.0. Friction coefficient values increased with time for all samples (Figure 4.9), which is in keeping with viscosity increases and more viscoelastic behavior seen in the rheological portion of this study. The friction curves of all samples showed predominately mixed and hydrodynamic behavior, with a small boundary region at sliding speeds up to 0.375 mm/s at the start of each curve. The only exception was the 1:3 NaCl:KCl formulation at day 14, which not only had the highest friction coefficient values overall but also had the longest and most pronounced boundary region, likely as a result of its decreased viscosity and weaker internal network seen in the rheological behavior.

All samples had a minimal boundary region, which is more common in thick or viscous materials because the higher viscosity of the substance makes it easier to completely separate the contact surfaces (Chen and Stokes 2012). Another possible explanation for the lack of a boundary region was deformation of the PDMS plates at low sliding speed (Joyner (Melito) and others 2014; Selway and Stokes 2013).

Samples after 14 days of storage had a longer mixed region compared to samples tested shortly after acidification. Malone and others (2003) suggested that polymers such as the hydrocolloids, particularly guar gum, suppress turbulent flow and reduce drag (and thus the friction coefficient), keeping the samples in the mixed region for a longer period of time. Polymer interactions during storage were strengthened, as evidenced by increased viscosity and viscoelastic behavior in the rheological study, which gives further credence to the idea that it was hydrocolloid interactions rather than salt that contributed to the more pronounced mixed regime.

Once sliding speed increased beyond 10 mm/s and samples entered the hydrodynamic region, differences between formulations and testing time point were minimal. In the hydrodynamic region, material friction behavior is typically governed by bulk viscosity due to full separation of the contact or testing surfaces (Prakash and others 2013). The shear rate sweep data (Figures 4.3) showed few significant differences among formulations at 25°C and pH 5.0, particularly at higher shear rates. Thus, it was not surprising to also observe negligible tribological differences in the hydrodynamic region.

#### Sensory study

The samples evaluated by tribometry were also evaluated by a general consumer panel. Demographic information between the first (day 1, n=66) and second (day 14, n=64) sensory panels was similar, with the biggest difference being the male-to-female ratio between the two panels. In the first panel, 36% of panelists were male and 62% female (2% of panelists preferred not to answer). In the second panel, 55% of panelists were male and 44% female (1% preferred not to answer). The majority of panelists were Caucasian or Asian, with at least some college education. Most panelists were not frequent consumers of cottage cheese, eating it a few times a month or fewer.

Significant differences were found among samples as compared to the control sample for all attributes except for liking of sample appearance (Table 4.6). The lack of significant differences for sample appearance liking was not surprising, as the formulation variations did not create significant visual differences, such as color or lumpy appearance. Samples containing CaCl<sub>2</sub> generally had significantly lower scores than the control; KCl-substituted dressing sample scores (particularly the 1:1 substitution ratio) were generally not significantly different from the control.

Bitterness appeared to strongly impact overall liking and flavor acceptability. Given that bitterness is generally considered a negative attribute of food, it is not surprising that samples that were more bitter would be more objectionable as a whole. CaCl<sub>2</sub> in particular is frequently deemed an unusable sodium substitute because of the imparted bitter flavor, especially at high concentrations (such as in the 1:3 substitution in this study) (de Almeida and others 2016; Fitzgerald and Buckley 1985; Luna-Guzman and Barrett 2000). Increased bitterness resulting from KCl use in food systems has also been reported, particularly when used at a replacement rate in excess of 30% (Katsiari and others 1997; Charlton and others 2007; Park and others 2009), although it does not appear to have had such a strong negative impact on the sensory results in either panel. Other studies have found samples using KCl as a salt replacement to

Sample		Attribute								
	Overal	l liking	Appea	arance	Tex	Texture		Flavor		rness
	Day 1	Day 14	Day 1	Day 14	Day 1	Day 14	Day 1	Day 14	Day 1	Day 14
Control	6.35 <sup>a</sup>	6.50 <sup>a</sup>	6.17 <sup>a</sup>	6.41 <sup>a</sup>	6.42 <sup>a</sup>	6.30 <sup>ab</sup>	6.35 <sup>a</sup>	6.45 <sup>a</sup>	6.23 <sup>a</sup>	6.25 <sup>a</sup>
1:3 KCl	5.46 <sup>b</sup>	5.75 <sup>a</sup>	5.46 <sup>a</sup> *	6.41 <sup>a</sup> *	6.30 <sup>ab</sup>	6.39ª	5.17 <sup>b</sup>	5.50 <sup>ab</sup>	5.18 <sup>b</sup>	5.44 <sup>ab</sup>
1:1 KCl	5.51 <sup>ab</sup>	5.55ª	5.82 <sup>a</sup>	6.36ª	5.81 <sup>ab</sup>	6.20 <sup>ab</sup>	5.53 <sup>ab</sup>	5.47 <sup>b</sup>	5.70 <sup>ab</sup>	5.34 <sup>b</sup>
1:3 CaCl <sub>2</sub>	3.55°	3.25 <sup>c</sup>	6.00 <sup>a</sup>	5.78 <sup>a</sup>	5.58 <sup>ab</sup>	5.59 <sup>b</sup>	3.44°	3.06 <sup>d</sup>	3.26 <sup>c</sup>	2.80 <sup>d</sup>
1:1 CaCl <sub>2</sub>	4.36 <sup>c</sup>	4.47 <sup>b</sup>	5.47 <sup>a</sup>	5.88ª	5.68 <sup>b</sup>	5.98 <sup>ab</sup>	4.23°	4.34 <sup>c</sup>	3.97°	4.28 <sup>c</sup>

