# Development and Characterization of a Novel Concentrated Milk Product Using Membrane Filtration Technology

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**Abstract**

In recent years microfiltration (MF) technology has gained prominence in dairy industry due to its ability to separate virtually every major component in milk without causing major damages to its properties. Current shelf-stable concentrated dairy products often have brown off-colors and “cooked” off-flavors due to heat-induced Maillard browning and whey protein denaturation that occur during sterilization. MF was used in this study to develop a novel concentrated milk product depleted of most of its lactose (to limit Maillard browning) and whey proteins. Acidified skim milk was concentrated to 8x microfiltration concentration factor and then diluted 1:1 with distilled water and sterilized in cans to produce a microfiltered milk concentrate (MMC).

The MMC was characterized by its chemical composition, color, apparent viscosity, flow behavior, and storage potential. Furthermore, these characteristics of the MMC were compared with a concentrated dairy product, commercial evaporated milk (CEM). The % (w/w) composition of the MMC was found to be 20.6% total solids, 13.4% true protein, 12.0% casein, 0.6% fat, 1.5% ash, 1.8% lactose and 0.7% whey proteins. It was comparable to CEM in total solids, greater in true protein, casein, and fat, and lower in ash, lactose and whey proteins. The MMC retained much more of the white color of HTST-pasteurized milk than the CEM. The apparent viscosity (~9.5 cP at 30°C) of the MMC was much greater than that of the CEM and the MMC was seen to be more pseudoplastic in nature ($n < 1$) than the CEM. The changes during 12-month storage were comparable between the CEM and MMC samples.

## Introduction

Recently membrane separation technology has gained some prominence in the dairy industry and has allowed for the development of several innovative new dairy processes and products (Nelson et al., 2005). This is due to its ability to efficiently separate many of the major components in milk without causing significant changes and/or damages to their properties (Brans et al., 2004). This study explores the possibilities of membrane technology to develop a novel concentrated milk product superior in quality and with more diverse applications as compared to current concentrated milk products. More specifically, microfiltration (MF) technology was used to develop a concentrated milk product that can not only match evaporation concentrated milk in nutritional content but also surpass it in sensory quality.

Conventionally, to make skim evaporated milk, skim milk is heated under vacuum until about 60 percent of the water has been removed, which concentrates all of its components. Then the evaporated milk is chilled and combined with additives (i.e. vitamin fortification), if any, and canned. Finally, the canned concentrated milk is sterilized to render it shelf stable for up to one year at room temperature storage. This last sterilization step is the crucial step in which the heat-sensitive components of the concentrated milk undergo various changes that result in a brown off-color and "cooked" off-flavor (Wyss et al., 2004).

The brown off-color that develops during the sterilization step is a result of Maillard reaction, which occurs between the amino acids and the reducing sugar, lactose, which are present in the milk (Tobias, 1990). The intense heat of the sterilization treatment catalyzes this reaction between the amino acids and lactose to produce brown color pigments known as melanoidins, which are ultimately responsible for the brown color in the finished product (Fennema, 1996). The "cooked" off-flavor is also a result of the heat during sterilization in which volatile sulfide and thiol compounds are released. The mechanisms for the formation of these compounds are still unclear however, it is theorized that they are formed as byproducts in the breakdown of $\beta$ - lactoglubolin and other whey proteins (Ferretti, 1973).

Despite its brown color and "cooked" flavor, evaporated milk has still maintained its own share within the marketplace due to its ability to be stored for a relatively long period of time at room temperature as compared to other milk products. Since its components are concentrated during the evaporation process, its nutritive value is greater than that of an equal serving of skim milk. Also, as mentioned previously, the sterilization process allows for the evaporated milk to be stored at room temperature for up to one year, which allows for easier distribution and handling. This makes the product more accessible to a greater percent of the world's population especially to those who do not have access to fresh or refrigerated milk.

