HEAT-INDUCED GELATION OF ULTRAFILTERED WHOLE MILK
CONCENTRATE AND PRODUCT APPLICATIONS

by

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ABSTRACT

Heat-Induced Gelation of Ultrafiltered Whole Milk Concentrate and Product Applications

by

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The heat-induced gelation properties of ultrafiltered (UF) whole milk concentrate were studied under different physical and chemical conditions. Total solids concentration, homogenization pressures, heating temperatures, and heating times were found to have a positive correlation with gel strength. The addition of calcium chloride, sodium chloride, or trisodium citrate produced gels of higher strengths and textural properties than the gels obtained with non-salt-treated concentrate. Calcium chloride produced the strongest gels with a cheese-like texture and poor spreadability. Sodium chloride produced gels of intermediate strength with a firm, elastic texture and poor spreadability. Trisodium citrate produced the softest gels with a smooth, creamy texture and good spreadability.

A shelf stable 40% total solids UF concentrate was manufactured using ultra-high temperature (UHT) processing by direct steam injection. The pourable concentrate had a shelf life of 75 to 90 days at 23°C and did not have the ability to produce heat-induced gels after a second heating. Addition of calcium chloride, sodium chloride, or trisodium citrate restored the heat-induced gelation ability of the retentate. However, the gels were
weaker and presented different characteristics than did the gels from non-UHT-treated concentrate.

Transmission electron microscopy (TEM) studies revealed a relationship between gel firmness and gel ultrastructure of the heat-induced gels. The gels consisted of a network of casein micelles connected with strands of a less dense protein material. The tighter the network the stronger the gel strength. High heating temperatures and calcium chloride addition caused fusion of the casein micelles in the network.

Sensory evaluation of two prototype gelled desserts by a general consumer population showed a good potential for the use of the heat-induced gelation property of UF-concentrated whole milk in the development of new gelled dessert applications.

(146 pages)
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I dedicate this work to my beloved and always patient wife, Margie, without whom this manuscript might have never been completed.

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<tr>
<td>$\alpha$-La</td>
<td>$\alpha$-lactalbumin</td>
</tr>
<tr>
<td>$\beta$-Lg</td>
<td>$\beta$-lactoglobulin</td>
</tr>
<tr>
<td>DF</td>
<td>Degrees of freedom</td>
</tr>
<tr>
<td>EDTA</td>
<td>Ethylenediamine tetraacetic acid</td>
</tr>
<tr>
<td>Gs</td>
<td>Gel strength</td>
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<tr>
<td>Kpoise</td>
<td>Kilopoise</td>
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<tr>
<td>LSD</td>
<td>Least significant difference</td>
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<td>min</td>
<td>Minutes</td>
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<tr>
<td>MPa</td>
<td>Mega pascal</td>
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<tr>
<td>MSE</td>
<td>Mean square error</td>
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<tr>
<td>N</td>
<td>Population size</td>
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<tr>
<td>$M$</td>
<td>Molar</td>
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<tr>
<td>NFDM</td>
<td>Nonfat dry milk</td>
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<tr>
<td>RO</td>
<td>Reverse osmosis</td>
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<td>s</td>
<td>Standard deviation</td>
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<tr>
<td>TEM</td>
<td>Transmission electron microscopy</td>
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<tr>
<td>TPC</td>
<td>Total plate count</td>
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<tr>
<td>TS</td>
<td>Total solids</td>
</tr>
<tr>
<td>UF</td>
<td>Ultrafiltration</td>
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<td>UHT</td>
<td>Ultra-high temperature</td>
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CHAPTER 1

GENERAL INTRODUCTION

Formation of gels with different strengths and textural properties has always been an important part in the development of food products with appeal to the consumer. For this reason food scientists continue to look for novel ways to generate gels with textural properties of interest to consumers. Milk has the unique functional property to form protein-based gels by enzymatic action, acidification, or heat. These have made milk one of the most popular and widely accepted food materials. Therefore, continuous exploration of milk’s unique heat-induced gelation property could be used to increase its consumption by its application in the development of new food products.

Heat-induced milk gels made from concentrated reconstituted skim milk powder have been studied. However, the use of ultrafiltration technology has generated new concentrated milk systems that have the ability to form heat-induced gels. The textural properties of heat-induced UF concentrated whole milk gels have not been studied. The objective of this research was to characterize the heat-induced gelation properties of a concentrated milk system under different physical and chemical conditions and to evaluate its potential use in the development of new dairy-based food products.

LITERATURE REVIEW

Heat Gelation of Milk

Heat-induced milk gels. A unique functional property of milk proteins is their ability to form gels. These gels are irreversible, and are classified into enzymic, acid, and heat-induced gels. Enzymic gels are formed as a result of enzyme action (rennet) on κ-casein, which destabilizes and aggregates the casein micelles in the presence of calcium ions (92, 119). Acid gels are formed by the acid fermentation or direct acidification of milk to the isoelectric point of casein (pH 4.6). Heat-induced gels are formed by heat
destabilization of the milk protein system as it occurs in condensed, evaporated, and ultra-high temperature (UHT) milks (22, 48, 96, 103, 110, 151).

Heat-induced gelation of concentrated milk systems has been considered undesirable in milk processing, and many efforts have been directed towards its understanding and prevention (4, 95, 111, 114). However, the unique properties of heat-induced gels offer the potential for the development of new and unconventional dairy products (74, 77).

Heat-induced milk gels are complex because they consist of caseins, whey proteins, and other components such as lactose and mineral ions (74, 76). They possess specific properties that differentiate them from other protein gels such as fibrin, gelatin, gluten, or soy protein gels (27, 44). Unlike gelatin gels, milk gels are irreversible, have low elasticity, and require a higher concentration of protein as compared with fibrin gels (44).

**Physical factors influencing gel firmness.** Firmness of heat-induced milk gels is controlled by physical factors including temperature and duration of heating, temperature at time of testing, and protein concentration (74, 145).

The effects of temperature and duration of heating are closely related to the heat transfer and the size and shape of heat-induced gels. Gelation occurs from the outer layers inwards. Therefore, large gels can present soft and sticky centers with outer layers of good consistency if short heating times are used. Prolonged or excessive heating can produce browning in the outer layers (77) and decreases in the protein efficiency ratio and available lysine, without affecting the overall nutritional value (72). Gel firmness is positively correlated to heating temperature and time, and negatively correlated to testing temperature (74).

Kalab et al. (74) prepared heat-induced milk gels by heating aqueous suspensions of 40 to 60% NFDM at 80 to 115°C for 10 to 30 min. Temperatures below 80°C for up
to 30 min did not induce gelation in suspensions equal to or less than 50% NFDM. Gel firmness increased with heating temperature, reaching a maximum at 100°C and declining above this temperature. Gel firmness depended markedly on the test temperature, indicating that the gels were held by weak hydrogen and salt bonds between protein particles (73). An 80% decrease in relative firmness was observed when temperature during testing increased from 10 to 60°C (74).

Kalab et al. showed that gel firmness has an exponential (73, 74) and hyperbolic dependence (74) on concentration at 40 to 60% total solids of NFDM gels. Gels are not formed from total solid concentrations of 30% or less at a given heat treatment (73). A 10% firmness change was observed with a 1% change in total solids above and below 50%.

Chemical factors influencing gel firmness. Firmness and consistency of heat-induced milk gels are affected by various chemical agents (51, 73, 75). Heat gelation of milk proteins is closely related to the spontaneous gelation of sterilized concentrated milk systems and involves changes in hydrogen and hydrophobic bonds, salt and disulfide linkages, and calcium bridges between protein molecules (110). Therefore, compounds reacting with calcium, phosphate, amino groups, sulphydryl groups, or disulfide linkages can be expected to modify the firmness and consistency of heat-induced milk gels (73).

Kalab and Emmons (75) studied several chemical factors influencing the firmness of heat-induced milk gels and found that calcium and other divalent cations promote gelation. Calcium-binding anions, such as citrate and EDTA, prevent gelation (4) by stabilizing the milk proteins and producing soft and sticky gels (51, 75). Sulphydryl-blocking agents retard gelation, whereas disulfide-reducing agents promote it (110). Oxidizing and reducing agents can increase the firmness of heat-induced gels (75, 110). Crosslinking agents, such as formaldehyde and glutaraldehyde, can increase the gel firmness at low concentrations, but can decrease it at high concentrations (75).
Microstructure of heat-induced gels. Electron microscopy has been used to study milk gels (57, 76). An increase in the casein micelle size was observed in heated milk (24) with coalescence of the micelles preceding gelation (11, 132).

Kalab and Harwalkar (76) used scanning and transmission electron microscopy to study heat-induced milk gels of concentrated aqueous suspensions of NFDM. The gels consisted of a network of aggregated casein micelle strands cross-linked with denatured β-lactoglobulin. Gels with 40 and 50% total solids presented casein micelles as individual entities linked by some bridging material; however, at 60% total solids the micelles were fused; producing gels of increased firmness. Disulfide bridging of the gel structure was involved since addition of sulphydryl-blocking agents reduced the gel strength (71). A close correlation between gel microstructure and firmness was found. The addition of additives such as CaCl₂ and ammonium persulphate caused the micelles to fuse, increasing the firmness of the gels, but the addition of hexametaphosphate, a gelation inhibitor, produced micelle disintegration and a decrease of gel firmness (76).

Ultrafiltration

Ultrafiltration (UF) is a continuous molecular separation process in which a pressure gradient across a semipermeable membrane forces solvent and small molecular weight species through the pores of the membrane (permeate), while larger molecular weight compounds are retained (retentate) (26).

Ultrafiltration appeared as a feasible separation technique in the early 1960's, but it was not until 1969 when Maubois, Macquot, and Vassal proposed its use in cheesemaking (97). Today, ultrafiltration is an accepted operation in the dairy industry, and it is used mainly in the fractionation of cheese whey and in the pre-concentration of milk for cheese manufacture (50).
**Milk ultrafiltration.** Milk ultrafiltration is a fractionation process in which milk is separated in two parts at temperatures between 50 and 55°C and pressures of 0.10 and 1.05 Kg/cm² using membranes with pore size of 0.001 to 0.02 microns (146). The concentrated milk fraction called "retentate" contains most of the protein and fat (42, 49, 50, 53) and has been used to manufacture hard (7), soft (148), and semi-soft (113) cheeses. The filtrate fraction commonly called "permeate" is essentially a waste material containing water, lactose, salts, vitamins, and a small amount of organic and nitrogenous material (42, 49, 50, 53).

The ultrafiltration process does not change serum casein, Ca, and P content of the casein micelle (87), or the size distribution of casein micelles (53). Fat, vitamin B12, and folic acid are completely retained by the membrane (25, 53). Protein is mostly retained, but some α-lactalbumin (α-La) and β-lactoglobulin (β-Lg) can pass the ultrafiltration membrane (9, 121). Lactose, water soluble vitamins, and most minerals are partially retained (53). Increased permeation of lactose and minerals can be achieved by combining ultrafiltration with diafiltration. Up to 99% of the lactose can be removed because of its high water solubility and relatively low molecular weight (87). However, only 36% of Ca and 42% of P can be removed because of their association with the casein micelles (87, 155).

**UF milk retentate.** UF milk retentate is a unique concentrated dairy medium because it is concentrated at low temperatures, and its final composition differs from concentrated milks prepared by evaporation, distillation, and crystallization. UF concentrated skim milk is more heat stable than evaporated milk and is not greatly affected by forewarming or addition of food grade stabilizers (137, 138). Temperature of -8°C produces flocculation of 3X UF concentrated milk after 1 to 3 wk of storage (41, 86).

The properties of UF milk retentate are affected mainly by its whey protein content because of the heat sensitivity of β-Lg and its role in the heat-induced gelation
phenomenon (39, 40). Addition of β-Lg to skim milk retentate destabilizes the system during heating by increasing the proportion of α-La in the α-La-β-Lg-k-casein gelation complex, but addition of sulfydryl blocking agents decreases it (39, 108, 109). A positive correlation exists between the casein:whey protein ratio and the recovery of α-La and β-Lg after heating (40).

In general, increased whey protein denaturation is observed when processing temperatures of milk are raised (112). However, whey protein denaturation is affected more by milk concentration than by temperature (99, 108, 109). Sweetser and Muir (138) reported that whey protein denaturation in skim milk was not significantly influenced by solids content up to 30% at heating temperatures of 65 and 82°C for 30 min, but at 71 and 76°C whey protein denaturation decreased with increased milk concentration. Yousif (156) reported a greater whey protein denaturation in concentrated milks as compared with unconcentrated milks, as well as greater whey protein denaturation in whole milk than in skim milk.

**UF dairy products.** Ultrafiltration technology has provided to the milk industry the potential of new dairy products prepared from the retentate and permeate fractions. Initially, the use of this technology was focused in the production of many different cheeses from the liquid precheese concentrate, including Cheddar cheese (29, 52, 135), Cottage cheese (96), cream cheese (28), mozzarella cheese (29), Colby/Brick cheese (21), and process cheese (42, 134). However, limited success was obtained for most of these products possibly due to the presence of whey proteins, which can affect the body, texture, and flavor profiles of the final products (42, 129, 134). Although the gelation of whey proteins is considered a defect in most of these products, such gelation can be used to an advantage in creating new products such as gelled dairy desserts (59, 60).

Today new product opportunities utilizing UF technology comprise numerous categories (65) including: natural and process cheeses (7, 113, 148), cheese bases (42),
butter-like spreads (153), milk beverages (82), coffee creamers (70), yogurts (141), frozen desserts (146), whey protein concentrates (64), and value-added products from permeates (142).

**Heat-Induced Changes on Milk Proteins**

Heating is one of the most fundamental processes in the food industry and is invariably used in the processing of milk and milk products. The unique chemical properties of milk and milk products influence their heat processing characteristics. It is, therefore, important to understand the effects of heat on the milk protein system.

*Protein denaturation*. Denaturation is defined as a drastic change from the native conformation that does not alter the amino acid sequence of the protein (140).

Heat treatments affect mainly hydrophobic bonds. For this reason, such treatments will be more important in those milk proteins that have large hydrophobicities (35, 120).

Heat denaturation of whey proteins is a phenomenon that includes two different processes. First, unfolding of the protein occurs; second, aggregation follows. Unfolding is usually a reversible process if heating is stopped before aggregation begins; aggregation is normally an irreversible process following unfolding (33, 34). Protein unfolding and aggregation behave differently with respect to heating, pH, protein concentration, and concentration of salts or other denaturing substances (12). Although denaturation can occur by the individual action of each one of these factors, they are interrelated and one of them cannot be studied without considering the others. Susceptibility to denaturation is largely determined by pH, and extent of aggregation is more dependent on the presence of calcium ions (33).

*Caseins*. The caseins constitute about 80% of the milk proteins and are found primarily as a colloidal dispersion of complexes called micelles. The casein micelle proteins
are α_{1}-, α_{2}-, β-, γ-, and κ-caseins, and they are found in approximate concentrations of 10.0, 2.6, 9.3, 0.8, and 3.3 g/kg in milk (147).

α_{41}-Casein is the principal component of α_{4}-casein. It has a highly charged, highly solvated region that contains several phosphorylated serine residues. Phosphoserine is negatively charged and binds calcium ions at pH > 6 (46, 136). Self-association of α_{41}-casein depends markedly on its concentration and on the pH, ionic strength, and kind of ion in the medium, but it is relatively independent of temperature. It aggregates and precipitates at very low concentrations of calcium (147).