*Table 4.6. Least squares means for sodium substituted cottage cheese dressing formulations, with a 2.2% (w/w) NaCl sample acting as the control.* 

Sample means followed by the same letter within each column are not significantly different. Least squares means followed by an asterisk indicate significant differences between day 1 and day 14 scores.

not be objectionable in terms of flavor, even at high substitution rates (Guardia and others 2008; Tangkham and LeMieux 2016).

Storage time did not have a significant impact on scores, except for liking of appearance of the 1:3 NaCl:KCl dressing formulation. Despite not being significantly different, liking of texture and appearance increased slightly for multiple formulations, which may be due to the slight viscosity and viscoelasticity increases that were also seen in the rheological portion of this study. This result may also have been due simply to having different panelists with different preferences evaluate samples.

Penalty analysis results are summarized in Table 4.7 and Figure 4.9. From Table 4.7, it was apparent that day 14 panelists penalized samples that differed from their ideal salt, acidity, and dressing consistency more strongly than panelists at day 1. This was an interesting result because overall liking scores between day 1 and day 14 were not significantly different for any formulations. Furthermore, too much acid in the 1:3 NaCl:KCl formulation in particular was strongly penalized at day 14, but the distribution of ranking scores did not change significantly between panels (Figure 4.9). The acid level of the 1:3 NaCl:CaCl<sub>2</sub> formulation

		Salt		Acid		Dressing Consistency		
		Day 1	Day 14	Day 1	Day 14	Day 1	Day 14	
1:3 NaCl:KCl	Too little	0.86	1.71	1.05	1.88	0.43	0.98	
1.5 NaCl.KCI	Too much	-0.33	1.74	0.02	2.82	0.19	1.15	
1:1 NaCl:KCl	Too little	1.73	1.18	1.71	1.24	0.48	1.75	
1.1 NaCl.KCI	Too much	1.25	2.74	1.76	1.44	0.08	0.87	
1:3 NaCl:CaCl2	Too little	0.39	-0.48	1.70	1.18	0.98	0.33	
1.5 NaCI.CaCI2	Too much	-0.26	0.29	1.95	1.06	-0.14	-0.03	
1:1 NaCl:CaCl <sub>2</sub>	Too little	0.27	0.91	-0.35	1.96	0.99	0.80	
	Too much	0.07	1.17	2.17	1.71	0.90	0.40	

Table 4.7. Mean drops in overall liking scores for samples panelists considered not justabout-right

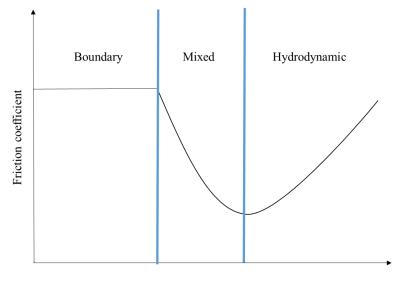
was one of the few that had a large change in JAR distribution between days 1 and 14, although it still was not significant.

Dressing consistency was the sensory characteristic most strongly related rheological and tribological behaviors. From looking at Figure 4.9, it is apparent that most panelists found the dressing consistency to be just about right. The only exception was the 1:1 NaCl:KCl dressing at day 1, which a high proportion of panelists found to be too thin. This was in contrast to the shear rate sweep results, where this dressing formulation had comparable viscosity to the other NaCl substituted formulations. It is possible that of the samples made for the sensory panel, this formulation was slightly less viscous in comparison to the other dressing formulations, leading more panelists to deem it too thin. In general, dressing consistency variations between NaCl substitutions and after storage were minimal, which was in keeping with the rheological and tribological data.

Overall, the 1:1 NaCl:KCl dressing formulation was most similar to the control. From the hedonic sensory data, it is apparent that this substitution ratio was less likely to differ significantly from the 2.2% NaCl dressing.