The goal of this study was to produce a novel concentrated milk product that can deliver the same nutritive value and storage benefits as commercial evaporated milk (CEM) while retaining more of the color and taste of fresh milk. MF was used to concentrate the skim milk and for the partial removal of whey protein and lactose. Due to the depletion of most of the lactose and some of the whey proteins, it was hypothesized that the heat treatment during sterilization of the MF concentrated milk product would not induce Maillard browning and whey protein denaturation. The resulting sterilized milk concentrate should be comparable to CEM in terms of shelf stability and utilization in the practical applications of cooking and consumption. However, it would be superior in quality due to the retention of the fresh flavor and white color of milk.

## Materials and Methods

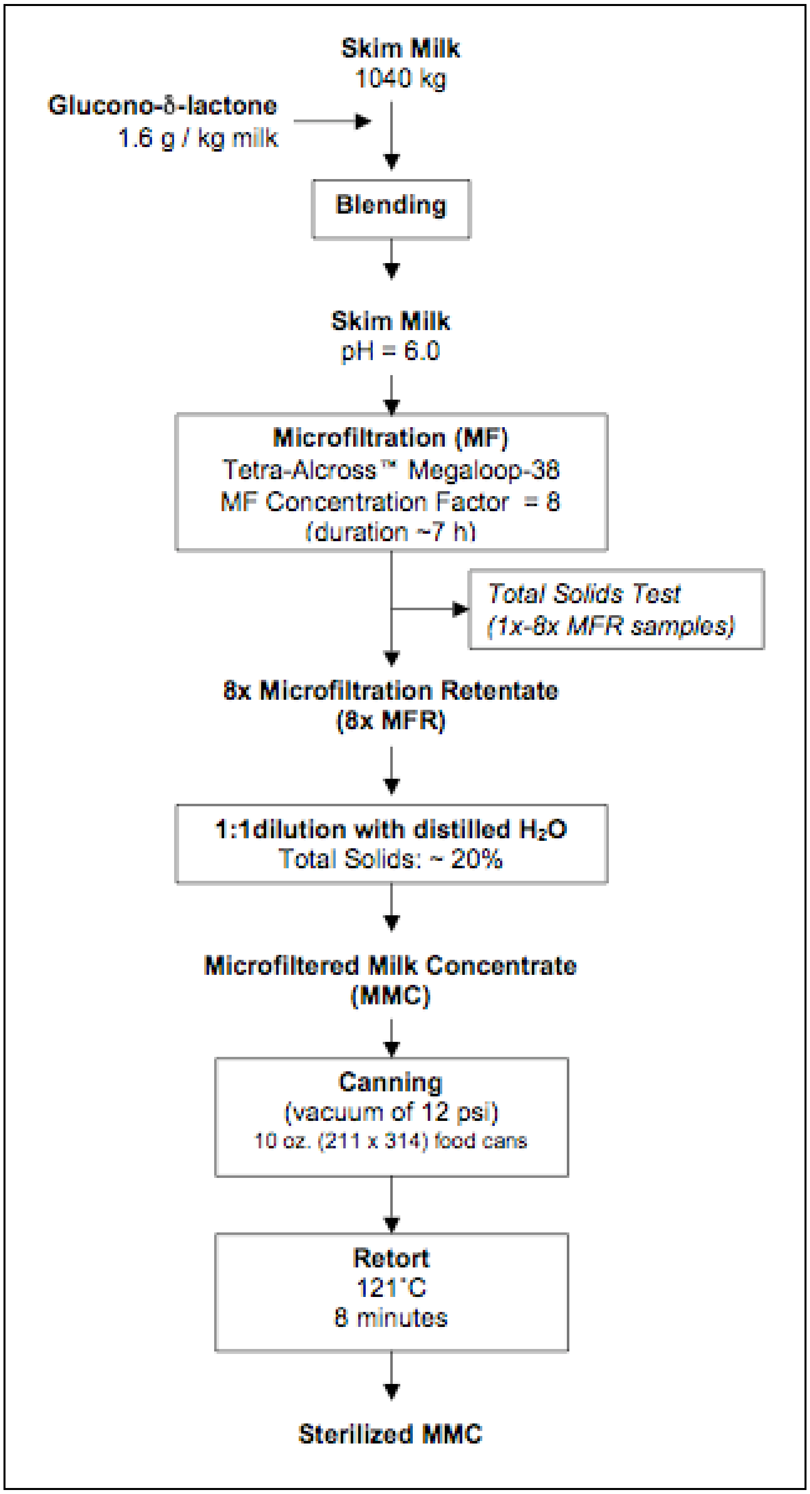


*Figure 1. Flow Diagram of Sterilized Microfiltered Milk Concentrate (MMC) production process*