α_{2}-Casein is the most hydrophilic of the caseins. It has two disulfide bonds that can interact with the disulfide bonds of β-Lg when a severe heat treatment is applied (136). Addition of this casein to milk systems can reduce heat stability of casein micelles (84). α_{4}-Caseins are especially sensitive to the calcium concentration because of their high phosphorylation levels and small amounts of secondary and tertiary structure (79).

β-Casein is a very hydrophobic protein and is temperature sensitive. Heating milk moves β-casein from serum into the micelles; cooling removes β-casein from micelles (37). Low temperature or removal of calcium causes dissociation of β-casein away from the micelle causing a destabilization of the remaining micelle (23, 31, 32).

γ-Casein is a group of caseins that correspond to C-terminal portions of the β-casein sequence. These are formed by cleavage of β-casein at different positions by the enzyme plasmin. The smaller fractions resulting from the cleavage appear in the whey when casein is precipitated by acid and constitute part of what has long been designated the proteose-peptone fraction of the whey (46, 147).

In solution, κ-Casein consists of a long chain polymer probably held together by intermolecular disulfide bonds. Two-thirds of the κ-casein molecule (the N-terminal) is hydrophobic and contains two disulfide bonds that can form cross-links with the disulfide bonds of β-Lg after a severe heat treatment. The remaining C-terminal end is hydrophilic,
polar, and charged (94). \( \kappa \)-Casein binds about 2 moles of \( \text{Ca}^{2+} \) per mole of protein at neutral pH but differs markedly from the other caseins in its solubility over a wide range of \( \text{Ca}^{2+} \) concentrations (147).

**Whey proteins.** The serum proteins are basically a mixture of \( \beta \)-Lg, \( \alpha \)-La, bovine serum albumin, and immunoglobulins. These are more heat sensitive and less calcium sensitive than caseins (79).

\( \beta \)-Lactoglobulin is the most prominent sulfhydryl-containing milk protein. It is a globular protein and is a dimer of two identical monomers under normal milk storage conditions (\(<4^\circ\text{C and pH 5-7}\) (35). Each monomer has two disulfide bridges and one free thiol group. The free thiol groups have the ability to interact with \( \kappa \)-casein and other proteins during heating (12). -Lactoglobulin is the least denaturable of the serum proteins with a denaturation temperature of \( 78^\circ\text{C} \) (12). It exhibits a second thermal change near \( 140^\circ\text{C} \) caused by breakdown of the disulfide bonds and additional unfolding of the molecule (33, 149). The denaturation of \( \beta \)-Lg is of second order with respect to time (91, 94). \( \beta \)-Lactoglobulin undergoes denaturation and aggregation as a result of reactivation of -SH groups by heat (100). The aggregates are of two sizes: small aggregates of \( \beta \)-Lg (3.7 S) with interlinking -SH groups, and larger aggregates of \( \beta \)-Lg (29 S) in which the formation of disulfide (S-S) bonds may be important (90, 100, 130).

\( \alpha \)-Lactalbumin is the least stable of whey proteins with denaturation temperature of \( 62^\circ\text{C} \), but it requires the largest amount of heat per gram for unfolding (12). It has four disulfide bridges between eight cysteine residues (35). Heating causes a reversible conformational change related to the disulfide bridges (91). Denaturation of \( \alpha \)-La is slower at pH 4 than at pH 6 or 9 (58). The denaturation of \( \alpha \)-La is of first order with respect to time (94).

Bovine serum albumin appears to be the most easily denatured serum protein since its denaturation is not as reversible as that of other serum proteins (35). The denaturation
temperature is 64°C (12). It precipitates between 40 to 50°C as a result of hydrophobicity-directed unfolding (85, 93). Some of the serum protein can remain undenatured after prolonged heating at 65°C as a result of the protection of the native proteins by some of the already denatured serum protein (143). Milk immunoglobulins are very heat labile especially below pH 6 (35).

**Protein interactions.** The properties of gels produced by heating concentrated suspensions of NFDM have been analyzed by rheological and electron microscopic techniques (76, 145). The gels consist of a network of aggregated casein micelle strands that are cross-linked with denatured β-Lg. Disulfide bridging of the gel structure is apparently involved since the addition of sulphydryl-blocking agents can reduce the gel strength (62, 75). Lower polymerization rates of the denatured whey proteins occurs in milk treated with SH-blocking agents (62). Casein molecules can crosslink with each other through amino groups when milk is heated to 120°C (88).

Heat denatured κ-casein and β-Lg interact through disulfide linkages, or by sulphydryl-disulfide interchange to form a protein complex (81, 130). This complex formation is favored by calcium salts since the sensitivity of serum proteins to calcium increases with severe heat treatments (12).

α₂-casein forms disulfide bridges when heated with β-Lg and can interfere with the ability of κ-casein to bind to β-Lg (79, 84). The total amount of whey proteins attached to casein increases as heat treatment is intensified, but the ratio of whey proteins attached remains constant (43).

Direct interaction between β-Lg and κ-casein occurs, but is reduced if the presence of α-La (6, 38, 39). Direct interactions between α-La and κ-casein are limited (55). However, interactions between α-La and κ-casein occur through the interaction of κ-casein with the complex formed between α-La and β-Lg (38, 66).
The degree of denaturation of β-Lg is not affected by the presence of α-La, but the presence of casein facilitates the formation of α-La and β-Lg complexes that interact with κ-casein (39, 40). Also, the degree of denaturation of α-La is greater when heated with β-Lg than when heated alone, and the effect is more pronounced at temperatures from 70 to 85°C, and pH between 6.4 and 7.2 (12).

**Factors affecting interactions.** Heat-denaturation reactions of milk proteins are mainly dependent on pH, salt balance, preheating, and milk concentration (12).

**pH.** Heat stability of milk shows a pH dependency. Milk can be classified as type A or type B depending on its heat stability response to changes in pH (12). Type A milk heat stability curve presents a maximum stability when adjusted to pH 6.7 and a minimum stability at pH 6.8. Type B milk does not present a minimum or a maximum in the heat stability-pH curve. Instead, it increases its heat stability as pH is increased from 6.2 to 7.4 (12, 46, 125, 126). Type A milk coagulates by a two-step mechanism while type B milk coagulates by a one-step mechanism (115, 139). Temperature affects the shape of these curves (46, 68, 139). Type A milk can be converted into type B milk by using higher temperatures (139). The minimum in the coagulation time-versus-pH curve of type A milk can be eliminated by adding κ-casein, thereby converting it into type B milk. In the same way, type B milk can be converted to type A by salting out some κ-casein or by adding β-lactoglobulin (144). At low pH, whey proteins denature onto micelles linking them together. At high pH, coagulation occurs only when the casein in the micelles has changed enough to allow crosslinking between neighboring micelles (30).

**Salts.** Concentrations of soluble and ionic calcium and phosphate are reduced during heating of the milk. When cooled, the milk becomes unsaturated with respect to these two salts (127). Therefore, the addition of appropriate phosphate or citrate salts can increase the heat stability of milk or concentrated milk. Added salts must move the milk along the coagulation time-versus-pH curve up to a stable point (137) to increase the heat
stability of the milk. Indiscriminate addition of buffers to milk can easily move past the proper point (12).

**Preheating.** Preheating increases the stability of proteins in milk if applied before further processing (109). Preheating relates to the ability of heat-denatured bovine serum albumin to protect native serum albumin from denaturation by additional heating (143). Precipitation of whey proteins on casein micelle surfaces occurs by sulphhydril reactions with κ-casein, which prevents later coagulation by diminishing the number of κ-casein sites available for clotting (118).

**Concentration.** Increased concentration of milk systems generally retards the apparent heat-induced denaturation of proteins (152), but decreases the general stability of the complex protein system of milk and whey (46, 108, 138). The effects of concentration are different for the individual proteins in milk. α-Lactalbumin is more easily denatured as concentration increases; β-Lg is less easily denatured with increased solids concentration in milk (59). Milk concentrated to 3X flocculates after 1 to 3 wk at -8°C (41, 86), and milk concentrated to 5X will produce a reversible gel at refrigeration temperatures.

Other substances in milk can also affect the heat stability of the system. The addition of lactose, glucose, and urea individually or in combination can increase the heat stability of milk (33, 83, 108, 122, 156).

**Ultra-High Temperature Processing of Milk**

Food sterilization can be defined as the total destruction of living microorganisms and their spores for the purpose of giving extended or unlimited shelf life without refrigerated storage to food aseptically packaged in a hermetically sealed container (20, 61, 147). In a strict microbiological sense absolute sterility cannot be obtained by any heat treatment process, since a treated product properly incubated will almost certainly show a portion of spoilage (20). The more proper term "commercial sterility," introduced
by the food industry, is used to refer to a product free from microorganisms that can develop under the normal conditions of handling and storage (20, 61).

The practice of sterilization started in the nineteenth century with the sterilization of closed containers by Nicholas Appert. By 1860 Louis Pasteur had successfully sterilized milk by heating it to 125°C at a pressure of 1.5 atm (61). It was not until the beginning of the twentieth century that commercial sterilized milk was produced. Since then, numerous advances in the heat sterilization of milk have been made (17, 18, 20).

Sterilization can be accomplished using a combination of different temperatures and times, 105 to 120°C for 10 to 30 min autoclaving, 130 to 140°C for 2 to 30 s, or 145 to 150°C for 1 to 2 s depending on the number and type of spores present (8, 20, 101, 147).

UHT sterilization is a continuous process in which the milk is heated by direct steam injection or by indirect heating in a plate, tubular, or scraped-surface heat exchanger to 120 to 150°C for one to several seconds. This is followed by cooling and aseptic filling of containers to prolong the shelf life of milk with a minimal change to other characteristics (19, 61, 106).

The International Dairy Federation considers that sterilized milk must remain stable and show no sign of bacterial development after incubation at two temperatures: 30 ± 1°C for 14 d, and 55 ± 1°C for 7 d (61).

*Types of UHT systems.* UHT systems can be divided into two major groups according to the heat-exchange method applied. In the first group, the product is heated indirectly in a plate, tubular, or scraped-surface heat exchanger; in the second, the product is heated directly by steam injection or infusion.

In indirect heating systems, the product and the heating medium are separated by a heat-conducting barrier. This requires a longer time to bring the product to ultra-high temperatures and, therefore, causes more product deterioration than direct steam UHT
processing (20). It can also cause product "burn on" in the heat exchanger, which reduces the heat transfer (47).

In the direct heating systems, the product is heated by injecting steam into milk or by infusing milk into steam. The condensing steam provides almost instantaneous distribution of heat throughout the product with the ultra-high temperature being reached in a fraction of a second. This makes directly heated UHT milk more susceptible to age gelation than its indirectly heated counterpart due to the lack of complete inactivation of milk and microbial proteinases (106). The product is then held at sterilization temperature for the required time before being cooled in a vacuum evaporator to remove the added water (106).

Direct and indirect UHT-processed milks can be distinguished from each other on the basis of their lactulose and hydromethylfurfural contents. These two substances have been used to detect the severity of a given milk heat treatment (20).

Changes of milk at ultra-high temperatures. Numerous physical, chemical, and biochemical changes occur in milk when heated at ultra-high sterilization temperatures. Some of these changes are easily recognized by the consumer (color and flavor), while others (nutritional, protein structure, etc.) are more subtle and difficult to detect due to their chemical nature. Many of these changes involve more than one of the milk constituents and are directly influenced by the time-temperature conditions of the sterilization process (20). Overall, however, the amount of chemical changes is less for UHT processing than it is for in-container sterilization for the same sporicidal effect (20).

Color changes. A whitening effect is observed in UHT milk and milk products compared with their unprocessed counterparts (15, 63, 78). A reversible whitening of milk occurs in the temperature range of 5 to 50°C. This change is very slight and can be measured only by monitoring changes in milk color reflectance between 400 nm and 700 nm of the visible spectrum (16). Burton (14) suggested that the changes may arise from
the migration of calcium into the micelle with increase in temperature, causing an increase in micelle size and an increase in light scattering. At temperatures above 60°C the whitening of milk becomes irreversible, increasing with time and temperature, and reaching its higher levels with higher temperatures (15). The denaturation and association of whey proteins with casein micelles is responsible for this irreversible change (20).

With increased severity of heating, milk becomes browner as a consequence of the Maillard reaction in which the ε-amino group of lysine condensates with the carbonyl group of lactose (118) to ultimately produce brown pigments or melanoidins. It has been reported that the degree of browning is a function of time and temperature and that the rate of browning increases with pH (13, 63, 78). Browning takes place in the nonfat part of the milk causing a more intense color in non-fat milks than in fat-containing milks treated under the same heating conditions (20).

**Flavor changes.** Two main flavor changes occur in milk during UHT processing. The first, called "heated flavor," occurs when milk is heated to temperatures above 70°C, which denatures the whey proteins, particularly β-Lg. It is associated with the liberation of free -SH groups arising from the denatured β-Lg (20, 89). The free -SH groups can be oxidized to the volatile hydrogen sulfide, which gives freshly heated milk its characteristic smell (20, 67).

At temperatures above 90°C the level of free -SH groups begins to fall (89) and a new flavor develops. This is called "sterilized flavor" and is characteristic of sterilized milk and milk products (20). This flavor is stable and increases in intensity during prolonged storage, even at room temperature, to become what is described as "stale" (3, 20, 106). Over 45 different compounds from both proteins and fat may contribute to the "UHT" flavor and have been identified (5, 69, 98, 117, 131). Some compounds are diacetyl, lactones, alcohol ketones, maltol, vanillin, benzaldehyde, and acetophenone. However, it is difficult to assess how significant a single compound can be (20, 101).
Protein changes. Electron microscopy studies have shown that sterilization of milk causes an increase in the casein micelle size (47), which is produced by the denaturation and aggregation of serum proteins onto the casein micelle (30, 47, 104, 128) and by the shift in location of calcium phosphate (48). Serum proteins are denatured to a greater or lesser extent depending on the severity of the heating process (20). Directly heated systems produce a 50 to 75% denaturation of the serum proteins while indirect systems and in-container sterilization produce 70 to 90% and 80 to 100% denaturation, respectively (123). β-Lactoglobulin complexes specifically with κ-casein (130). However, it has also been reported that α-La complexes with β-Lg at temperatures below 90°C (6) and at ultra-high temperatures (102). Melo and Hansen (102) isolated a protein complex from UHT milk consisting of β-Lg with α₂-casein and κ-casein. β-Lactoglobulin (30 to 40%) is denatured at relatively mild UHT processes, while up to 90% of denaturation can occur at the top severity range of UHT process (140°C for 30 s) (20).

Enzymatic changes. Raw milk contains numerous enzymes from natural and microbial sources. Naturally occurring enzymes as alkaline phosphatase, catalase, lipase, xanthine oxidase, peroxidase, and acid phosphatase are inactivated by comparatively mild heat treatments (118). However, alkaline phosphatase, which is inactivated at pasteurization temperatures, can be reactivated if short-time treatments at higher temperatures are used depending on storage temperature (154). Microbial enzymes, such as lipases and proteases, are much more heat resistant (1) and can survive UHT processes leading to off-flavor development and age gelation during storage (20, 56, 133). Microbial enzymes have very small Q₁₀ values (1), which means that as the processing temperature is raised, the survival of the enzyme increases for the same sterilizing performance. The probability of enzymic deterioration during storage of the processed product increases with increasing temperature of heat treatment (20). The microbiological quality of the milk before UHT will therefore determine the final quality of the UHT milk.
product. Pilot-scale laboratory experiments have shown that the use of preholding times of 1 h at 55°C before UHT treatments reduces gelation and bitter flavors caused by microbial enzymes (150).