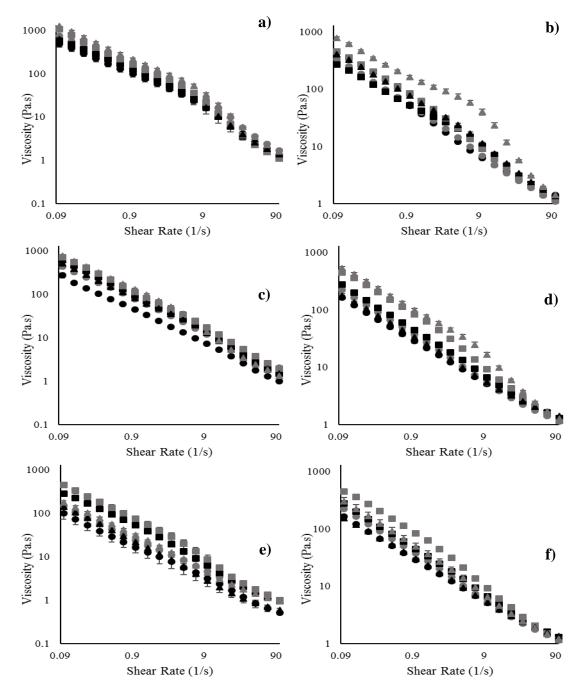
#### CONCLUSIONS

The rheological behaviors of cottage cheese cream dressings prepared with different substitution ratios of NaCl to KCl and CaCl<sub>2</sub> were dependent on not only salt content, but also temperature, pH, and time. Usage of a divalent versus a monovalent salt resulted in noticeable differences across all results, even at the same substitution ratio. Differences in storage time, pH, and salt substitution ratio also resulted in differing behaviors, but salt type was the most significant contributor to behavioral and sensory differences. Based on these results, KCl seems to be a more viable salt substitute than CaCl<sub>2</sub>, particularly because KCl-substituted dressing samples preferred by consumers in the sensory panel. In particular, a 1:1 NaCl:KCl substitution ratio appeared to be the sodium-substituted formulation most likely to be accepted by consumers. This study highlights the importance of understanding how different salts interact with the cottage cheese cream dressing matrix, and how pH and substitution ratio can be manipulated to find the best formulation.

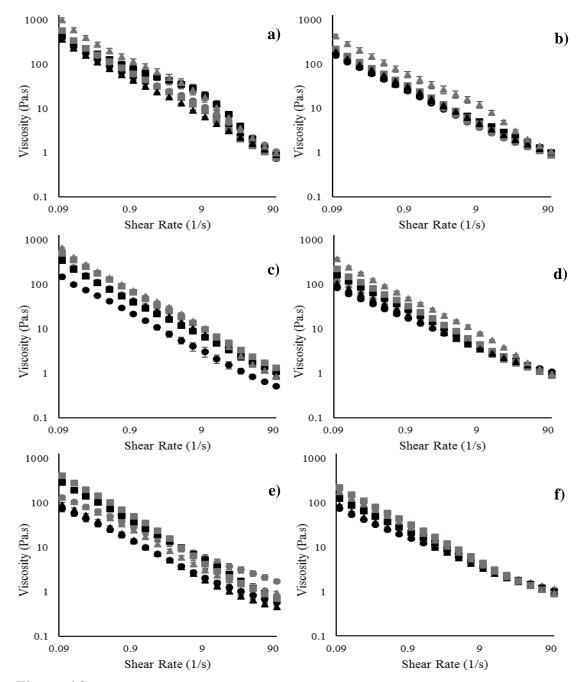


Sliding speed

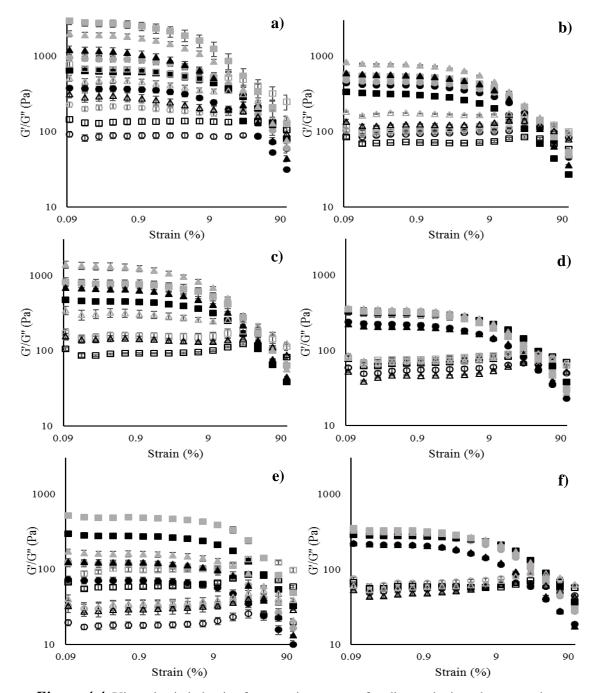
*Figure 4.1.* An ideal Stribeck curve, such as one that might be generated from testing an ideal, Newtonian fluid. The three tribological regions (boundary, mixed, and hydrodynamic) are delineated by vertical blue lines.