*Microfiltration*

The starting material was 1040 g of HTST (high-temperature, short time) pasteurized skim milk at 4˚C obtained from Cornell Dairy (Ithaca, NY). One hour prior to microfiltration, glucono- δ -lactone was added at 1.6 g per kg of skim milk to reduce the pH of the milk to 6.0. This acidification causes the partial collapse of the hairy layer of the casein micelles and consequently the solubilization of the micellar calcium (Banon et al., 1992). The micellar calcium gets dispersed into the water phase of the skim milk thereby increasing the amount of calcium in the microfiltration permeate (MFP). Thus, the acidification in combination with the microfiltration (MF) process will cause a reduction in the calcium to casein ratio of the microfiltration retentate (MFR).

After acidification, the skim milk was concentrated to a factor of 8x (taking a total of about 7 hours) using the Tetra-Alcross™ Megaloop-38 operating at uniform transmembrane pressure of 14.5 psi at 50˚C. The Megaloop consisted of 0.1 µm ceramic membranes with total effective filtration area of 9.1 $m^2$. The permeate flux, retentate pH, retentate temperature, and retentate and permeate inlet and outlet pressures were regulated throughout the process. The flux was determined by weighing the permeate every 10 minutes. Samples for total solids analyses were gathered at 1-8x concentration factors. Once the MFR was concentrated to 8x, it was gathered and refrigerated overnight at 4˚C.

*Dilution and Sterilization*

The 8x MFR was warmed up to room temperature (~25°C) and was diluted 1:1 with distilled water. The resultant Microfiltered Milk Concentrate (MMC) was used instead of the 4x MFR obtained from the MF process due to the greater degree of lactose and whey protein depletion gained from concentrating to 8x. The MMC was canned in 10 oz. (211 x 314) food cans under vacuum pressure at 12 psi. For the retort process, the product temperature at the center of the cans (as monitored by thermocouples) were brought to 121°C and held for at 8 minutes before cooling down.

The targeted $F_0$ value range set for this study was 8-16 minutes, which far surpasses the minimum of 3.5 minutes as required by the Evaporated Milk Association (Ellertson, 1979). The over-processing of the MMC was desired to test the product's resistance to changes in color due to the extended heat treatment. The sterilizing value was obtained by calculating the area under the curve of the plot of $F_0$/min versus time (minutes). The $F_0$/min value was calculated using the equation

$$F_0/\min = \frac{1}{\log^{-1}\frac{(250-T)}{18}}$$

where T is the product temperature (°F) of the geometric center of the can (Geankoplis, 1993). The total $F_0$ value was obtained by summing the $F_0$/min values.

*Product Characterization*

The sterilized MMC samples were sent to Dairy One (Ithaca, NY) for the analyses of its chemical composition: total solids, total protein, casein, fat, lactose, and ash. The whey protein content of the samples was estimated based on data from past research as shown in the Appendix, Figure 1 (Ardisson-Korat et al., 2004).

The color of the MMC was compared with a commercial evaporated skim milk (CEM) product (Wegmans brand; produced December 2005). Color was measured in triplicate using the Macbeth Color-Eye Colorimeter at room temperature (~25˚C). The L (light/dark), a (green/red), b (blue/yellow) values were obtained and the ∆E value was calculated against the white tile standard using the equation

$$\Delta E = \sqrt{(\Delta L^2) + (\Delta a^2) + (\Delta b^2)}\,.$$

The viscosity and flow behavior of the CEM and MMC samples were measured in triplicate with a Brookfield DV-II Viscometer equipped with a UL adapter using the Wingather software program. The apparent viscosities of the CEM and MMC samples were measured at three different temperatures (20˚C, 30˚C, 40˚C) and three different shear rates (122.3s$^{-1}$, 183.4s$^{-1}$, and 244.6s$^{-1}$). The flow behavior was calculated by the Wingather program using the Power Law model

$$\eta_{app} = K\dot{\gamma}_w^{\,n-1}$$

where $\eta_{app}$ is the apparent viscosity, K is the flow constant, $\dot{\gamma}$ is the shear rate and n is the flow behavior index.