**Nutritional changes.** Severely heated milks suffer a loss of available lysine as a result of the Maillard reaction. However, typical UHT treatments have little or no adverse effect on milk proteins’ digestibility and nutritional quality as measured in rat feeding trials (45, 124).

In general, vitamins are more stable under UHT processing than with pasteurization (17). These thermostable vitamins include A, D, E, and β-carotene as well as the vitamins of the B complex, riboflavin, nicotinic acid, pantothenic acid, and biotin (45). Significant losses of riboflavin and ascorbic acid of UHT milk during prolonged storage have been observed (101). UHT processing of milk by direct heating at temperatures of 120 to 145°C with holding times of 2 to 32 s produces a thiamin loss up to 22%, while processing by indirect heating produces losses up to 29% (10). Although free calcium is reduced, availability of calcium does not change in direct steam, UHT-processed milk (101). Cysteine, cystine, and methionine concentrations are reduced by about 34% by UHT processing (54), while a decrease of less than 10% of chemically available lysine has been observed (36). When milk is heated, lysine becomes unavailable through the formation of the modified amino acid, lysinoalanine (2) and through the formation of the compound lactulosyl-lysine, an intermediate compound of the Maillard reaction (20).

**Concentrated UHT milk.** Concentrated UHT milk has not been commercially adopted in the United States. However, the manufacture of RO-UHT milk concentrate has been suggested by Kocak (80).

Milk can be concentrated before sterilization using vacuum evaporation (116) or membrane separation (80). Processing requires forewarming of the concentrated milk
before UHT sterilization to stabilize proteins and prevent age gelation during storage (105, 107, 116). Aseptic homogenization after sterilization is also necessary to stabilize the milk fat emulsion against separation during storage and to provide added heat stability of the protein system (106). Heat denaturation of protein solutions is generally retarded by concentration. However, increasing total milk solids lowers both the heat and age gelation stability of the concentrate (106, 116). Addition of sodium or potassium polyphosphate before sterilization is very effective in preventing age gelation in concentrated UHT milk (116).

**OBJECTIVES**

The main objective of this research was to characterize the heat-induced gelation of UF concentrated whole milk under different physical and chemical conditions and to evaluate its potential application in the development of gelled dairy desserts. The specific research objectives were:

1. To determine the effects of total solids, homogenization pressure, heating temperature, heating time, and the addition of calcium chloride, trisodium citrate, and sodium chloride on the gel strength and textural properties of heat-induced gels made from UF concentrated whole milk.

2. To determine the feasibility of manufacturing a pourable shelf stable ultrafiltered whole milk concentrate that could be used as an ingredient for the development of new gelled dairy products.

3. To determine the effects of homogenization pressure, heating temperature, heating time, and the addition of calcium chloride, trisodium citrate, and sodium chloride on the gel strength and textural properties of heat-induced gels made from shelf stable whole milk retentate.
4. To determine the relationship between gel strength and gel ultrastructure of UF concentrated and UF concentrated UHT treated 40% TS whole milk gels, under different physical and chemical conditions using transmission electron microscopy (TEM).

5. To evaluate the use of the heat-induced gelation property of UF concentrated whole milk systems in dairy desserts applications, by conducting a sensory evaluation study of two concept product applications in a general consumer population.

REFERENCES


CHAPTER 2

PHYSICAL AND CHEMICAL FACTORS INFLUENCING THE GEL STRENGTH OF HEAT-INDUCED ULTRAFILTERED CONCENTRATED WHOLE MILK GELS

ABSTRACT

Physical and chemical factors influencing the heat-induced gelation of ultrafiltered concentrated whole milk were studied. Whole milk was UF concentrated to 40% total solids using a spiral wound (20,000 mol wt cutoff) polysulfone membrane system. The retentate was two-stage homogenized at 6.68, 20.68, or 34.47 MPa (1000, 3000, or 5000 psi) with the second stage at 15% of total pressure. Milk concentrate samples (150 ml) were treated with 0.1 $M$ or 0.3 $M$ calcium chloride or sodium chloride, and 0.03 $M$ or 0.15 $M$ trisodium citrate, placed in thin-walled aluminum cans, and heat treated in a conventional electric oven at 135°C or 190°C for 10, 20, or 30 min. The samples were cooled to room temperature, and the gel strength was measured using a penetrometer. Significant differences in gel strength and gel texture among the treatments were found. In general, higher homogenization pressures, heating temperatures, and heating times resulted in higher gel strength. Addition of any of the salts studied produced gels with higher strength than the no-salt control gels but with different textural properties. Increased salt concentrations produced an increase in gel strength in gels treated with trisodium citrate, a decrease in gels treated with calcium chloride, and no effect in gels treated with sodium chloride. Trisodium citrate and sodium chloride produced gels of similar strength, with trisodium citrate producing more spreadable, sticky gels, and with sodium chloride, firmer and less spreadable gel textures. Calcium chloride produced substantially stronger gels with syneresis, a brittle structure, and poor spreadability. The
INTRODUCTION

Milk is one of the most popular and widely accepted food materials and has the unique functional property to form gels, which is important in the development of food products. These gels, such as yogurt and cheese, are irreversible and can be formed by enzymatic action on the protein structure or by acidification resulting from microbial fermentation or direct acid addition. Ultrafiltration technology has been used to produce unique concentrated milk systems due to the retention of undenatured whey proteins at the low concentration temperatures (50°C maximum) (2, 3, 6). These and other concentrated milk systems have the property of forming gels when heated (17, 23, 28, 33). This heat-induced gelation is considered undesirable in milk processing, and many efforts have been directed towards its understanding and prevention (1, 25). However, it has also been suggested (16, 17) that heat-induced milk gels have some properties that can be used to produce unconventional products such as gelled dairy desserts (13).

Heat-induced milk gels consist of a complex mixture of caseins, whey proteins, and other components such as lactose and mineral ions (18, 20) and generally require higher concentrations of proteins as compared with other protein gels (7, 29). One of the main mechanisms involved in the gelation process is the formation of disulfide bridging between milk proteins (15), particularly between β-Lg and α-La with κ-casein (14, 23, 27) on the surface of the casein micelles and with the fat globule membrane proteins in whole milk systems (22, 28, 30). These protein bridges produce a tridimensional network of casein micelles connected through strands of denatured whey proteins (18). However, the heat-induced gelation of concentrated milk systems cannot be explained solely by disulfide linkage formations (12, 22) since other factors such as milk salt system balance (11),

observed results can be used to produce gels of different textures with possible new dairy dessert applications.
hydrogen bonding (33), and hydrophobic interactions (5, 21, 24, 29) have been found to play a contributing role in the formation of heat-induced milk gels.

Kalab and Harwalkar (16), and Kalab and Emmons (18) studied the physical and chemical factors influencing the firmness of heat-induced gels made from concentrated aqueous suspensions of nonfat dry milk. The firmness of the gels depended on protein concentration, temperature and time of heating, and on temperature of testing. Addition of various agents markedly affected the properties of such gels. Calcium and other divalent cations promoted gelation, and calcium-binding anions produced softer and stickier gels (17, 18). Oxidizing and reducing agents produced up to five times firmer gels than did the control treatments. Sulfhydryls produced softer gels at low concentration, but at concentrations greater than 70 mM firmness increased several fold above the standard gels. The physico-chemical parameters that affect the gelation of ultrafiltered whole milk concentrate have not been studied.

**OBJECTIVE**

The objective of this study was to determine the effects of total solids; homogenization pressure; heating temperature; heating time; and the addition of calcium chloride, trisodium citrate, and sodium chloride on the gel strength and textural properties of heat-induced gels made from UF concentrated whole milk.

**MATERIALS AND METHODS**

**Milk and Gel Samples Preparation**

Raw whole milk from the Utah State University dairy farm was collected and vat pasteurized at 63°C for 30 min. The milk was UF concentrated to 40% total solids using a spiral wound (20,000 mol wt cutoff) polysulfone membrane system (Osmonics, Inc., Minnetonka MN). Two-stage homogenization of the milk was performed at 6.68, 20.68,
or 34.47 MPa (1000, 3000, or 5000 psi), with the second stage at 15% of total pressure. Salt solutions were prepared by dissolving the needed amount of salt in a minimum amount of permeate and then mixing with 1000 ml of retentate to obtain a final salt concentration of 0.1 or 0.3 M for sodium chloride and calcium chloride, and 0.03 or 0.15 M for trisodium citrate. The samples were allowed to stabilize for 20 min at room temperature before the heat treatment was applied. One hundred fifty-gram portions of the retentates were placed in standard soft drink aluminum containers (0.15 mm thickness, 6.5 cm diameter, 12 oz capacity), that were previously cut to approximately 8 cm in height, and heated in a conventional electric oven at 135°C or 190°C for 10, 20, or 30 min. The gels were cooled overnight (10 h) before measuring the gel strength (Figure 2.1). The 40% TS untreated retentate was diluted to 39, 37, 35, and 33% total solids using permeate obtained from the last part of the ultrafiltration process to conduct the study of concentration effect on gel strength. The rest of the study was conducted with 40% TS retentate only.

**Measurement of Gel Strength**

Gel strength, defined as the maximum penetration force (g) through the milk gel samples, was measured using a Utah State University penetrometer (4) with a 15.5-mm cylindrical probe descending at a constant speed of 1 cm/min into the center of the gels. Gel strength was measured after the gels had cooled overnight at 22°C. The top of each gel (3 mm) was cut off to eliminate any crust formed during heating, and discarded before measuring gel strength.

**Textural Properties**

Subjective observations of the body, texture, and spreadability characteristics of the different gels were made and reported as textural properties (Tables 2.1 and 2.2).
GEL PREPARATION

Raw Whole Milk

Vat Pasteurization
63°C, 30 min

UF Concentration
40% TS

Homogenization
6.7, 20.7, or 34.5 MPa

UF Retentate - Salt Solutions
0.1 or 0.3 M for CaCl₂ and NaCl
0.03 or 0.15 M for Na₃Citrate

Predissolved Salts
CaCl₂, NaCl, Na₃Citrate

Convection Oven
135 or 190°C
10, 20, or 30 min

Overnight Cooling

Gel Strength Measurement

Figure 2.1. Schematic diagram of UF concentrated milk gels preparation.
TABLE 2.1. Effect of salt treatments on textural properties of heat-induced UF concentrated 40% TS whole milk gels.

<table>
<thead>
<tr>
<th>Salt</th>
<th>Body</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Salt</td>
<td>Compact, soft</td>
<td>Rubbery, poor spreadability</td>
</tr>
<tr>
<td>NaCl</td>
<td>Compact, firm</td>
<td>Elastic, poor spreadability</td>
</tr>
<tr>
<td>Na₃Citrate</td>
<td>Smooth, soft</td>
<td>Creamy, sticky, good spreadability</td>
</tr>
<tr>
<td>CaCl₂</td>
<td>Hard, Cheese</td>
<td>Porous, crumbly when spread, Syneresis present</td>
</tr>
</tbody>
</table>

TABLE 2.2. Effect of physical factors on textural properties of heat-induced UF concentrated 40% TS whole milk gels.

<table>
<thead>
<tr>
<th>Factor</th>
<th>Body</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low Homogenization</td>
<td>Compact, soft</td>
<td>Elastic, good spreadability</td>
</tr>
<tr>
<td>High Homogenization</td>
<td>Compact, firm</td>
<td>Elastic, spreadable</td>
</tr>
<tr>
<td>Low Heating Temp.</td>
<td>Smooth, soft</td>
<td>Creamy, good spreadability</td>
</tr>
<tr>
<td>High Heating Temp.</td>
<td>Compact, Firm</td>
<td>Rubbery, poor spreadability</td>
</tr>
<tr>
<td>Low Heating Time</td>
<td>Very soft</td>
<td>Creamy, good spreadability</td>
</tr>
<tr>
<td>High Heating Time</td>
<td>Compact, firm</td>
<td>Rubbery, poor spreadability</td>
</tr>
</tbody>
</table>
Statistical Analysis

A 3x3x2x3x3x2 completely randomized block factorial design was used. The experiment was replicated three times. Homogenization, salt type, salt concentration, temperature, and time were used as main factors. The following linear model was used:

\[ Y_{ijklmn} = \mu + B_i + H_j + T_k + M_l + S_m + C_n + HT_{jk} + HM_{jl} + HS_{jm} + HC_{jn} + TM_{kl} + TS_{km} + TC_{kn} + MS_{lm} + MC_{ln} + SC_{mn} + HTM_{jkl} + HTS_{jkm} + HTC_{jkn} + HMS_{jlm} + HMC_{jln} + HSC_{jmn} + TMS_{klm} + TMC_{kln} + TSC_{kmn} + MSC_{lnm} + HTSC_{jklmn} + HTMSC_{jklmn} + e_{ijklmn} \]

where \( Y_{ijklmn} \) is the dependent variable (gel strength), \( \mu \) is the overall mean of the population, and independent variables \( B_i, H_j, T_k, M_l, S_m, \) and \( C_n \) are the coefficients for the averages of block, homogenization pressure, heating temperature, heating time, salt type, and salt concentration effects. Two-way interactions (\( HT_{jk}, HM_{jl}, HS_{jm}, HC_{jn}, TM_{kl}, TS_{km}, TC_{kn}, MS_{lm}, MC_{ln}, SC_{mn} \)), three-way interactions (\( HTM_{jkl}, HTS_{jkm}, HTC_{jkn}, HMS_{jlm}, HMC_{jln}, HSC_{jmn}, TMS_{klm}, TMC_{kln}, TSC_{kmn}, MSC_{lnm} \)), four-way interactions (\( HTSC_{jklmn}, HMSC_{jln}, HTMS_{jklm}, HTMC_{jkln}, TMSC_{klmn} \)), and five-way interaction (\( HTMSC_{jklmn} \)) are included. The experimental error \( e_{ijklmn} \) was assumed to be normal and independently distributed.

Analysis of variance was performed using FCTCVR factorial statistical software (Utah State University, Logan UT). Treatment means were separated using Fisher’s protected LSD (32).

RESULTS AND DISCUSSION

All variables were statistically significant (Table 2.3) showing multiple two- and three-way interactions.
TABLE 2.3. Analysis of variance for treatment effects on gel strength of heat-induced UF concentrated 40% TS whole milk gels.