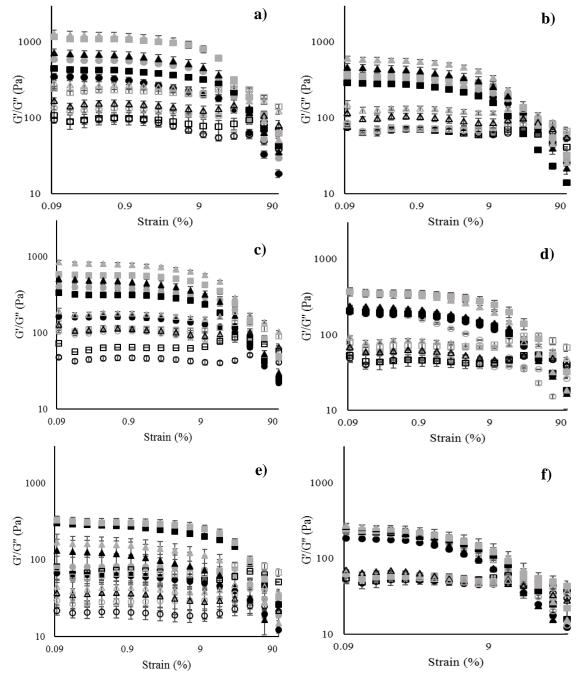
*Figure 4.2.* Viscosity profiles of sodium substituted cottage cheese cream dressing at 8°C. Graph contents in order: a) KCl, pH 4.5 b) CaCl<sub>2</sub>, pH 4.5 c) KCl, pH 5.0 d) CaCl<sub>2</sub>, pH 5.0 e) KCl, pH 5.5 f) CaCl<sub>2</sub>, pH 5.5. Circles represent a 1:3 NaCl to KCl or CaCl<sub>2</sub> substitution ratio, squares a 1:1 NaCl to KCl or CaCl<sub>2</sub> substitution ratio, and triangles a 3:1 NaCl to KCl or CaCl<sub>2</sub> ratio. Black symbols are values at day 1, gray at day 14. Error bars represent standard error.



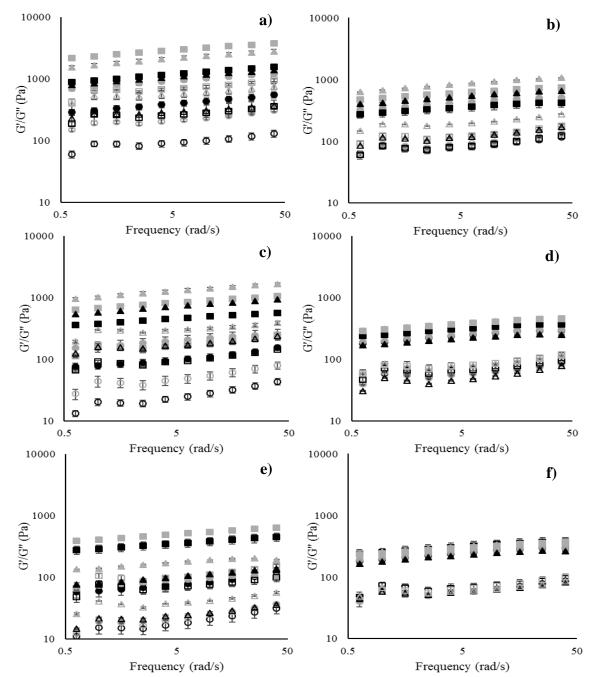
*Figure 4.3.* Viscosity profiles of sodium substituted cottage cheese cream dressing at 25°C. Graph contents in order: a) KCl, pH 4.5 b) CaCl<sub>2</sub>, pH 4.5 c) KCl, pH 5.0 d) CaCl<sub>2</sub>, pH 5.0 e) KCl, pH 5.5 f) CaCl<sub>2</sub>, pH 5.5. Circles represent a 1:3 NaCl to KCl or CaCl<sub>2</sub> substitution ratio, squares a 1:1 NaCl to KCl or CaCl<sub>2</sub> substitution ratio, and triangles a 3:1 NaCl to KCl or CaCl<sub>2</sub> ratio. Black symbols are values at day 1, gray at day 14. Error bars represent standard error.