The color, apparent viscosity and flow behavior of the CEM and MMC samples were evaluated and compared after 3-month, 6-month, and 12-month storage. Additionally, the apparent viscosities of the MMC samples were also measured at 1-week and 1-month storage to further characterize some of the physical properties of this novel product. The first analyses for the CEM sample occur at 3-month storage because fresher samples could not be obtained.

Microsoft Excel software was used to calculate the averages and standard deviations for triplicate data sets. T-test was performed with SPSS 14.0 for Windows; results were considered significant for $P < 0.05$.

## Results and Discussion

A total of four different sets of Microfiltered Milk Concentrate (MMC) were produced. Each run is identified by its respective date of production; Jul. 14, 2005, Aug. 8, 2005, Sept. 8, 2005, and Feb. 8, 2007.

### *Product Sterilization*

The sterilizations of all four MMC runs were calculated to have $F_0$ values of 12.2, 16.0, 13.3 and 15.4 minutes for the Jul. 14, 2005, Aug. 8, 2005, Sept. 8, 2005, and Feb. 8, 2007 runs, respectively. These values fall within the targeted $F_0$ range of 8-16 minutes for this study (for calculations, refer to Appendix, Tables 1-4 and Figures 2-5). In addition, the obtained $F_0$ values confirm that the produced MMC met the minimum requirement of the 3.5 set forth by Evaporated Milk Association.

### *Chemical Composition*

The chemical composition (by weight % of each component) of the commercial evaporated milk (CEM) and the MMC (average of Sept. 8, 2005 and Feb 8, 2007 runs) are shown in Figure 2. The CEM had a total solids content of 20.6%, which was greater than that of the MMC at 17.0%. MMC samples had almost twice as much true protein and casein at 13% and 12%, respectively, than CEM samples at 7.5% and 6%, respectively. The MF process enabled the depletion of most of the lactose in the MMC, which had 1.8% lactose versus the 11.3% in CEM. In addition, the estimated whey protein content of the MMC (0.7%) was only half that of the CEM (1.4%).

The fat content of the MMC (0.6%) was greater than that of the CEM (0.2%). This is due to the fact that the small amount of fat present in skim milk was concentrated to a greater degree during the MF process compared with the evaporation process. For the MMC product, skim milk was concentrated to a factor of 8x and then diluted 1:1 with water to reduce the final concentration factor to 4x. On the other hand, the evaporation process removes 60% of the water in milk thereby concentrating the skim milk to a factor of only 1.67x.

Finally, the ash was lower in the MMC (1.2%) than the CEM sample (1.5%). This is because the acidification of the skim milk caused the solubilization of some of the micellar calcium, which becomes dispersed in the water phase of the milk (Banon et al., 1992). The solubilized calcium and some other minerals are then lost in the permeate as they are small enough to pass through the 0.1 μm membrane.