<table>
<thead>
<tr>
<th>SOURCE</th>
<th>DF</th>
<th>MS</th>
<th>F</th>
</tr>
</thead>
<tbody>
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<td>Block</td>
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<tr>
<td>Homogenization (H)</td>
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<tr>
<td>Time (M)</td>
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<td>1487.54</td>
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<tr>
<td>Salt (S)</td>
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<td>812.63</td>
</tr>
<tr>
<td>Salt Concentration (C)</td>
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<td>6.33</td>
</tr>
<tr>
<td>H*T</td>
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<td>72155.22</td>
<td>16.87</td>
</tr>
<tr>
<td>H*M</td>
<td>4</td>
<td>115767.66</td>
<td>27.07</td>
</tr>
<tr>
<td>H*S</td>
<td>4</td>
<td>3464.78</td>
<td>0.81</td>
</tr>
<tr>
<td>H*C</td>
<td>2</td>
<td>12962.93</td>
<td>3.03</td>
</tr>
<tr>
<td>T*M</td>
<td>2</td>
<td>660471.55</td>
<td>154.43</td>
</tr>
<tr>
<td>T*S</td>
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<td>29.89</td>
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<td>T*C</td>
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<td>M*S</td>
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<tr>
<td>M*C</td>
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<td>0.61</td>
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<td>S*C</td>
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<td>H<em>T</em>M</td>
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<td>70094.24</td>
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<td>H<em>T</em>S</td>
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<td>4130.25</td>
<td>0.97</td>
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<td>7710.01</td>
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<tr>
<td>H<em>M</em>C</td>
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<td>8821.45</td>
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</tr>
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<td>H<em>S</em>C</td>
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<td>6574.36</td>
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<tr>
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<td>17440.55</td>
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<tr>
<td>T<em>M</em>C</td>
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<td>24054.23</td>
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<tr>
<td>T<em>S</em>C</td>
<td>2</td>
<td>33958.74</td>
<td>7.94</td>
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<tr>
<td>M<em>S</em>C</td>
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<td>62353.27</td>
<td>14.58</td>
</tr>
<tr>
<td>H<em>T</em>S*C</td>
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<td>4736.76</td>
<td>1.11</td>
</tr>
<tr>
<td>H<em>M</em>S*C</td>
<td>8</td>
<td>2862.31</td>
<td>0.67</td>
</tr>
<tr>
<td>H<em>T</em>M*S</td>
<td>8</td>
<td>11429.17</td>
<td>2.67</td>
</tr>
<tr>
<td>H<em>T</em>M*C</td>
<td>4</td>
<td>13303.22</td>
<td>3.11</td>
</tr>
<tr>
<td>T<em>M</em>S*C</td>
<td>4</td>
<td>27481.29</td>
<td>6.43</td>
</tr>
<tr>
<td>H<em>T</em>M<em>S</em>C</td>
<td>8</td>
<td>11676.62</td>
<td>2.73</td>
</tr>
<tr>
<td>Error</td>
<td>250</td>
<td>4276.75</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>377</td>
<td>80802.15</td>
<td></td>
</tr>
</tbody>
</table>

NS  Nonsignificant
*  Significant \( p < 0.05 \)
**  Significant \( p < 0.01 \)
Effect of Total Milk Solids Concentration

Gel strength increased nonlinearly between 35 to 43% total milk solids, increasing more rapidly at higher levels of total solids (Figure 2.2). A gel was not obtained at and below 33% TS under the heating conditions and sample size studied. Gel strength of gels above 43% TS could not be evaluated due to the concentration limitations of the UF system. However, there is a tendency to approach an upper value asymptotically. A similar behavior in heat-induced milk gels made from nonfat dry milk was described by Kalab et al. (19). A typical curve for gels formed by heating at 190.6°C for 25 min is presented in Figure 2.2. Nonlinear regression was used to fit several mathematical models to the experimental data and to describe the relationship between gel strength (Gs) and total solids concentration (TS). The best fit was obtained with a polynomial equation of third degree, followed by a hyperbolic function of the kind $G_s = \frac{TS}{a-bTS} + c$, where $a$, $b$, and $c$ are numerical constants. Even though the concentration limitations of the UF system fit the behavior of the hyperbolic function better, it is unlikely that the value of the gel strength will reach a very large value at the maximum solids level of the membrane. Rather, a high but finite value can be predicted better by a polynomial function. Following are the fitted equations with their corresponding sum square deviations used as the criterion for best fit:

Polynomial:

$G_s = -41723 + 3555.1 \, TS - 101.27 \, TS^2 + 0.96521 \, TS^3 \quad \text{SSE} = 249.01$

Hyperbolic:

$G_s = \frac{TS}{0.84237 - 0.01831 \, TS} - 144.1685 \quad \text{SSE} = 944.06$
Small variations in the total solids at higher solids concentration have a substantial impact on the final firmness of the whole milk gels as proved experimentally.

**Effect of Heating Temperature and Heating Time**

In general, gel strength increased with increasing heating temperatures and heating times (Figure 2.3). However, at the longest heating times (30 min) there was no statistically significant difference between the lower and higher heating temperatures (Figure 2.4). This was mainly due to a salt interaction effect at high calcium chloride concentration levels. The observed heating temperature and time effects were directly correlated to the level of whey protein denaturation that plays a role in the formation of
Figure 2.3. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to main effects: homogenization level (LSD_{0.01} = 21.22), heating temperature (LSD_{0.01} = 17.33), heating time (LSD_{0.01} = 21.22), and salt addition (LSD_{0.01} = 28.08). Means represent the average of the main effect across the other effects. Values with the same superscript letter within the same main effect are not significantly different at the LSD_{0.01} indicated.
Figure 2.4. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to heating temperature and heating time differences, averaging on homogenization level, salt addition, and salt concentration. Values with the same superscript letter (a, b, c) between heating times and superscript letter (x, y) between heating temperatures are not significantly different (LSD_{0.01} = 30.02).
the gels as reported by several researchers (27, 31). The greater the whey protein denaturation the stronger the three-dimensional gel structure.

Effect of Homogenization Pressure

Overall, gel strength increased with higher homogenization pressures (Figure 2.3). This positive correlation was most clear at the higher heating temperatures while at lower heating temperatures the correlation was only evident at the longer heating times (Figures 2.5 and 2.6). With low heating times (10 min), homogenization level had no impact on the gel strength at all heating temperatures, while at intermediate heating times (20 min) the homogenization effect was observed only at higher homogenization pressures (Figures 2.6 and 2.7). The impact of low heating times could be explained by the incomplete denaturation of the whey proteins that produced a weak tridimensional gel network (11, 14).

Increasing gel strength caused by higher homogenization pressures was evident across all salt type additions (Figure 2.8), most particularly at the higher heating temperature (Figure 2.9). At the lower heating temperature the positive homogenization effect was only observed with the control and sodium chloride treatments (Figure 2.9).

The increase of gel strength at higher homogenization levels could be explained by the increased number of smaller fat globules, present under high homogenization pressures, that may allow for a denser network of denatured proteins that reinforces the gel structure. Larger globule sizes, on the other hand, would produce larger, more compressible fat pockets in the gel matrix that would be reflected as more elastic gels with lower gel strengths. Heat-induced effects on whole milk proteins under nonreducing and reducing conditions were studied by Kim (22). It was determined that β-Lg and other milk serum proteins interact readily with milk fat globule membrane and membrane proteins. The degree of contribution to the gel strength of UF concentrated whole milk gels by
Figure 2.5. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to homogenization level and heating temperature differences, averaging on salt addition, salt concentration, and heating time. Values with the same superscript letter (a, b, c) between homogenization levels and superscript letter (x, y) between heating temperatures are not significantly different (LSD$_{0.01} = 30.02$).
Figure 2.6. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to homogenization level, heating temperature, and heating time differences, averaging on salt addition and salt concentration. Values with the same superscript letter (a, b, c, d) between homogenization level and heating temperature and with the same superscript letter (x, y, z) between heating times are not significantly different ($\text{LSD}_{0.05} = 39.56$).
Figure 2.7. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to homogenization level and heating time differences, with averaging on heating temperature, salt type, and salt concentration. Values with the same superscript letter (a, b, c) between homogenization levels and with the same superscript letter (x, y, z) between heating times are not significantly different (LSD_{0.01} = 36.76).
Figure 2.8. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to homogenization level and salt addition differences, with averaging on heating time, heating temperature, and salt concentration. Values with the same superscript letter (a, b, c, d) between salt type are not significantly different (LSD$_{0.05}$ = 30.21 for salt and LSD$_{0.05}$ = 37.0 for no salt). Values with the same superscript letter (x, y, z) between homogenization levels are not significantly different (LSD$_{0.05}$ = 30.21 for salt and LSD$_{0.05}$ = 42.73 for no salt).
Figure 2.9. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to homogenization level, salt addition, and heating temperature differences, with averaging on heating time and salt concentration. Values with the same superscript letter (a, b, c, d, e) between homogenization level and heating temperature are not significantly different (LSD$_{0.05}$ = 42.73 for salt type and LSD$_{0.05}$ = 52.33 for no salt). Values with the same superscript letter (w, x, y, z) between salt type are not significantly different (LSD$_{0.05}$ = 42.73 for salt type and LSD$_{0.05}$ = 62.42 for no salt).
these interactions is unclear under the experimental conditions of this study, and we can only assume that the mechanical effect of homogenization overshadowed any contributing effect by the fat globule membrane proteins.

**Effect of Salt Type and Concentration**

Addition of salts produced gel strengths higher than gel strengths of the controls (Figure 2.3). Calcium chloride produced the strongest gels at all heating temperatures and times, higher than all the other salt treatments (Figure 2.10). At low temperatures sodium chloride and trisodium citrate produced gels of similar strength, but at high temperatures sodium chloride produced gels of higher strength than trisodium citrate produced (Figures 2.10 and 2.12). Trisodium citrate and sodium chloride produced gels of strength similar to the controls heated at the lowest heating time at both high and low temperatures. At intermediate heating times they produce gels of similar strength only at the lower heating temperatures (Figures 2.10 and 2.11).

Higher concentrations of calcium chloride produced weaker gels than the lower concentrations when higher heating temperatures were applied (Figures 2.13, 2.14, and 2.15). This was probably caused by the existence of small open pockets in the gels, causing a sponginess effect when the gel strength was measured. Presence of syneresis was also observed in the samples with the higher calcium chloride treatments. Similar observations have been made by other researchers in heat-induced gelation of concentrated milk systems (18) and milk stability studies (9, 26). The ability of divalent cations, such as calcium, to accelerate the onset of gelation and to increase the gel strength of heat-induced milk gels results from their ability to form salt bridges between casein micelles creating a stronger tridimensional protein matrix. Excess levels of calcium can produce a shrinkage of the matrix, causing the expulsion of excess liquid known as syneresis. In general, calcium chloride addition produced strong and crumbly gels with
Figure 2.10. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to salt addition, heating temperature, and heating time differences, averaging on homogenization level and salt concentration. Values with the same superscript letter (a, b, c, d, e) between temperature and heating times are not significantly different (LSD$_{0.05}$ = 42.73 for salt type and LSD$_{0.05}$ = 52.33 for no salt). Values with the same superscript letter (w, x, y, z) between salt type are not significantly different (LSD$_{0.05}$ = 42.73 for salt type and LSD$_{0.05}$ = 60.42 for no salt).
Figure 2.11. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to salt addition and heating time differences, averaging on homogenization level, heating temperature, and salt concentration. Values with the same superscript letter (a, b, c, d) between salt type are not significantly different (LSD_{0.05} = 30.21 for salt and LSD_{0.01} = 37.05 for no salt) and superscript letter (x, y, z) between heating times are not significantly different (LSD_{0.01} = 37.05 for salt and LSD_{0.05} = 42.73 for no salt).
Figure 2.12. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to salt addition and heating temperature differences, with averaging on homogenization level, heating time, and salt concentration. Values with the same superscript letter (a, b, c, d) between salt type are not significantly different (LSD$_{0.05}$ = 24.67 for salt and LSD$_{0.05}$ = 30.21 for no salt). Values with the same superscript letter (x, y) between heating temperatures are not significantly different (LSD$_{0.05}$ = 24.67 for salt and LSD$_{0.05}$ = 34.89 for no salt).
Fig 2.13. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to salt addition and salt concentration differences, averaging on homogenization level, heating temperature, and heating time. Values with the same superscript letter (a, b, c) between salt type and superscript letter (x, y, z) between salt concentration are not significantly different ($\text{LSD}_{0.01} = 32.42$).
Figure 2.14. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to heating temperature, salt type, and salt concentration differences, with averaging on heating time and homogenization level. Values with the same superscript letter (a, b, c, d, e) between heating temperature and salt concentration and with the same superscript letter (x, y, z) between salt type are not significantly different (LSD_{0.05} = 34.89). Salt concentration (high = 0.3 M for NaCl and CaCl₂ and 0.15 M for Na₃Citrate, low = 0.1 M for NaCl and CaCl₂ and 0.03 M for Na₃Citrate).
Figure 2.15. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to heating time, salt type, and salt concentration differences, with averaging on homogenization level and heating temperature. Values with the same superscript letter (a, b, c, d, e, f, g) between heating times and salt concentration and with the same superscript letter (x, y, z) between salt types are not significantly different (LSD$_{0.05}$ = 42.73). Salt concentration (high = 0.3 $M$ for NaCl and CaCl$_2$ and 0.15 $M$ for Na$_3$Citrate, low = 0.1 $M$ for NaCl and CaCl$_2$ and 0.03 $M$ for Na$_3$Citrate).
extremely poor spreadability, particularly at the higher concentration levels, and also produced high levels of syneresis.

Sodium chloride gels were stronger than the controls but showed no difference in gel strength between the low and high salt concentrations (Figures 2.13, 2.14, and 2.15). The gels were generally firm, elastic, and with poor spreadability (Table 2.2). The addition of this neutral salt resulted in increased interactions between casein particles as evidenced by the increased gel strengths obtained. These results differ from previous work from Kalab et al. (18) who reported that low concentrations, up to 0.25 M, of sodium chloride did not significantly affect gel firmness of heat-induced milk gels made from nonfat dry milk. However, their study does not offer any statistical analysis of the results to support their claim of whether their reported 23% firmness difference between the sodium chloride-treated gel and the controls is not significant as compared to a higher percentage firmness difference. Added sodium chloride may affect interactions between casein particles by influencing hydrophobic interactions or hydration forces or by inducing protein conformational changes (10).

Trisodium citrate addition caused higher gel strengths only at the higher concentration levels. The gels were generally smoother and more spreadable than the other treatments, even at the lower salt levels that presented a similar gel strength than the controls (Figures 2.13, 2.14, and 2.15). These findings contradict the observations of other researchers who have reported a decrease in gel strength in heat-induced gels from concentrated skim milk systems at normal (18) and low pH levels (10). These results are supported by the findings of other researchers indicating that critical low concentrations of calcium binding salts will stabilize milk, while higher concentrations will actually increase gelation (1, 8). The observed behavior in trisodium citrate gels is related to this salt’s ability to change the micellar calcium phosphate by chelation of calcium ions (26). At low concentration levels trisodium citrate produces a heat stabilization effect, but above a
critical limit it actually increases gelation by increasing the viscosity of the retentate and interfering with the formation of a more structured tridimensional network of denatured proteins, which in turn will account for the observed increased spreadability (Table 2.2). This supports the concept that milk salt balance plays a role in heat-induced gelation of concentrated milk systems (11).

CONCLUSION

The functional property of UF concentrated whole milk of producing heat-induced gels can be obtained at concentrations above 35% TS and can be optimized with a combination of factors such as homogenization levels, temperature and heating times, and the addition of salts. The use of trisodium citrate, low homogenization pressures, low heating temperatures, and short heating times produced smoother, softer gels with good spreadability that could be used for the development of products such as pudding and spreads. The use of calcium chloride, high homogenization pressures, high heating temperatures, and long heating times produced firmer gels that could be used to develop products such as cheesecake.