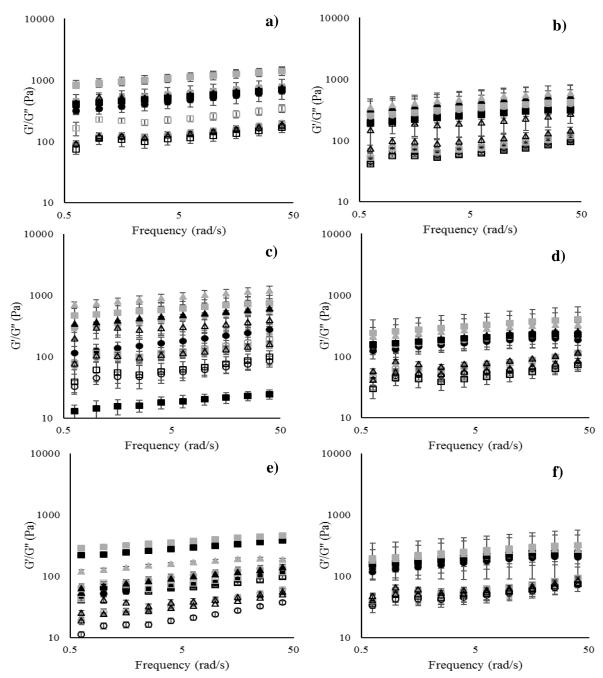
*Figure 4.4.* Viscoelastic behavior from strain sweeps of sodium substituted cottage cheese cream dressing at 8°C. Graph contents in order: a) KCl, pH 4.5 b) CaCl<sub>2</sub>, pH 4.5 c) KCl, pH 5.0 d) CaCl<sub>2</sub>, pH 5.0 e) KCl, pH 5.5 f) CaCl<sub>2</sub>, pH 5.5. Circles represent a 1:3 NaCl to KCl or CaCl<sub>2</sub> substitution ratio, squares a 1:1 NaCl to KCl or CaCl<sub>2</sub> substitution ratio, and triangles a 3:1 NaCl to KCl or CaCl<sub>2</sub> ratio. Closed symbols represent G' values, open G". Black symbols are values at day 1, gray at day 14. Error bars represent standard error



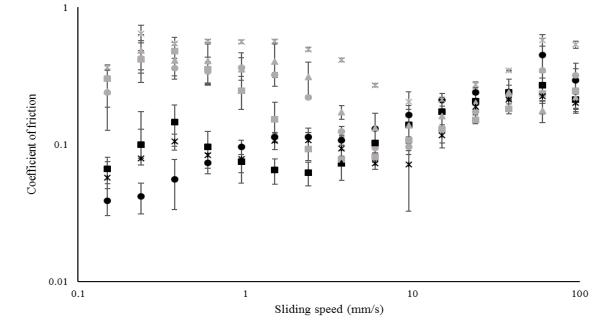
*Figure 4.5.* Viscoelastic behavior from strain sweeps of sodium substituted cottage cheese cream dressing at 25°C. Graph contents in order: a) KCl, pH 4.5 b) CaCl<sub>2</sub>, pH 4.5 c) KCl, pH 5.0 d) CaCl<sub>2</sub>, pH 5.0 e) KCl, pH 5.5 f) CaCl<sub>2</sub>, pH 5.5. Circles represent a 1:3 NaCl to KCl or CaCl<sub>2</sub> substitution ratio, squares a 1:1 NaCl to KCl or CaCl<sub>2</sub> substitution ratio, and triangles a 3:1 NaCl to KCl or CaCl<sub>2</sub> ratio. Closed symbols represent G' values, open G". Black symbols are values at day 1, gray at day 14. Error bars represent standard error.



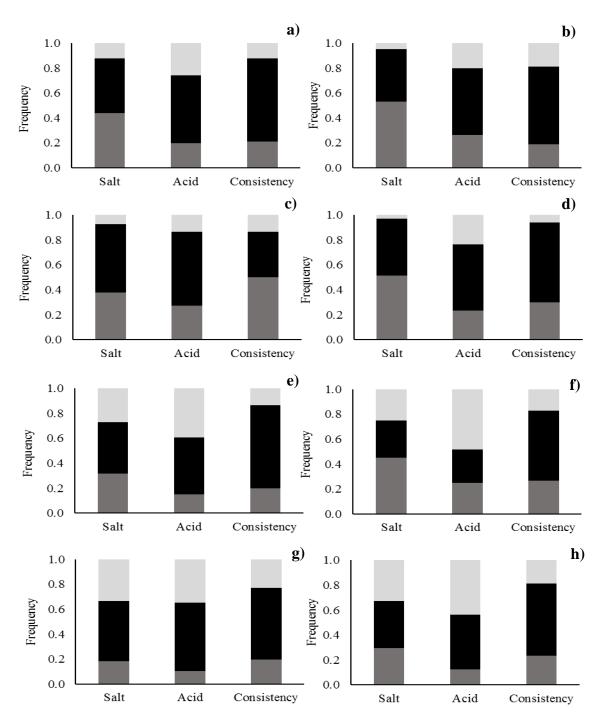
*Figure 4.6.* Viscoelastic behavior from frequency sweeps of sodium substituted cottage cheese cream dressing at 8°C. Graph contents in order: a) KCl, pH 4.5 b) CaCl<sub>2</sub>, pH 4.5 c) KCl, pH 5.0 d) CaCl<sub>2</sub>, pH 5.0 e) KCl, pH 5.5 f) CaCl<sub>2</sub>, pH 5.5 Circles represent a 1:3 NaCl to KCl or CaCl<sub>2</sub> substitution ratio, squares a 1:1 NaCl to KCl or CaCl<sub>2</sub> substitution ratio, and triangles a 3:1 NaCl to KCl or CaCl<sub>2</sub> ratio. Closed symbols represent G', open G''. Black symbols are values at day 1, gray at day 14. Error bars represent standard error.