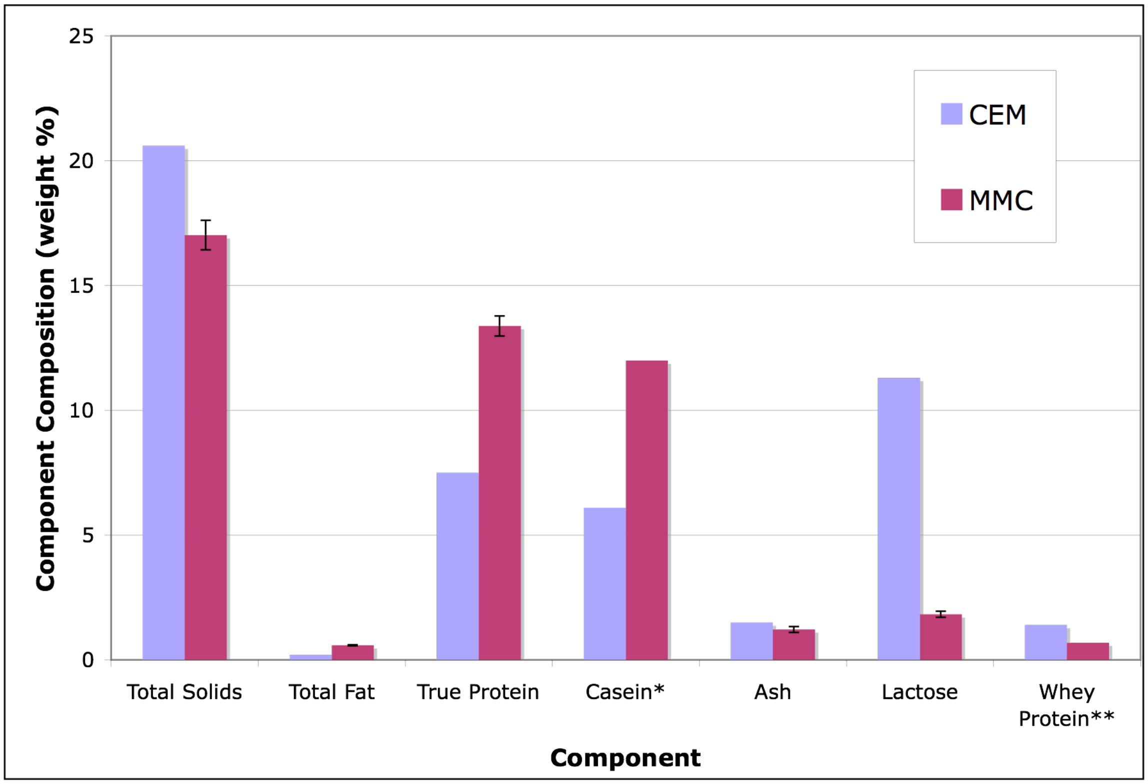


*Figure 2. Chemical composition of Commercial Evaporated Milk (CEM) and Microfiltered Milk Concentrate (MMC) by weight % of each component*

** Data from only one MMC run: Sept. 8, 2005*

***% whey protein of MMC was estimated from literature values (see Appendix, Figure 1)*

*Color*

A photographic comparison of the CEM and MMC (Jul. 14, 2005 run) is shown in Figure 3 to visually depict the color difference between the two. The samples were placed in clear test tubes and a dark blue background was used for better visual contrast. The CEM (left) was described by a panel of 5 food scientists as much darker, more yellow-brown, in color compared to the MMC (right).


*Figure 3. Photographic comparison of Commercial Evaporated Milk (left) and Microfiltered Milk Concentrate; Jul. 14, 2005 run (right)*

The calculated ∆E values for the CEM and MMR can be seen in Table 1 (for sample calculation of ∆E, refer to Appendix, Figure 6). Both the CEM and MMC were significantly different in color from the white tile standard (∆E>1). In comparison, the ∆E values for MMC were about half that of the ∆E values for CEM. This shows that the color of the MMC was much more similar to the white tile standard than the CEM.

*Table 1. Average ∆E values ± the standard deviation (from three replicates) of Commercial Evaporated Milk (CEM) and Microfiltered Milk Concentrate (MMC)*

| Storage Time | **CEM** | **MMC** |
|---|---|---|
| **1 week** | | 11.96 ± *0.06* |
| **1 month** | | 12.74 ± *0.08* |
| **3 month** | 28.67 ± *0.07* | 15.18 ± *0.07* |
| **6 month** | 32.51 ± *0.05* | 17.78 ± *0.07* |
| **12 month** | 32.45 ± *0.35* | 18.81 ± *0.01* |

Table 2 represents the L, a, b values for the CEM and MMC after 3-month storage along with the values for the white tile standard (included for comparison). The 3-month storage samples of the CEM and MMC were chosen for this comparison because it was the first comprehensive data set for both the CEM and MMC.