REFERENCES


CHAPTER 3

PHYSICAL AND CHEMICAL FACTORS INFLUENCING THE GEL STRENGTH OF HEAT-INDUCED MILK GELS MADE FROM SHELF-STABLE 40% TS ULTRAFILTERED WHOLE MILK RETENTATE

ABSTRACT

Physical and chemical factors influencing the firmness and texture of heat-induced milk gels made from shelf-stable ultrafiltered whole milk retentate were studied. Whole milk was UF concentrated to 40% total solids using a spiral wound (20,000 mol wt cutoff) polysulfone membrane system. Ultra-high temperature processing by direct steam injection of the retentate was conducted in an Alfa-Laval Sterilab™ pilot plant with down-stream two-stage homogenization at 6.68, 20.68, or 34.47 MPa (1000, 3000, or 5000 psi) with the second stage at 15% of total pressure. Shelf-stable milk retentate was collected in pre-sterilized plastic cups and stored at room temperature for shelf-life studies. UHT-treated milk concentrate samples (150 ml) were treated with 0.1 M or 0.3 M calcium and sodium chloride, and 0.03 M or 0.15 M trisodium citrate, placed in thin-walled aluminum cans, and heat treated in a conventional electric oven at 135°C or 190°C for 10, 20, or 30 min. The samples were cooled to room temperature, and the gel strength was measured using a penetrometer. Significant differences in gel strength and gel texture among the treatments were found. The UHT-treated retentate had a shelf-life of 75 to 90 d. The shelf-stable retentate did not gel after a second heat treatment in the convection electric oven regardless of heating temperatures or heating times applied. The addition of any of the salts studied restored the ability of the retentate to produce a heat-induced gel and produced gels of different textural properties. Salt-treated samples had higher gel strengths at higher heating temperatures and heating times. Higher gel strengths were
found at intermediate homogenization pressures, and the lowest gel strengths were found at the highest homogenization pressures. Increased salt concentration produced an increase in gel strength in all salts used. Calcium chloride produced the strongest gels with some syneresis, a brittle structure, and poor spreadability. Sodium chloride gels presented gels of intermediate strength with firm body but good spreadability. Trisodium citrate gels presented the lowest gel strengths with very soft sticky gels and smooth spreadability. The observed results can be used to manufacture a pourable shelf-stable retentate that can later be used to produce gels of different textures by the addition of different salts in various dairy dessert applications.

**INTRODUCTION**

Preliminary work had shown that heat-induced milk gels made from concentrated skim milk powder (12, 14, 16) and UF concentrated whole milk may have properties that could be exploited in the development of new and unconventional dairy-based food products (10). In order to use these heat-gelling concentrated milk systems in a food application, it is necessary to have them in a shelf-stable form that is readily available for their use. A liquid form of these concentrates will require the use of thermal processing coupled with aseptic packaging in order to deliver a shelf-stable product.

To manufacture a shelf-stable concentrated milk system, with the concentration levels necessary for this heat gelation to occur, it is necessary to first overcome the tendency of these systems to gel during thermal processing and second to prevent the closely related spontaneous incipient gelation that occurs in concentrated milk systems during storage (2, 3, 6, 19, 20, 28).

Heat-induced gelation of concentrated milk systems is mainly dependent on temperature and duration of heating (14, 27) but other chemical and physical factors also play a role (7, 13, 14). The main mechanism consists of the disulfide bridging formations
between the milk proteins β-Lg, α-La, and κ-casein (9, 11, 18, 23). Other mechanisms are also involved, including changes in hydrogen and hydrophobic bonds (5, 12, 27), and salt and calcium bridges between protein molecules (8, 19). Therefore, calcium-binding salts will tend to prevent gelation by stabilizing the milk proteins and producing soft and sticky gels (7, 13) while calcium salt additions will tend to accelerate gelation and produce harder gels (13).

**OBJECTIVES**

The objectives of this study were:

1. To determine the feasibility of manufacturing a pourable shelf-stable ultrafiltered whole milk concentrate that could be used as an ingredient for the development of new gelled dairy products.

2. To determine the effects of homogenization pressure, heating temperature, heating time, and the addition of calcium chloride, trisodium citrate, and sodium chloride on the gel strength and textural properties of heat-induced gels made from shelf-stable whole milk retentate, that could be used to produce a wide range of textural properties in new gelled dairy products.

**MATERIALS AND METHODS**

**Milk and Shelf-Stable Retentate Preparation**

Whole milk from the Utah State University dairy farm was vat pasteurized at 63°C for 30 min. The pasteurized milk was UF concentrated to 40% TS via a spiral wound (20,000 mol wt cutoff) polysulfone membrane system (Osmonics, Inc., Minnetonka MN) between 46 to 48°C. Ultra-high temperature processing of the retentate was conducted in
an Alfa-Laval Sterilab™ pilot plant (Alfa-Laval, Lund, Sweden) using direct steam injection with preheating at 75 ± 1°C for 30 s, sterilization at 141 ± 1°C for 4 s, and flash cooling to 72 ± 1°C in a vacuum flash evaporator. Two-stage down-stream aseptic homogenization was done at 6.68, 20.68, or 34.47 MPa with the second stage at 15% of total pressure. A final cooling of the sterile retentate to 60 ± 1°C was performed before aseptic collection in 120-ml presterilized plastic screw cap containers (Fisher Labware, St. Louis, MO) using a Stericab™ hyperfiltered unit (Alfa-Laval, Lund, Sweden) under positive pressure conditions to prevent post-contamination of the samples. The sterile retentate samples were stored at room temperature (25 ± 1°C) for the duration of the shelf-life or for a minimum of 20 d before the heat-induced gelation studies. Ultrafiltered permeate was treated under the same UHT conditions than the retentate and stored at room temperature until required for the salt treatments.

**Product Sterility**

Product sterility was determined by total plate count method (22) on each sample in duplicate before any other measurements were performed. Liquid samples of UHT retentate (1 to 2 ml) were diluted 1:1 in sterile water before plating. Plates were incubated at room temperature (25 ± 1°C) for 72 h before counting.

**Retentate Shelf-Life**

Shelf-life of the sterile retentate was established as the point at which age gelation occurred. Viscosity of the retentate was monitored using a Brookfield LVT viscometer (Brookfield Engineering Laboratories, Stoughton, MA) every 15 d. Onset of spontaneous gelation was arbitrarily defined as the point at which microbial free samples presented a 100% increase in viscosity with reference to the 0 d viscosity control. Microbial analysis by total plate count was used to verify the absence of microbial activity.
Analytical Tests

**pH.** pH was measured on each sample before heat-induced gelation or viscosity studies using a standard glass electrode potentiometer (Model 811, Orion Research, Inc., Cambridge, MA).

**Total solids.** Total solids of each sample were determined by microwave oven (AVC™-80, CEM Corporation, Indian Trial, NC) method (AOAC 977.11) (1).

**Fat.** Fat content was determined by the Babcock fat test (AOAC 989.04) (1).

**Total protein.** Total protein content was determined using the semi-micro Kjeldahl (Labconco Rapid Steam II Kjeldahl system, Kansas City, MO) method (AOAC 960.52) (1). A factor of 6.38 was used to convert percent nitrogen to total percent protein.

All analytical tests were performed in duplicate.

Gel Samples Preparation

Salt solutions were prepared by dissolving the needed amount of salt in a minimum amount of UHT treated permeate and mixing with 1000 ml of shelf-stable retentate to obtain a final salt concentration of 0.1 or 0.3 M for sodium chloride and calcium chloride, and 0.03 or 0.15 M for trisodium citrate. The samples were allowed to stabilize for 20 min before the heat treatment. One hundred fifty-gram portions of the retentates were placed in standard soft drink aluminum containers (0.15 mm thickness, 6.5 cm diameter, 12 oz capacity), that were previously cut to approximately 8 cm in height, and heated in a conventional electric oven at 135°C or 190°C for 10, 20, or 30 min. The gels were cooled overnight (10 h) before measuring the gel strength (Figure 3.1).

Measurement of Gel Strength

Gel strength, defined as the maximum penetration force (g) through the milk gel samples, was measured using a Utah State University penetrometer (4) with a 15.5-mm
GEL PREPARATION

1. Raw Whole Milk
2. Vat Pasteurization
   63°C, 30 min
3. UF Concentration
   40% TS
4. Homogenization
   6.7, 20.7, or 34.5 MPa
5. UHT Direct Steam Sterilization
   141°C, 4 sec
6. UF Retentate - Salt solutions
   0.1 or 0.3 M for CaCl₂ and NaCl
   0.03 or 0.15 M for Na₃Citrate
7. Convection Oven
   135 or 190°C
   10, 20, or 30 min
8. Overnight Cooling
9. Gel Strength Measurement

Figure 3.1. Schematic diagram of UF concentrated UHT-treated milk gels preparation.
cylindrical probe descending at a constant speed of 1 cm/min into the center of the gels. Gel strength was measured after the gels cooled overnight at 22°C. The top of the gels (3 mm) was cut off to eliminate any crust formed during heating, and discarded before reading.

**Textural Properties**

Subjective observations of the body, texture, and spreadability characteristics of the different gels were made and reported as textural properties.

**Statistical Analysis**

A 2x3x2x3x3x2 split plot completely randomized block factorial design was used. The experiment was replicated twice. Homogenization, salt type, salt concentration, temperature, and time were used as main factors. The following linear model was used:

\[
Y_{ijklmn} = \mu + B_i + H_j + T_k + M_l + S_m + C_n + HT_{jk} + HM_{jl} + HS_{jm} + HC_{jn} + TM_{kl} \\
+ TS_{km} + TC_{kn} + MS_{lm} + MC_{ln} + SC_{mn} + HTM_{jkl} + HTS_{jkm} + HTC_{jkn} + HMS_{jlm} \\
+ HMC_{jln} + HSC_{jmn} + TMS_{klm} + TMC_{kln} + TSC_{kmn} + MSC_{lmm} + HTSC_{jkmn} \\
+ HMSC_{jlnm} + HTMS_{jklm} + HTMC_{jkln} + TMSC_{klmn} + HTMSC_{jklmn} + e_{ijklmn}
\]

where \( Y_{ijklmn} \) is the dependent variable (gel strength), \( \mu \) is the overall mean of the population, and independent variables \( B_i, H_j, T_k, M_l, S_m, \) and \( C_n \) are the coefficients for the averages of block, homogenization pressure, heating temperature, heating time, salt type, and salt concentration effects. Two-way interactions (\( HT_{jk} , HM_{jl} , HS_{jm} , HC_{jn} , TM_{kl} , TS_{km} , TC_{kn} , MS_{lm} , MC_{ln} , SC_{mn} \)), three-way interactions (\( HTM_{jkl} , HTS_{jkm} , HTC_{jkn} , HMS_{jln} , HMC_{jln} , HSC_{jmn} , TMS_{klm} , TMC_{kln} , TSC_{kmn} , MSC_{lmm} \)), four-way interactions (\( HTSC_{jkmn} , HMSC_{jlnm} , HTMS_{jklm} , HTMC_{jkln} , TMSC_{klmn} \)), and five-way interactions
(HTMSC_{ijklmn}) are included. The plot error e_{ij} and the experimental error e_{ijklmn} were assumed to be normal and independently distributed.

Analysis of variance was performed using FCTCVR factorial statistical software (Utah State University, Logan UT). Treatment means were separated using Fisher's protected LSD (26).

**RESULTS AND DISCUSSION**

All variables were statistically significant (Table 3.1) showing multiple two- and three-way interactions.

**Shelf-Life of UHT Retentate**

Viscosity studies showed that the shelf-life of UHT-treated 40% TS retentate was between 75 to 90 d after which gelation occurred (Figure 3.2). The gelation observed is most likely an age gelation since no microbial activity was detected (Table 3.2).

**Effect of Temperature and Heating Time**

Heat-induced gelation did not occur in all non-salt-treated samples at all temperatures and heating times regardless of homogenization level (Figures 3.3, 3.4, and 3.5).

In salt-treated samples, gel strength increased with increasing heating temperatures and heating times (Figure 3.6). However, at the longest heating time (30 min) the higher heating temperature produced lower gel strengths than the lower heating temperature (Figure 3.7). This was mainly due to a salt interaction effect at high calcium chloride and trisodium citrate concentration levels (Figure 3.8). Low heating temperatures and heating times produced creamy soft gels with good spreadability (Table 3.3). At the lowest heating time, heat-induced gelation was obtained only when calcium chloride was used (Figure 3.4).
TABLE 3.1. Split plot analysis of variance for treatment effects on gel strength of heat-induced UF concentrated UHT-treated 40% TS whole milk gels.

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<th>SOURCE</th>
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</thead>
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<td>0.70</td>
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<td>Error A</td>
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<td>1550.38</td>
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<td></td>
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<td>Temperature (T)</td>
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<tr>
<td>Time (M)</td>
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<td>1278733.30</td>
<td>5453.25</td>
<td>**</td>
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<td>Salt (S)</td>
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<td>3898547.50</td>
<td>16625.65</td>
<td>**</td>
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<td>Salt Concentration (C)</td>
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</tr>
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<td>**</td>
</tr>
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<td>**</td>
</tr>
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<td>H*C</td>
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<td>53259.14</td>
<td>227.13</td>
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<td>T*M</td>
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<td>M*C</td>
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<tr>
<td>H<em>T</em>S</td>
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<td>H<em>M</em>S</td>
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<td>H<em>S</em>C</td>
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<td>5183.31</td>
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<td>46.48</td>
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<td>Error B</td>
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<td>Total</td>
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<td>65040.71</td>
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NS Nonsignificant
** Significant p < 0.01
Figure 3.2. Shelf-life viscosity changes of direct steam (UHT)-treated UF concentrated (40% TS) whole milk.

TABLE 3.2. Shelf-life physico-chemical characteristics of direct steam (UHT) treated UF concentrated (40% TS) whole milk.