*Figure 4.7.* Viscoelastic behavior from frequency sweeps of sodium substituted cottage cheese cream dressing at 25°C. Graph contents in order: a) KCl, pH 4.5 b) CaCl<sub>2</sub>, pH 4.5 c) KCl, pH 5.0 d) CaCl<sub>2</sub>, pH 5.0 e) KCl, pH 5.5 f) CaCl<sub>2</sub>, pH 5.5 Circles represent a 1:3 NaCl to KCl or CaCl<sub>2</sub> substitution ratio, squares a 1:1 NaCl to KCl or CaCl<sub>2</sub> substitution ratio, and triangles a 3:1 NaCl to KCl or CaCl<sub>2</sub> ratio. Closed symbols represent G', open G''. Black symbols are values at day 1, gray at day 14. Error bars represent standard error.



*Figure 4.8.* Friction curves for sodium-substituted cottage cheese dressing formulations. Circles represent the 1:1 NaCl:CaCl<sub>2</sub> formulation, squares represent the 1:3 NaCl:CaCl<sub>2</sub> formulation, triangles represent the 1:1 NaCl:KCl formulation, and asterisks represent the 1:3 NaCl:KCl formulation. Black symbols indicate coefficient of friction values at day 1, and gray symbols are day 14 values. Error bars represent standard error.



*Figure 4.9.* Just about right frequency of response data for salt, acidity, and dressing consistency. Panelist responses were grouped into three categories: too low, just about right, and too high, with the relative frequencies of each adding up to 1. The light gray portions of each bar indicate the too high category, black just-about-right, and dark gray too low. In order, the graphs represent the following: a) 1:3 NaCl:KCl at day 1 b) 1:3 NaCl:KCl dressing at day 1 d) 1:1 NaCl:KCl dressing at day 14 e) 1:3 NaCl:CaCl<sub>2</sub> at day 14 g) 1:1 NaCl:CaCl<sub>2</sub> at day 1 h) 1:1 NaCl:CaCl<sub>2</sub> at day 14.

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### **CHAPTER 5: CONCLUSIONS**

Reduction and/or replacement of salt in food products can impact not only the texture and flavor, but the rheological and tribological behaviors of the reformulated product, as evidenced in this study. Regardless of salt concentration in cottage cheese cream dressing, all samples tested were shear thinning, pseudoplastic, and displayed some weak gel behavior. Reduction and replacement of (sodium) salt in the dressing resulted in different viscosities and viscoelastic behaviors, although these differences were not always significant. pH and the presence of a monovalent salt (sodium or potassium) versus a divalent one (calcium) had the most significant impact on the results. The consumer sensory panel indicated that a simple reduction in sodium levels as well as partial substitution with KCl resulted in a product that was still well-liked. Samples that incorporated CaCl<sub>2</sub> are a partial salt replacer did not fare as well among consumers, although their rheological and tribological behaviors were fairly similar to KCl-substituted and reduced NaCl formulations.

Commercial cottage cheese dressing is generally acidified to approximately pH 5.0, and testing at 25°C is less practical in terms of immediate consumer perceptions, as cottage cheese is not served at room temperature. However, once oral processing begins, the temperature and thus the rheological behavior changes, so testing at 25°C would give a better indication of what consumers experience during oral processing. Thus, the information gathered in this study still provided data that may prove useful for future studies on dairy products. Additionally, salt (sodium) reduction in food products has been an important issue to consumers for some time, and improved understanding the role of salt in food systems is beneficial for researchers studying fundamental mechanisms of food functional behavior.

This work highlights several opportunities for future study. The role of salt in cottage cheese dressing is not only to provide flavor to the finished product. Salt is an important antimicrobial, and the high salt levels in cottage cheese dressing help inhibit microbial growth. A shelf life study or other microbial tests were not conducted over the course of this research but would be an important next step in assessing the feasibility of a reduced salt cottage cheese dressing. Microscopy work to examine structure of reduced salt dressings could also be combined with quantitative descriptive analysis, rheology, and tribology to determine structure/function/texture relationships.

The impact of bitter masking agents, such as additional flavorings in the dressing could also be explored in subsequent studies. Consumers of cottage cheese often combine it with other flavors, whether savory (e.g. olive oil and pepper) or sweet (e.g. pineapple). Such flavor enhancements would help conceal any bitter off-flavors noticed by consumers during the sensory panel while also making up for the loss of flavor associated with reducing the concentration of NaCl.