*Table 2. L, a, b values for Commercial Evaporated Milk (CEM) and Microfiltered Milk Concentrate (MMC) samples at 3-month storage compared with the white tile standard*

| | **White Tile** | **CEM** | **MMC** |
|---|---|---|---|
| **L:** white/black | 93.542 | 70.205 | 80.495 |
| **a:** green/red | -0.263 | 3.968 | 0.001 |
| **b:** blue/yellow | 0.5893 | 16.690 | 8.351 |

The L value indicates the white / black character of the samples where 100 is pure white and 0 is pure black. The MMC sample (L=80.495) is 10% closer to the pure white than the CEM sample (L=70.205). The a value describes the green / red character of the product. The MMC (a=0.001) is more similar to the white tile standard (a=-0.263) than the CEM (a=3.968) showing that the CEM has more red character than the MMC. Finally, the b value is used to define the blue / yellow character of a sample. Once again, MMC (b=8.351) was much closer to the white tile standard (b=0.5893) than the CEM (b=16.690). These b values show that the CEM had twice the yellow character of the MMC.

*Apparent Viscosity*

The measured apparent viscosities (cP) of the CEM and MMC (Sept. 8, 2005 run) samples at 3-month storage are shown in Figure 4. The apparent viscosities of the MMC were seen to be significantly different (P < 0.05) than those of the CEM (refer to Appendix, Table 5 for statistical tests).

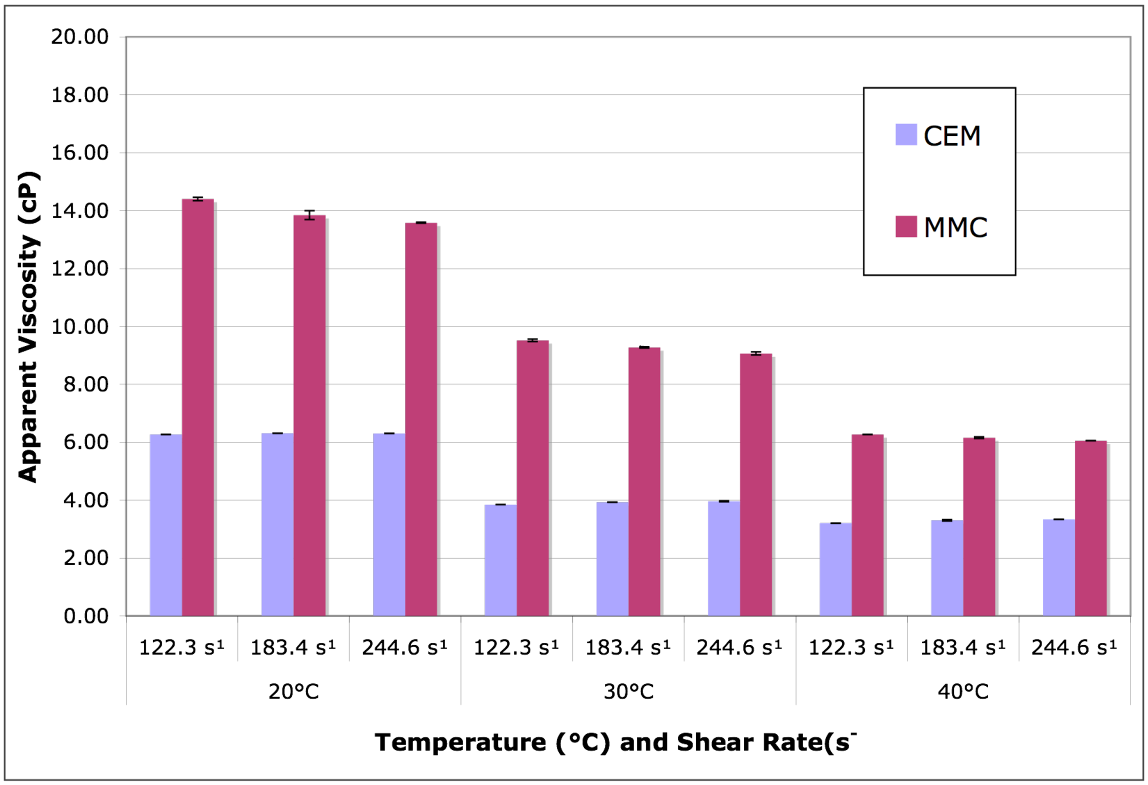


*Figure 4. Apparent viscosities (cP) of Commerial Evaporated Milk (CEM) and Microfiltered Milk Concentrate (MMC; Sept. 8, 2005 run) samples after 3-month storage (error bars signify the standard deviations from 3 replicates)*