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<tr>
<th>Storage Time (Days)</th>
<th>pH (n=3) X ± SE</th>
<th>Total Plate Count (n=3) X ± SE</th>
<th>Total Solids (%) (n=3) X ± SE</th>
<th>Moisture (%) (n=3) X ± SE</th>
<th>Total Protein (%) (n=3) X ± SE</th>
<th>Fat (%) (n=3) X ± SE</th>
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<tr>
<td>1</td>
<td>6.7 ± .5 &lt; 10</td>
<td>39.8 ± .34</td>
<td>60.32 ± .62</td>
<td>13.8 ± .3</td>
<td>18.7 ± .4</td>
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<tr>
<td>15</td>
<td>6.6 ± .5 &lt; 10</td>
<td>39.7 ± .38</td>
<td>60.35 ± .59</td>
<td>13.8 ± .4</td>
<td>18.7 ± .4</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>6.6 ± .5 &lt; 10</td>
<td>39.7 ± .38</td>
<td>59.83 ± .63</td>
<td>13.6 ± .3</td>
<td>19.0 ± .5</td>
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<tr>
<td>45</td>
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<td>59.76 ± .61</td>
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<td>18.7 ± .4</td>
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</table>
Figure 3.3. Comparison of gel strength means of heat-induced UF concentrated UHT-treated 40% TS whole milk gels due to homogenization level and salt addition differences, with averaging on heating time, heating temperature, and salt concentration. Values with the same superscript letter (a, b, c, d) between salt type are not significantly different (LSD$_{0.05}$ = 8.75 for salt and LSD$_{0.05}$ = 10.72 for no salt). Values with the same superscript letter (x, y, z) between homogenization levels are not significantly different (LSD$_{0.05}$ = 8.75 for salt and LSD$_{0.05}$ = 12.38 for no salt).
Figure 3.4. Comparison of gel strength means of heat-induced UF concentrated UHT-treated 40% TS whole milk gels due to salt addition, heating temperature, and heating time differences, averaging on homogenization level and salt concentration. Values with the same superscript letter (a, b, c, d, e, f) between temperature and heating times are not significantly different (LSD_0.05 = 12.38 for salt type and LSD_0.05 = 17.51 for no salt). Values with the same superscript letter (w, x, y, z) between salt type are not significantly different (LSD_0.05 = 12.38 for salt type and LSD_0.05 = 15.16 for no salt).
Figure 3.5. Comparison of gel strength means of heat-induced UF concentrated UHT-treated 40% TS whole milk gels due to salt addition and heating temperature differences, with averaging on homogenization level, heating time, and salt concentration. Values with the same superscript letter (a, b, c, d) between salt type are not significantly different (LSD_{0.05} = 7.15 for salt and LSD_{0.05} = 8.75 for no salt). Values with the same superscript letter (x, y) between heating temperatures are not significantly different (LSD_{0.05} = 7.15 for salt and LSD_{0.05} = 10.11 for no salt).
Figure 3.6. Comparison of gel strength means of heat-induced UF concentrated UHT-treated 40% TS whole milk gels due to main effects: homogenization level ($\text{LSD}_{0.01} = 6.18$), heating temperature ($\text{LSD}_{0.01} = 5.05$), heating time ($\text{LSD}_{0.01} = 6.18$), and salt addition ($\text{LSD}_{0.01} = 8.18$). Means represent the average of the main effect across the other effects. Values with the same superscript letter within the same main effect are not significantly different at the $\text{LSD}_{0.01}$ indicated.
Figure 3.7. Comparison of gel strength means of heat-induced UF concentrated UHT-treated 40% TS whole milk gels due to heating temperature and heating time differences, averaging on homogenization level, salt addition, and salt concentration. Values with the same superscript letter (a, b, c) between heating times and superscript letter (x, y) between heating temperatures are not significantly different (LSD$_{0.01} = 8.75$).
Figure 3.8. Comparison of gel strength means of heat-induced UF concentrated UHT-treated 40% TS whole milk gels due to heating temperature, salt type, and salt concentration differences, with averaging on homogenization level and heating time. Values with the same superscript letter (a, b, c, d, e) between heating temperature and salt level and with the same superscript letter (x, y, z) between salt type are not significantly different (LSD_{0.05} = 10.0). Salt concentration (high = 0.3 M for NaCl and CaCl\(_2\) and 0.15 M for Na\(_3\)Citrate, low = 0.1 M for NaCl and CaCl\(_2\) and 0.03 M for Na\(_3\)Citrate).
TABLE 3.3. Effect of physical factors on textural properties of salt-treated, heat-induced UF concentrated UHT-treated 40% TS whole milk gels.

<table>
<thead>
<tr>
<th>Factor</th>
<th>Body</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low Homogenization</td>
<td>Compact, firm</td>
<td>Elastic, good spreadability</td>
</tr>
<tr>
<td>Medium Homogenization</td>
<td>Compact, firm</td>
<td>Elastic, good spreadability</td>
</tr>
<tr>
<td>High Homogenization</td>
<td>Compact, soft</td>
<td>Elastic, good spreadability</td>
</tr>
<tr>
<td>Low Heating Temp.</td>
<td>Smooth, soft</td>
<td>Creamy, good spreadability</td>
</tr>
<tr>
<td>High Heating Temp.</td>
<td>Compact, Firm</td>
<td>Rubbery, poor spreadability</td>
</tr>
<tr>
<td>Low Heating Time</td>
<td>Very soft</td>
<td>Creamy, good spreadability</td>
</tr>
<tr>
<td>High Heating Time</td>
<td>Compact, firm</td>
<td>Rubbery, poor spreadability</td>
</tr>
</tbody>
</table>

**Effect of Homogenization Pressure**

In non-salt-treated samples heat-induced gelation did not occur at all homogenization levels (Figure 3.3).

In salt-treated samples, intermediate homogenization levels (20.7 MPa) produced the highest gel strengths (Figure 3.6) at all heating times (Figure 3.9) and salt type addition (Figure 3.3), but particularly at the lower heating temperature (Figures 3.10, 3.11, and 3.12). At higher heating temperatures only the lowest heating time showed this behavior (Figure 3.11). The highest homogenization level produced the lowest gel strengths at all temperatures, heating times, and salt additions (Figures 3.3, 3.6, 3.9, and 3.10).

An explanation of this observation could be found by the formation of an optimal balance between the sizes of the denatured milk protein agglomerates and the milkfat globules that could allow for the formation of a protein tridimensional network (24) of maximum strength at intermediate homogenization pressures. The size and strength of the
Figure 3.9. Comparison of gel strength means of heat-induced UF concentrated UHT-treated 40% TS whole milk gels due to homogenization level and heating time differences, with averaging on heating temperature, salt type, and salt concentration. Values with the same superscript letter (a, b, c) between homogenization levels and with the same superscript letter (x, y, z) between heating times are not significantly different (LSD_{0.01} = 10.71).
Figure 3.10. Comparison of gel strength means of heat-induced UF concentrated UHT-treated 40% TS whole milk gels due to homogenization level and heating temperature differences, averaging on salt addition, salt concentration, and heating time. Values with the same superscript letter (a, b, c) between homogenization levels and superscript letter (x, y) between heating temperatures are not significantly different (LSD0.01 = 8.75).
Figure 3.11. Comparison of gel strength means of heat-induced UF concentrated UHT-treated 40% TS whole milk gels due to homogenization level, heating temperature, and heating time differences, averaging on salt addition and salt concentration. Values with the same superscript letter (a, b, c, d) between homogenization level and heating temperature and with the same superscript letter (x, y, z) between heating times are not significantly different (LSD_{0.05} = 11.34).
Figure 3.12. Comparison of gel strength means of heat-induced UF concentrated UHT-treated whole milk gels due to homogenization level, salt addition, and heating temperature differences, with averaging on heating time and salt concentration. Values with the same superscript letter (a, b, c, d, e, f) between homogenization level and heating temperature are not significantly different (LSD$_{0.05}$ = 12.25 for salt type and LSD$_{0.05}$ = 17.33 for no salt). Values with the same superscript letter (w, x, y, z) between salt types are not significantly different (LSD$_{0.05}$ = 12.25 between salts and LSD$_{0.05}$ = 15.01 for no salt).
protein agglomerates will be determined by the homogenization step before heating. The higher the homogenization pressure the larger the number of fat globules with smaller average size that will be formed. Under no pre-heat treatment (UHT), this would have allowed for the formation of a denser network of denatured proteins during the final heating in the convection oven. However, the UHT pre-treatment denatures the whey proteins almost completely, weakening the ability of the smaller protein agglomerates to recomplex in the second heat treatment on the convection oven (14) and, therefore, producing weaker gels at the highest homogenization pressures.

**Effect of Salt Type and Concentration**

Addition of salts had a positive effect in restoring and increasing the gel strength of heat-induced shelf-stable UF concentrated milk gels, with the highest gel strengths produced by calcium chloride, followed by sodium chloride and trisodium citrate. Retentate without the addition of salt did not produce a gel (Figure 3.6). This could be explained due to the partial-to-complete denaturation of whey proteins occurring during the UHT treatment that supports the theory that disulfide bridge formation is the main mechanism involved in the heat-induced gelation of concentrated milk (9, 17). The fact that the addition of salts restored the heat-induced gelation ability of the concentrated UHT-treated milk would appear to support the hypothesis that a mechanism other than disulfide bridges alone was also involved in the heat-induced gelation process (8, 10, 25).

Calcium chloride produced the highest gel strengths at all temperatures and heating times when compared to the other salts (Figures 3.5 and 3.13). This could be explained by the calcium’s ability to form salt bridges between casein micelles during the heat treatment (13). At the higher heating temperature calcium chloride produced gels of lower strength than when heated at the lower heating temperature (Figure 3.4). Overall, calcium chloride had lower gel strengths at higher concentrations when compared to lower concentrations
Figure 3.13. Comparison of gel strength means of heat-induced UF concentrated UHT-treated 40% TS whole milk gels due to salt addition and heating time differences, averaging on homogenization level, heating temperature, and salt concentration. Values with the same superscript letter (a, b, c, d) between salt type are not significantly different (LSD$_{0.05}$ = 8.75 for salt and LSD$_{0.05}$ = 10.72 for no salt) and superscript letter (x, y, z) between heating times are not significantly different (LSD$_{0.05}$ = 8.75 for salt and LSD$_{0.05}$ = 12.38 for no salt).
This reduction in gel strength was caused by the formation of small pockets during heating that in turn created a sponginess effect during gel strength measurement similar to the one observed in non-UHT-treated retentate gels. Calcium chloride gels were semiporous and harder than the other salt-produced gels; they were brittle when spread and presented some syneresis when higher temperatures and heating times were used (Table 3.4). This syneresis indicates that the ability to immobilize water by the gel protein network has been severely impaired by the presence of calcium chloride (14).

Sodium chloride addition produced increased gel strengths with higher salt concentration, temperatures, and heating times (Figures 3.5, 3.8, and 3.13). Very soft gels were produced at the lowest heating times (Figure 3.4). In general sodium chloride gels had a firm body but with good spreadability (Figure 3.2). The impact of the UHT treatment made the sodium chloride gels more spreadable than the non-UHT-treated ones.

TABLE 3.4. Effect of salt treatments on textural properties of heat-induced UF concentrated UHT-treated 40% TS whole milk gels.

<table>
<thead>
<tr>
<th>Salt</th>
<th>Body</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Salt</td>
<td>No gel</td>
<td>Viscous pourable liquid</td>
</tr>
<tr>
<td>NaCl</td>
<td>Compact, firm</td>
<td>Rubbery, good spreadability</td>
</tr>
<tr>
<td>Na₃Citrate</td>
<td>Smooth, very soft</td>
<td>Lightly creamy, sticky, good spreadability</td>
</tr>
<tr>
<td>CaCl₂</td>
<td>Hard, Cheesy, like appearance</td>
<td>Semiporous, brittle when spread, some syneresis present</td>
</tr>
</tbody>
</table>
Fig 3.14. Comparison of gel strength means of heat-induced UF concentrated UHT-treated 40% TS whole milk gels due to salt addition and salt concentration differences, averaging on homogenization level, heating temperature, and heating time. Values with the same superscript letter (a, b, c) between salt type and superscript letter (x, y, z) between salt concentration are not significantly different (LSD$_{0.01} = 9.45$).
The ability of sodium chloride to restore the heat-induced gelation property of UHT-treated retentate may be related to its ability to influence the hydrophobic interactions or hydration forces between casein particles that induce protein conformational changes (10), which in turn allow for the formation of a gel during a second heat treatment in the convection oven.

Trisodium citrate produced very soft and smooth gels with the exception of the combination of high salt concentration, intermediate homogenization pressures, and low heating times (Figure 3.15) that produced, when compared to the other salt treatments, a very smooth and spreadable texture (Table 3.4). This behavior is related to the ability of trisodium citrate to chelate micellar calcium and, therefore, alter the milk salt balance of concentrated milk systems (8, 10, 21).

Further work is needed to clarify the mechanisms of action of salts on the texture and gel strength of heat-induced, UF concentrated, UHT-treated whole milk gels.

CONCLUSIONS

1. A pourable, shelf-stable UF concentrated whole milk product could be manufactured using UHT processing coupled with aseptic packaging. This concentrated milk system had a shelf-life at 23°C of 75 to 90 d, but did not have the ability to produce heat-induced gels after a second heating. High homogenization pressures in addition to the high turbulence environment during direct steam sterilization appeared to be responsible for the decrease or elimination of the concentrated whole milk system’s ability to produce a gel when heated a second time. However, the addition of calcium chloride, sodium chloride, or trisodium citrate salts restored this heat-induced gelation ability.
Figure 3.15. Comparison of gel strength means of heat-induced UF concentrated UHT-treated 40% TS whole milk gels due to homogenization level, salt type, and salt concentration differences, with averaging on heating temperature and heating time. Values with the same superscript letter (a, b, c, d, e, f, g) between homogenization level and salt concentration and with the same superscript letter (x, y, z) between salt types are not significantly different (LSD_{0.05} = 12.25). Salt concentration (high = 0.3 \textit{M} for \text{NaCl} and \text{CaCl}_2 and 0.15 \textit{M} for \text{Na}_3\text{Citrate}, low = 0.1 \textit{M} for \text{NaCl} and \text{CaCl}_2 and 0.03 \textit{M} for \text{Na}_3\text{Citrate}).
2. The use of trisodium citrate, high homogenization pressures, and low heating temperatures produced smooth and soft gels with good spreadability. The combination of these factors could be used for the development of gelled food products with good spreadability or smoother mouthfeel. The use of calcium chloride, sodium chloride, intermediate homogenization pressures, high heating temperatures, and long heating times produced firmer and more elastic gels that could be used for the development of gelled food products with a requirement for a more dense mouthfeel.

REFERENCES


CHAPTER 4

RELATIONSHIP OF GEL STRENGTH TO ULTRASTRUCTURE OF HEAT-INDUCED UF CONCENTRATED AND UHT-TREATED 40% TS WHOLE MILK GELS AND PRODUCT APPLICATIONS.

ABSTRACT

Physical and chemical factors influencing the firmness and ultrastructure of heat-induced milk gels made from UF concentrated and UF concentrated UHT-treated 40% total solids whole milk gels were studied. Whole milk was UF concentrated to 40% total solids using a spiral wound (20,000 mol wt cutoff) polysulfone membrane system. A portion of the retentate was two-staged homogenized at 6.68 or 34.47 MPa (1000 or 5000 psi) with the second stage at 15% of total pressure. Ultra-high temperature processing by direct steam injection of the remainder of the retentate was conducted in an Alfa-Laval Sterilab™ pilot plant with down-stream two-stage homogenization at 20.68 MPa (3000 psi) with the second stage at 15% of total pressure. Ultrafiltration concentrated and UF concentrated-UHT-treated milk concentrate samples (150 ml) were treated with 0.3 M calcium chloride or sodium chloride, or 0.15 M trisodium citrate, placed in thin-walled aluminum cans, and heat treated in a conventional electric oven at 135°C or 190°C for 20 or 30 min. The samples were cooled to room temperature, and the gel strength was measured using a penetrometer. Transmission electron microscopy was used to determine the gel ultrastructure of selected treatments. Ultrafiltration concentrated retentate was the main ingredient to formulate two cheesecake-type prototypes that were evaluated using a hedonic taste panel test by a general consumer population. Electron microscopy results showed that there is a direct correlation between gel firmness and gel ultrastructure of heat-induced UF concentrated and UF concentrated-UHT-treated whole milk gels prepared under different physical and chemical conditions.
The gels consisted of a tridimensional network of casein micelles connected with strands of less dense denatured whey proteins. The gels treated with calcium chloride presented a gelled network of fused micelles that in turn had the highest gel strengths. In gels containing trisodium citrate, a calcium binding agent, some of the casein micelles showed disintegration producing softer gels. The restoration of the ability to form gels on the UHT-treated retentate by the addition of salt treatments suggests that mechanisms other than the disulfide bridge formation between protein structures play a contributing significant role in the heat-induced gelation of UF concentrated whole milk systems. The consumer sensory panels showed that the property of heat-induced gelation of a UF concentrated milk system can be successfully used in the development of new gelled dairy dessert applications, with texture and flavor having the greatest impact on the overall acceptability of these kinds of desserts.