While future study is needed to fully explore the impact of salt reduction and replacement on cottage cheese cream dressing physicochemical, mechanical, and sensory properties, the results of this study were promising in terms of creating a reduced salt cottage cheese dressing. They also provided a solid foundation on which further research in this previously neglected dairy product can be conducted.

# APPENDIX A: PROTOCOL APPROVAL FROM INSTITUTIONAL REVIEW

# BOARD

	University of Idaho
	Institutional Review Board 875 Perimeter Drive, MS 301 Wascew ID 83844-301 Phone: 208-885-6160 Fax: 208-885-5750
To:	Helen S Joyner
Cc:	Hannah Damiano, MS
From:	Jennifer Walker, IRB Coordinator University of Idaho Office of Research Assurances
Date:	May 23, 2016
Title:	The Effect of Sodium Reduction on Cottage Cheese Dressing Rheology, Tribology, and Sensory Characteristics
Project:	16-014 Certified as exempt under category 6 at 45 CFR 46.101(b)(6).
On behalt	of the Institutional Review Board at the University of Idaho. Lam pleased to inform you that
the proto Rheology	of the Institutional Review Board at the University of Idaho, I am pleased to inform you that col for the research project The Effect of Sodium Reduction on Cottage Cheese Dressing Tribology, and Sensory Characteristics has been certified as exempt under the category and enumber listed above.
the proto Rheology, reference This certi are not si made to t implement populatio research,	col for the research project The Effect of Sodium Reduction on Cottage Cheese Dressing
the proto Rheology, reference This certi are not si made to t implemen populatio research, system by As Princip regulation ensure th identified Investigat	col for the research project The Effect of Sodium Reduction on Cottage Cheese Dressing , Tribology, and Sensory Characteristics has been certified as exempt under the category and number listed above. fication is valid only for the study protocol as it was submitted. Studies certified as Exempt ubject to continuing review and this certification does not expire. However, if changes are the study protocol, you must submit the changes through <u>VERAS</u> for review before thing the changes. Amendments may include but are not limited to, changes in study n, study personnel, study instruments, consent documents, recruitment materials, sites of etc. If you have any additional questions, please contact me through the VERAS messaging
the proto Rheology, reference This certi are not su made to t implemen populatio research, system by As Princip regulation ensure th identified Investigat subjects t	col for the research project The Effect of Sodium Reduction on Cottage Cheese Dressing , Tribology, and Sensory Characteristics has been certified as exempt under the category and number listed above. fication is valid only for the study protocol as it was submitted. Studies certified as Exempt abject to continuing review and this certification does not expire. However, if changes are the study protocol, you must submit the changes through <u>VERAS</u> for review before thing the changes. Amendments may include but are not limited to, changes in study n, study personnel, study instruments, consent documents, recruitment materials, sites of etc. If you have any additional questions, please contact me through the VERAS messaging vclicking the 'Reply' button. wal Investigator, you are responsible for ensuring compliance with all applicable FERPA ns, University of Idaho policies, state and federal regulations. Every effort should be made to at the project is conducted in a maner consistent with the three fundamental principles to in the Belmont Report: respect for persons; beneficence; and justice. The Principal tor is responsible for ensuring that all study personnel have completed the online human

## APPENDIX B: COTTAGE CHEESE CREAM DRESSING SENSORY BALLOT

	Welcome to our Cottage Cheese Panel!	
	Click the next button to begin	
Please enter the gue	est number listed on the top right corner of your consent form.	
Your quest number k	a configured. If the supplies displayed does not match the sum has an usual or	
form, please signal th	s confirmed. If the number displayed does not match the number on your co he experimenter.	nsent
form, please signal th	he experimenter.	insent
form, please signal th	the experimenter.	nsent
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form, please signal th What is your gende	123	nsen
form, please signal th What is your gende	I23 I23 Female	nsen
form, please signal th What is your gende	I23 I23 Female	nsen
form, please signal th What is your gende	I23 I23 I23 I23 I23 I23 I23 I24 I25	nsen
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Please indicate your race (select all that apply).

### Generated by Compusense Cloud

	White/Caucasian, European American, Non-Hispanic
Ō	Hispanic or Latino American
	American Indian or Alaskan Native
0	Asian
	Black or African American
	Middle Eastern, Middle Eastern American
	Native Hawaiian Pacific Islander
	Other

## What is your marital status?

0	Single	
0	Married	
0	Divorced	
0	Widowed	

## What is your highest level of education?

0	Some High School	
0	High School graduate	
0	Some College or Associate Degree	
	Bachelor's Degree	

Advanced or Professional Degree

# Which of the following categories best represents your employment status? (select all that apply)

0	Full time employed	
Ŭ.	Part time employed	
	Unemployed	
0	Retired	
	Homemaker/caregiver	
0	Student	

### What is your household income?

0	Less than \$19,999	
0	\$20,000 to \$49,999	
0	\$50,000 to \$79,999	
0	\$80,000 to \$99,999	
0	\$100,000 to \$149,999	
0	\$150,000 to \$199,999	
0	\$200,000 or more	
0	Prefer not to state	

How often do you consume cottage cheese?