INTRODUCTION

Heat-induced gelation of milk systems has been studied by different researchers with the objective of its understanding and preventing such gelation (1, 14, 16, 18), but recently the attention has focused in its application as a potential property for new product development (11, 13).

Heat-induced gelation of concentrated milk systems is a complex phenomenon that involves multiple changes in hydrogen and hydrophobic bonds, salt and disulfide linkages, and calcium bridges between protein molecules (15). The firmness and texture of such gels are determined by several physical and chemical factors. These factors include temperature and duration of heating, temperature of testing, protein concentration, and addition of various chemical agents (6, 9, 10, 11, 20), all of which presumably act at the submicroscopic level.
Electron microscopy techniques have been applied in the study of casein micelles in milk (8) and milk gels (12). In general an increase in the casein micelle size was observed in heated milk (4) with coalescence of the micelles preceding gelation (3, 19). Studies on heat-induced milk gels of concentrated aqueous suspensions of NFDM, using scanning and transmission electron microscopy, showed that these gels consist of a network of aggregated casein micelle strands cross-linked with denatured β-Lg. The gels presented a close correlation between microstructure and firmness. Gels with total solids concentration of 40 and 50% were characterized by a network of individual casein micelles linked by some less dense bridging material and lower gel strengths, while gels with 60% total solids presented a network of fused micelles with higher gel strengths. The addition of calcium chloride and ammonium persulphate to the NFDM concentrated gels caused the micelles to fuse and produced gels of appreciable increased firmness while the addition of hexametaphosphate caused micelle disintegration and gels of decreased firmness (12).

The relationship between microstructure and firmness in heat-induced gels made from UF concentrated whole milk has not been studied.

OBJECTIVES

The objectives of this study were:

1. To determine the relationship between gel strength and gel ultrastructure of UF concentrated and UF concentrated UHT-treated 40% TS whole milk gels, under different physical and chemical conditions using transmission electron microscopy (TEM).

2. To evaluate the use of the heat-induced gelation property of UF concentrated whole milk systems in dairy desserts applications, by conducting a sensory evaluation study of two concept product applications in a general consumer population.
MATERIALS AND METHODS

Milk and UF Retentate Preparation

Whole milk from the Utah State University dairy farm was vat pasteurized at 63°C for 30 min. The pasteurized milk was UF concentrated to 40% TS via a spiral wound (20,000 mol wt cutoff) polysulfone membrane system (Osmonics, Inc., Minnetonka, MN) between 46 to 48°C. One half of the UF retentate was two-stage homogenized at 6.68, or 34.47 MPa (1000, or 5000 psi) with the second stage at 15% of total pressure and stored under refrigeration conditions (4 ± 1°C) until required for gel treatment and product preparation. The other half of the UF retentate was first UHT processed in an Alfa-Laval Sterilab™ pilot plant (Alfa-Laval, Lund, Sweden) using direct steam injection with preheating at 75 ± 1°C for 30 s, sterilization at 141 ± 1°C for 4 s, and then flash cooling to 72 ± 1°C in a vacuum flash evaporator before a two-stage down-stream aseptic homogenization was done at 20.68 MPa (3000 psi) with the second stage at 15% of total pressure. A final cooling of the retentate to 60 ± 1°C was performed before collection in open containers followed by storage under refrigerated conditions (4 ± 1°C) until used for gel treatment and product preparation. Untreated and UHT-treated permeate was stored under refrigerated condition until salt treatment preparation.

Gel Samples Preparation

Salt solutions were prepared by dissolving the needed amount of salt in a minimum amount of the corresponding permeate and then mixed with 1000 ml of the non-treated and UHT-treated retentate to obtain a final salt concentration of 0.3 M for sodium chloride or calcium chloride, or 0.05 M for trisodium citrate. The samples were allowed to stabilize for 20 min before the heat treatment. One hundred fifty-gram portions of the retentates were placed in standard soft drink aluminum containers (0.15 mm thickness, 6.5 cm diameter, 12 oz capacity), that were previously cut to approximately 8 cm in height,
and heated in a conventional electric oven at 135°C or 190°C for 20 or 30 min as required. The gels were cooled overnight (10 h) before measuring the gel strength.

**Measurement of Gel Strength**

Gel strength defined as the maximum penetration force (g) through the milk gel samples was measured using a Utah State University penetrometer (5) with a 15.5-mm cylindrical probe descending at a constant speed of 1 cm/min into the center of the gels. Gel strength was measured in the gels cooled overnight at 22°C. The tops of the gels (3 mm) were cut off and discarded before reading.

**Microstructure**

Strips of gel 1x1x15 mm in size were obtained from the center of representative gels and soaked in a 2.5% glutaraldehyde solution in cacodylate buffer (pH 7.0). Transmission electron micrographs (TEM) were prepared from each sample at the electron microscopy facility at Utah State University using the method described by Kalab (12).

**Prototype Product Preparation**

Two prototype cheesecake-type formulations were selected arbitrarily. The formulations were required to have a minimum number of ingredients to better determine the impact of heat-induced gel textures produced with UF concentrated whole milk systems as evaluated by a general consumer population. An acid-baked cheesecake product was prepared by mixing equal parts of sweetened condensed milk with a 0.03 M trisodium citrate-treated UF retentate, followed by acidification with lime juice to a pH of 4.9 ± 0.1 and baking at 135°C for 10 min. A second baked cheesecake product was prepared by adding 10% sucrose to a 0.05 M calcium chloride-treated retentate followed by baking at 190°C for 20 min. Both products were allowed to cool before being served at room temperature (23°C).
Sensory Analysis

Sensory panels with untrained judges (18 years and older), representing a general consumer population, were used. A 9-point hedonic scale (9 being “like extremely,” 5 being “neither like nor dislike,” and 1 being “dislike extremely”) was used to determine the consumer acceptability of the two heat-gelled desserts (2). The appearance, flavor, texture, and overall acceptability of the samples were rated by 89 panelists.

Statistical Analysis

Hedonic data were evaluated using frequency distributions, sample means, sample standard deviations, and 95% confidence intervals (17). Linear correlation and regression analysis between overall acceptability and appearance, flavor, and texture were conducted. All statistical analysis was performed using Minitab® release 11.2 (State College, PA).

RESULTS AND DISCUSSION

Transmission electron microscopy sections of all the gel treatments selected showed that gels were formed by a network of interconnected micelles. The projections reaching from one micelle to another were composed of a less dense material presumably from denatured whey proteins in the system (12). The changes in the total protein network showed visual changes in the density of the interconnecting protein material as well as on the size and integrity of the micelles forming the network.

Effect of Heating Temperatures and Heating Times on Gel Strength and Gel Ultrastructure

Higher heating temperatures and heating times produced increased gel strength in non-salt-treated gel samples (Figure 4.1). Transmission electron microscopy examination
Figure 4.1. Comparison of gel strength means of heat-induced UF concentrated 40% TS whole milk gels due to homogenization level, heating temperature, and heating time. (A) No homogenization, 135°C for 20 min; (B) No homogenization, 190°C for 20 min; (C) 6.7 Mpa, 190°C for 30 min; (D) 34.5 Mpa, 190°C for 30 min.
of sections of these gels showed that the density of the protein material connecting the micelles increased with higher temperatures and heating times (Figures 4.2, 4.3, and 4.4). An increase in denatured whey protein material on the surface of the micelles was also observed as the heating temperature was increased from 135°C to 191°C (Figure 4.3). At the highest heating temperature and heating time the micelles started to fuse together, losing their individuality (Figure 4.4), creating a reinforced protein network that accounted for the higher gel strengths observed.

**Effect of Homogenization Levels on Gel Strength and Gel Ultrastructure**

Higher homogenization pressures produced higher gel strengths in non-UHT, non-salt-treated gels (Figure 4.1). Examination of section of these gels by TEM showed that homogenization pressures of 34.47 MPa caused a reduction in the average size of the interconnected casein micelles with no signs of micelle fusion (Figure 4.5) as compared to the samples treated at 6.89 Mpa, which had a larger average micelle size with fused micelles participating in the protein network (Figure 4.4). Apparently the larger number of interconnecting micelles in the protein network caused by high homogenization pressures (34.47 MPa) has the tendency to reinforce the protein network due to the additional contact points through the matrix, which in turn will be responsible for the measured gel strength differences between the two homogenization levels.

**Effect of Salt Treatment on Gel Strength and Gel Ultrastructure**

In general calcium chloride gels tended to produce stronger gels, followed by sodium chloride and trisodium citrate (Figure 4.6). Changes in the ultrastructure between the different salt-treated gels were evident.

Addition of calcium produced gels with a protein network of mostly fused micelles. Very few individual non-fused micelles remained in the network (Figure 4.7).
Figure 4.2. Transmission electron micrograph of a heat-induced UF concentrated 40% TS non-homogenized whole milk gel heated at 135°C for 20 min.
Figure 4.3. Transmission electron micrograph of a heat-induced UF concentrated 40% TS non-homogenized whole milk gel heated at 190.6°C for 20 min.
Figure 4.4. Transmission electron micrograph of a heat-induced UF concentrated 40% TS whole milk gel homogenized at 6.89 MPa, and heated at 190.6°C for 30 min.
Figure 4.5. Transmission electron micrograph of a heat-induced UF concentrated 40% TS whole milk gel homogenized at 34.47 MPa, and heated at 190.6°C for 30 min.
Figure 4.6. Comparison of gel strength means of heat-induced UF concentrated and UF concentrated-UHT-treated 40% TS whole milk gels by salt treatment (0.3 $M$ for NaCl and CaCl$_2$ and 0.15 $M$ Na$_3$Citrate).
Figure 4.7. Transmission electron micrograph of a heat-induced UF concentrated 40% TS whole milk gel treated with 0.3 \( M \) calcium chloride, homogenized at 20.68 MPa, and heated at 190.6°C for 20 min.
The calcium chloride-supplemented gels also showed some syneresis as compared to the other gels, indicating that there was some loss in the ability of the gels to immobilize water.

Sodium chloride-treated gels presented a more clearly defined network of intact casein micelles interconnected with less dense protein material (Figure 4.8) when compared with the other salt treatments (Figures 4.7 and 4.9). However, these gels had a similar structure when compared with non-salt-treated gels (Figures 4.3 and 4.5).

Gels made with added trisodium citrate had the tendency to be less firm than the other salt-treated gels. These gels presented a very large amount of very small components evenly distributed over the entire field of vision and very limited interconnecting material between the casein micelles (Figure 4.9). It seems that these small particles are the breakdown product of the micelles, since the gradual disintegration of the casein micelles was apparent in some areas of the micrograph. Similar behavior was observed by Kalab (12) in NFDM gels treated with sodium hexametaphosphate, which is used to stabilize heated milk systems. The disintegration of casein micelles in a milk protein gel was closely associated with its loss of cohesiveness, which accounted for its lower gel strength. Since there was no syneresis observed in these gels, it also suggested that different forces were involved in the water immobilization process.

Effect of UHT Treatment on Gel Strength and Gel Ultrastructure

No gels were formed in UHT-treated samples after a second heat treatment. However, the addition of calcium chloride and sodium chloride salts restored the ability of these UHT-treated samples to form gels when heated a second time. It is unclear how this process occurs, but it could be related to salt balance mechanisms and the ability of calcium to form bridges between proteins (7, 12). Under the conditions used to generate the gels for TEM analysis, trisodium citrate produced a very weak gel during the second
Figure 4.8. Transmission electron micrograph of a heat-induced UF concentrated 40% TS whole milk gel treated with 0.3 $M$ sodium chloride, homogenized at 20.68 MPa, and heated at 190.6°C for 20 min.
Figure 4.9. Transmission electron micrograph of a heat-induced UF concentrated 40% TS whole milk gel treated with 0.15 M trisodium citrate, homogenized at 20.68 MPa, and heated at 190.6°C for 20 min.
heat treatment (Figure 4.6), but it did produce stronger gels under different conditions as previously determined. Overall, these treatments had lower gel strengths than their non-UHT-treated gel counterparts (Figure 4.6). This observation can be explained by the partial to total denaturation of the milk proteins during UHT processing temperatures (7). Transmission electron microscopy micrographs appear to support this assumption since the calcium chloride and sodium chloride UHT treatments presented an increase in the average micelle size due to the complexing of whey proteins on the surface of the casein micelles (Figures 4.10, 4.11, and 4.12).

Ultra-high temperature-treated calcium chloride, and to a lesser extent sodium chloride, treatments showed clusters of fused casein micelles in the protein network. They also showed the presence of fewer, but better defined, strands of the less dense protein material connecting the micelle network (Figures 4.10 and 4.11) as compared to their non-UHT-treated gel counterparts (Figures 4.7 and 4.8). It is assumed that these better defined strands are the result of the more severe whey protein denaturation occurring during the UHT treatment step. What is still unknown is how the addition of these salts allows for the restoration of this heavily denatured protein network.

Sensory Analysis Results

**Acid-baked cheesecake.** The acid-baked cheesecake prototype was rated favorably by the general consumer taste panel (Figure 4.13). It was rated for all its sensorial properties with average ratings ranging from 7.3 to 7.9 on the hedonic scale (1 to 9) where 7 was rated as “liked moderately” (Table 4.1). Comments from the panelists indicated that overall this product was well liked due mostly to its textural properties and mouth feel, with some comments made on reducing the intensity of the acidified flavor imparted by the lime juice used in the formulation. The statistical analysis of the data, which included analysis of variance of overall acceptability by the other textural properties
Figure 4.10. Transmission electron micrograph of a heat-induced UF Concentrated-UHT-treated 40% TS whole milk gel treated with 0.3 M calcium chloride, homogenized at 20.68 MPa, and heated at 190.6°C for 20 min.
Figure 4.11. Transmission electron micrograph of a heat-induced UF Concentrated-UHT-treated 40% TS whole milk gel treated with 0.3 $M$ sodium chloride, homogenized at 20.68 MPa, and heated at 190.6°C for 20 min.
Figure 4.12. Transmission electron micrograph of a heat-induced UF Concentrated-UHT-treated 40% TS whole milk gel treated with 0.15 M trisodium citrate, homogenized at 20.68 MPa, and heated at 190.6°C for 20 min.
Figure 4.13. Appearance, flavor, texture and overall acceptability frequency distributions (N=89) of hedonic scale sensory data (9 being “like extremely,” 5 being “neither like nor dislike,” and 1 “dislike extremely”) for acid baked cheesecake.
TABLE 4.1 Descriptive statistics of hedonic rating data for acidified baked cheesecake and baked cheesecake.