### Generated by Compusense Cloud

O Daily
O 4-5 times a week
O 2-3 times a week
Once per week
<ul> <li>A few times a month</li> </ul>
Once per month
Rarely
O Never

You will next be asked a series of questions regarding your perception of cottage cheese samples. These questions will focus on three different components:

- Curd-- the small, semisolid particulates in the sample
- · Dressing-- the liquid portion surrounding the curds
- · Curd + dressing the sample as a whole

Press the blue button when you are ready to begin

### How much do you like sample BC111 OVERALL?

Sam pl e	: BC111
----------	---------

Dislike Extremely	Dislike Very Much	Dislike Moderately	Dislike Slightly	Neither Like nor Dislike	Like Slightly	Like Moderately Like Very Much	Like Extremely

How much do you like the overall APPEARANCE of sample BC111?

#### Sample: BC111

Dislike Extremely	Dislike Very Much	Disilke Moderately	Dislike Slightly	Neither Like nor Dislike	Like Slightly	Like Moderately Like Very Much	Like Extremely

How much do you like the overall TEXTURE of sample BC111?

4/6

Generate	i by Com	pusense	Cloud
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Sample: BC11	1						
Dislike Extremely	Dislike Very Much	Dislike Moderately	Dislike Slightly	Neither Like nor Dislike	Like Slightly	Like Moderately Like Very Much	Like Extremely
How much	do you like	e the overa	all FLAVOF	of sample	BC111?		
Sample: BC11	1						
Dislike Extremely	Dislike Very Much	Dislike Moderately	Dislike Slightly	Neither Like nor Dislike	Like Slightly	Like Moderately Like Very Much	Like Extremely
How much	do you like	e the level	of BITTER	NESS in sa	mple BC1	11?	
Sample: BC11	1						

Dislike Extremely	Dislike Very Much	Dislike Moderately	Dislike Slightly	Neither Like nor Dislike	Like Slightly	Like Moderately Like Very Much	h Like Extremely

What do you think about the level of SALTINESS in sample BC111?

### Sample: BC111

Much too little	Slightly too little	Just-about-right	Slightly too high	Much to high
1	2	3	4	5

What do you think about the level of ACIDITY in sample BC111?

Sample: BC111

Much too little	Slightly too little	Just-about-right	Slightly too high	Much too high
1	2	3	4	5

## What do you think about the DRESSING CONSISTENCY of sample BC111?

Sample: BC111

Much too thin	Slightly too thin	Just-about-right	Slightly too thick	Much too thick
1	2	3	4	5

Please tell us why you like or dislike sample BC111

Sample: BC111



Please take a few seconds to rinse your palate with water and crackers before the next sample. Signal the experimenter when you are ready to proceed by pressing the blue button.





Thank you for completing this test!

Please select the 'sign out' prompt so the log-in screen is ready for the next panelist.

Push the blue switch to receive your Ferdinand's certificate.



		Day 1	Day 14
No. of panelists		66	64
	Male	24	35
Gender	Female	41	28
	Prefer not to answer	1	1
	Average	37	39
Age	Minimum	20	18
	Maximum	74	75
	White/Caucasian	39	44
	Hispanic/Latino American	4	3
	American Indian or Alaskan Native	1	0
Race	Asian	23	18
Race	Black/African American	0	1
	Middle Eastern	0	0
	Hawaiian or Pacific Islander	0	0
	Other	2	1
	Single	36	31
Man's 1 and a	Married	58	29
Marital status	Divorced	1	1
	Widowed	1	3
	Some high school	0	0
	High school	3	4
Education	Some college or Associate degree	12	5
	Bachelor's degree	21	31
	Advanced or professional degree	30	24
	Full time employed	29	29
	Part time employed	12	10
	Unemployed	3	1
Employment status	Retired	2	6
	Homemaker/caregiver	$\frac{2}{0}$	1
	Student	23	22
	Less than \$19,999	20	18
	\$20,000-\$49,999	23	10
	\$50,000-\$79,999	7	9
	\$80,000-\$99,999	2	6
Income	\$100,000-\$\$149,999	3	7
	\$150,000-\$199,999	2	2
	\$200,000+	2	2
	Prefer not to answer	7	6
		2	
	Daily		0
	4-5 times/week	3	4
<b>E</b>	2-3 times/week	11	9
Frequency of cottage	Once a week	2	5
cheese consumption	A few times a month	12	13
	Once a month	12	11
	Rarely	23	20
	Never	1	2

# **APPENDIX C: SENSORY PANEL DEMOGRAPHICS**