<table>
<thead>
<tr>
<th>Variable</th>
<th>N</th>
<th>Mean</th>
<th>s</th>
<th>95% Confidence Interval</th>
<th>95% Confidence Interval</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Mean</td>
<td></td>
<td>Mean</td>
<td>s</td>
</tr>
<tr>
<td>Acidified Cheesecake</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Appearance</td>
<td>89</td>
<td>7.89</td>
<td>1.07</td>
<td>(7.66 - 8.11)</td>
<td>(0.93 - 1.26)</td>
</tr>
<tr>
<td>Flavor</td>
<td>89</td>
<td>7.26</td>
<td>1.79</td>
<td>(6.88 - 7.63)</td>
<td>(1.56 - 2.10)</td>
</tr>
<tr>
<td>Texture</td>
<td>89</td>
<td>7.53</td>
<td>1.51</td>
<td>(7.21 - 7.85)</td>
<td>(1.31 - 1.77)</td>
</tr>
<tr>
<td>Overall Acceptability</td>
<td>89</td>
<td>7.46</td>
<td>1.52</td>
<td>(7.14 - 7.78)</td>
<td>(1.32 - 1.78)</td>
</tr>
<tr>
<td>Baked Cheesecake</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Appearance</td>
<td>89</td>
<td>5.85</td>
<td>1.96</td>
<td>(5.44 - 6.27)</td>
<td>(1.71 - 2.30)</td>
</tr>
<tr>
<td>Flavor</td>
<td>89</td>
<td>5.18</td>
<td>2.11</td>
<td>(4.73 - 5.63)</td>
<td>(1.84 - 2.48)</td>
</tr>
<tr>
<td>Texture</td>
<td>89</td>
<td>4.69</td>
<td>2.28</td>
<td>(4.20 - 5.17)</td>
<td>(1.99 - 2.67)</td>
</tr>
<tr>
<td>Overall Acceptability</td>
<td>89</td>
<td>4.93</td>
<td>2.11</td>
<td>(4.49 - 5.38)</td>
<td>(1.84 - 2.47)</td>
</tr>
</tbody>
</table>

as well as Pearson and linear correlation analysis, showed that overall acceptability was statistically determined by the ratings of the other three textural properties with texture having the higher correlation coefficients followed by flavor (Tables 4.2, 4.3, and 4.4). Correlation coefficients on appearance showed that this textural property was not a significant determinant of the overall acceptability of the acid baked cheesecake.

**Baked cheesecake.** The baked cheesecake prototype had a moderate rating by the consumers in the taste panels (Figure 4.14). It was rated for all its sensorial properties with average ratings ranging from 4.7 to 5.9 on the hedonic scale (1 to 9) where 4 was rated as “disliked slightly” and 5 “neither liked nor disliked” (Table 4.1). Comments from the panelists indicated that the flavor was weak and that the texture was too firm and rubbery and that a creamier, softer texture would be preferred. The frequency
### TABLE 4.2 Analysis of variance for sensory attributes on overall acceptability of acid-baked cheesecake.

<table>
<thead>
<tr>
<th>SOURCE</th>
<th>DF</th>
<th>MS</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>5</td>
<td>0.632</td>
<td>4.17</td>
<td>0.002</td>
</tr>
<tr>
<td>Flavor</td>
<td>7</td>
<td>2.367</td>
<td>15.64</td>
<td>0.000</td>
</tr>
<tr>
<td>Texture</td>
<td>7</td>
<td>3.260</td>
<td>21.54</td>
<td>0.000</td>
</tr>
<tr>
<td>Error</td>
<td>69</td>
<td>0.151</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>88</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### TABLE 4.3 Pearson correlation between sensory attributes of acid-baked cheesecake and baked cheesecake.

<table>
<thead>
<tr>
<th>Acid Bake Cheesecake</th>
<th>Appearance</th>
<th>Flavor</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flavor</td>
<td>0.330</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Texture</td>
<td>0.305</td>
<td>0.733</td>
<td></td>
</tr>
<tr>
<td>Overall Acceptability</td>
<td>0.382</td>
<td>0.870</td>
<td>0.907</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Bake Cheesecake</th>
<th>Appearance</th>
<th>Flavor</th>
<th>Texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flavor</td>
<td>0.368</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Texture</td>
<td>0.559</td>
<td>0.738</td>
<td></td>
</tr>
<tr>
<td>Overall Acceptability</td>
<td>0.459</td>
<td>0.908</td>
<td>0.844</td>
</tr>
</tbody>
</table>
TABLE 4.4 Regression analysis for sensory attributes on overall acceptability of acid-baked cheesecake.

Regression equation

Overall Acceptability = -0.249 + 0.094 Appearance + 0.364 Flavor + 0.574 Texture

<table>
<thead>
<tr>
<th>Predictor</th>
<th>Coefficient</th>
<th>s</th>
<th>T</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>-0.249</td>
<td>0.378</td>
<td>-0.66</td>
<td>0.511</td>
</tr>
<tr>
<td>Appearance</td>
<td>0.094</td>
<td>0.047</td>
<td>2.01</td>
<td>0.048</td>
</tr>
<tr>
<td>Flavor</td>
<td>0.364</td>
<td>0.039</td>
<td>9.23</td>
<td>0.000</td>
</tr>
<tr>
<td>Texture</td>
<td>0.574</td>
<td>0.046</td>
<td>12.40</td>
<td>0.000</td>
</tr>
</tbody>
</table>

\[ R^2 = 91.7 \]

\[ p \text{ value} = 0.000 \]

distributions of all the properties evaluated appeared to present a bimodal normal distribution that seemed to suggest that the general consumer population was separated in two different groups based on their liking of this product, with one group having a greater acceptance of the product than the other. The statistical analysis of the data indicated that overall acceptability was significantly determined only by the ratings on flavor and texture. Appearance did not have a statistically significant impact on the overall acceptability of the baked cheesecake prototype as indicated by the results on the analysis of variance and correlation coefficients analysis (Tables 4.3, 4.5, and 4.6).

CONCLUSIONS

1. There is a direct relationship between gel firmness and gel ultrastructure of heat-induced UF concentrated and UF concentrated-UHT-treated whole milk gels prepared under different physical and chemical conditions. The gels consisted of a tridimensional network of casein micelles connected with strands of less dense protein material, assumed to be denatured whey proteins. The strength of the heat-induced milk gels
Figure 4.14. Appearance, flavor, texture and overall acceptability frequency distributions (N=89) of hedonic scale sensory data (9 being “like extremely,” 5 being “neither like nor dislike,” and 1 “dislike extremely”) for baked cheesecake.
TABLE 4.5 Analysis of variance for sensory attributes on overall acceptability of baked cheesecake.

<table>
<thead>
<tr>
<th>SOURCE</th>
<th>DF</th>
<th>MS</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>8</td>
<td>0.687</td>
<td>1.37</td>
<td>0.227</td>
</tr>
<tr>
<td>Flavor</td>
<td>8</td>
<td>5.766</td>
<td>11.49</td>
<td>0.000</td>
</tr>
<tr>
<td>Texture</td>
<td>8</td>
<td>2.505</td>
<td>4.99</td>
<td>0.000</td>
</tr>
<tr>
<td>Error</td>
<td>64</td>
<td>0.502</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>88</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

TABLE 4.6 Regression analysis for sensory attributes on overall acceptability of baked cheesecake.

Regression equation

Overall Acceptability = -0.047 + 0.024 Appearance + 0.626 Flavor + 0.341 Texture

<table>
<thead>
<tr>
<th>Predictor</th>
<th>Coefficient</th>
<th>s</th>
<th>T</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>-0.047</td>
<td>0.268</td>
<td>-0.17</td>
<td>0.862</td>
</tr>
<tr>
<td>Appearance</td>
<td>0.024</td>
<td>0.047</td>
<td>0.51</td>
<td>0.614</td>
</tr>
<tr>
<td>Flavor</td>
<td>0.626</td>
<td>0.053</td>
<td>11.75</td>
<td>0.000</td>
</tr>
<tr>
<td>Texture</td>
<td>0.341</td>
<td>0.055</td>
<td>6.15</td>
<td>0.000</td>
</tr>
</tbody>
</table>

R² = 89.0

*p value = 0.000

was correlated to the amount of less dense protein material present and its location in the protein network structure. The location and amount of the less dense protein material in the protein network was also dependent on the physical treatments applied to the concentrated milk before the final heating conditions to generate the heat-induced milk gels were used. These conditions included homogenization levels and levels of turbulence present during direct steam UHT sterilization of the concentrated
milk system. The chemical agents studied play an important contributing role in the heat-induced gelation of UF concentrated whole milk systems, and a determining role in the formation of heat-induced gels of UF concentrated-UHT-treated milk systems during a secondary heat treatment.

2. The heat-induced gelation property of UF concentrated whole milk gels can be used in the development of new gelled dairy dessert applications as the results of the hedonic general consumer test on the prototypes used in these studies have shown. These new products when fully and properly formulated can be targeted to a specific market population that could prove to be commercially successful.

REFERENCES


CHAPTER 5
GENERAL SUMMARY AND CONCLUSIONS

Heat-induced gelation of UF concentrated whole milk has the ability to produce gels of various strengths and textural properties that can be used in the development of gelled food products in which the concentrated milk systems is used as the main ingredient. The conclusions of this research are:

1. The functional property of UF concentrated whole milk of producing heat-induced gels can be obtained at concentrations above 35% TS and can be optimized with a combination of factors such as homogenization levels, temperature and heating times, and the addition of salts. The use of trisodium citrate, low homogenization pressures, low heating temperatures, and short heating times produced smoother, softer gels with good spreadability that could be used for the development of products such as pudding and spreads. The use of calcium chloride, high homogenization pressures, high heating temperatures, and long heating times produced firmer gels that could be used to develop products such as cheesecake.

2. A pourable, shelf-stable UF concentrated whole milk product could be manufactured using UHT processing coupled with aseptic packaging. This concentrated milk system had a shelf life at 23°C of 75 to 90 d, but did not have the ability to produce heat-induced gels after a second heating. High homogenization pressures in addition to the high turbulence environment during direct steam sterilization appeared to be responsible for the decrease or elimination of the concentrated whole milk system’s ability to produce a gel when heated a second time. However, the addition of calcium chloride, sodium chloride, or trisodium citrate salts restored this heat-induced gelation ability.
3. The use of trisodium citrate, high homogenization pressures, and low heating temperatures produced smooth and soft gels with good spreadability. The combination of these factors could be used for the development of gelled food products with good spreadability or smoother mouthfeel. The use of calcium chloride, sodium chloride, intermediate homogenization pressures, high heating temperatures, and long heating times produced firmer and more elastic gels that could be used for the development of gelled food products with a requirement for a more dense mouthfeel.

4. There is a direct relationship between gel firmness and gel ultrastructure of heat-induced UF concentrated and UF concentrated-UHT-treated whole milk gels prepared under different physical and chemical conditions. The gels consisted of a tridimensional network of casein micelles connected with strands of less dense protein material, assumed to be denatured whey proteins. The strength of the heat-induced milk gels was correlated to the amount of less dense protein material present and its location in the protein network structure. The location and amount of the less dense protein material in the protein network was also dependent on the physical treatments applied to the concentrated milk before the final heating conditions to generate the heat-induced milk gels were used. These conditions included homogenization levels and levels of turbulence present during direct steam UHT sterilization of the concentrated milk system. The chemical agents studied play an important contributing role in the heat-induced gelation of UF concentrated whole milk systems, and a determining role in the formation of heat-induced gels of UF concentrated-UHT-treated milk systems during a secondary heat treatment.

5. The heat-induced gelation property of UF concentrated whole milk gels can be used in the development of new gelled dairy dessert applications as the results of the hedonic general consumer test on the prototypes used in these studies have shown. These new
products when fully and properly formulated can be targeted to a specific market population that could prove to be commercially successful.
Sensory Evaluation of ____________________

Name: ____________________________________ Date: ____________

Please evaluate the samples presented based on the following scale:

<table>
<thead>
<tr>
<th>Number</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>9</td>
<td>like extremely</td>
</tr>
<tr>
<td>8</td>
<td>like very much</td>
</tr>
<tr>
<td>7</td>
<td>like moderately</td>
</tr>
<tr>
<td>6</td>
<td>like slightly</td>
</tr>
<tr>
<td>5</td>
<td>neither like nor dislike</td>
</tr>
<tr>
<td>4</td>
<td>dislike slightly</td>
</tr>
<tr>
<td>3</td>
<td>dislike moderately</td>
</tr>
<tr>
<td>2</td>
<td>dislike very much</td>
</tr>
<tr>
<td>1</td>
<td>dislike extremely</td>
</tr>
</tbody>
</table>

Sample ______

Check the appropriate number for the given characteristic.

<table>
<thead>
<tr>
<th>Appearance:</th>
<th>Flavor:</th>
<th>Texture:</th>
<th>Overall Acceptability:</th>
</tr>
</thead>
<tbody>
<tr>
<td>9</td>
<td>_______</td>
<td>_______</td>
<td>_______</td>
</tr>
<tr>
<td>8</td>
<td>_______</td>
<td>_______</td>
<td>_______</td>
</tr>
<tr>
<td>7</td>
<td>_______</td>
<td>_______</td>
<td>_______</td>
</tr>
<tr>
<td>6</td>
<td>_______</td>
<td>_______</td>
<td>_______</td>
</tr>
<tr>
<td>5</td>
<td>_______</td>
<td>_______</td>
<td>_______</td>
</tr>
<tr>
<td>4</td>
<td>_______</td>
<td>_______</td>
<td>_______</td>
</tr>
<tr>
<td>3</td>
<td>_______</td>
<td>_______</td>
<td>_______</td>
</tr>
<tr>
<td>2</td>
<td>_______</td>
<td>_______</td>
<td>_______</td>
</tr>
<tr>
<td>1</td>
<td>_______</td>
<td>_______</td>
<td>_______</td>
</tr>
</tbody>
</table>

What do you think about the product? ____________________________________________
VITA

Hector Alejandro Solorio
Candidate for the Degree of
Doctor of Philosophy

Dissertation: Heat-Induced Gelation of Ultrafiltered Whole Milk Concentrate and Product Applications

Major Field: Nutrition and Food Sciences

Education:

B.S. National School of Agriculture, Mexico. Agricultural Engineering, 1982
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Ph.D. Utah State University, Nutrition and Food Sciences, 1999.

Experience:


Director of Research and Development, Crown Laboratories, Las Vegas, NV (1995). Directed all the technical aspects in the construction of Crown’s UHT aseptic facility, including FDA certification process, for the manufacture of nutraceutical liquid food products.

Research Assistant, Utah State University, Logan, UT (1988-1992). Planned and conducted research related to determining the different factors affecting the textural properties of heat-induced gels of UF concentrated milk, and UF concentrated UHT-treated milk.

Research Technician, Brigham Young University, Provo, UT (1987-1988). Responsible for finances of Bolivian project and manuscript writing of technology transfer of small-scale agriculture projects in Latin America.

Research Assistant, Brigham Young University, Provo, UT (1985-1987). Planned and conducted research related to determining the optimum economic lactation cycle using computerized statistical analysis.

Professional Memberships:

Phi Kappa Phi Honor Society
Institute of Food Technologists (IFT)
American Dairy Science Association (ADSA)
American Oil Chemist Society (AOCS)
American Association of Cereal Chemists (AACC)

Honors:

Academic Scholarship, National School of Agriculture, Mexico
Mexican Presidential Scholarship, National School of Agriculture
Academic Scholarship, Brigham Young University, AgEcon. Department
Phi Kappa Phi, Brigham Young University Chapter
Phi Kappa Phi, Utah State University Chapter

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Born 16 June 1959, Mexico City, Mexico
Married Margie Nielsen, 19 December 1987
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