As the temperature increased from 20°C to 40°C, the viscosity of both the CEM and the MMC decreased significantly (P < 0.05). The MMC exhibited a larger decrease (8 cP) in apparent viscosity as the temperature increased by 20°C showing a greater sensitivity to temperature compared to the CEM (3 cP decrease). As a result, there was less of a difference in viscosity between the CEM and MMC as the temperature increased. However, even at 40°C, the viscosity of the MMC (6.3 cP) was almost double that of the CEM (3.2 cP).

The MMC showed a decrease in apparent viscosity as the shear rate increased. At 20°C, there was about a 1 cP decrease as the shear rate increased from 122.3$s^{-1}$ to 244.6$s^{-1}$; at 30°C it was 0.2 CP and at 40°C it 0.1 cP. On the other hand, the CEM sample showed an increase in apparent viscosity as the shear rate increased. At 20°C, 30°C, and 40°C the viscosity increased by 0.5 cP, 1.0 cP, and 1.5 cP, respectively, as the shear rate increased.

*Flow Behavior*

Using the Power Law, the K (flow constant) and n (flow behavior index) were calculated and these values for the CEM and MMC are shown in Table 3. The MMC had much greater K values compared to the CEM, indicating that it is a much more viscous fluid. For both the CEM and MMC, the K values decreased steadily as the temperature was raised from 20 to 40°C. The CEM samples had K values of 4.7, 2.8, and 2.1 (Pa•$s^n$) for 20°C, 30°C, and 40°C, respectively, showing a total decrease of 2.6 Pa•$s^n$. The MMC, however, at these same temperatures had K values of 21.3, 12.9, and 7.0 Pa•$s^n$, respectively, showing a net decrease of 14.3 Pa•$s^n$.

*Table 3. Average K (flow constant) and n (flow behavior index) values ± standard deviation (from 3 replicates) for the Commercial Evaporated Milk (CEM) and Microfiltered Milk Concentrate (MMC; Sept. 8, 2005) at 3-month storage*

| | | CEM | MMC |
|---|---|---|---|
| **20*C** | **K** (Pa•$s^n$) | 4.71 ± 0.00 | 21.33 ± 0.15 |
| | **n** | 1.06 ± 0.00 | 0.92 ± 0.00 |
| **30*C** | **K** (Pa•$s^n$) | 2.77 ± 0.01 | 12.87 ± 0.06 |
| | **n** | 1.07 ± 0.00 | 0.94 ± 0.00 |
| **40*C** | **K** (Pa•$s^n$) | 2.08 ± 0.01 | 7.00 ± 0.01 |
| | **n** | 1.09 ± 0.00 | 0.98 ± 0.00 |

The n values for the CEM were 1.06, 1.07 and 1.09 for the temperatures of 20, 30, and 40°C, respectively. Since the n value is greater than 1, this indicates CEM is slightly dilatant. The MMC on the other hand displayed slightly pseudoplastic behavior (n < 1) with values of 0.92, 0.94, and 0.98 at 20, 30, and 40°C, respectively. The n values for both CEM and MMC increased slightly as the temperature of the sample increased. Therefore, as the temperature increases, the CEM samples behave more dilatant and the MMC samples behave more like a Newtonian liquid.

*Storage*

The changes in the ∆E of the CEM and MMR over a period of one year are shown in Figure 5. As the storage time increases, both the CEM and MMC samples show significant changes (P < 0.05) in the ∆E value (refer to the Appendix, Tables 8-9 for statistical tests). The ∆E for both CEM and MMC increase over time indicating that the colors of both samples become more different from the white tile standard. In comparison the degree of increase for the CEM and MMC were very close at 3.78 and 3.63, respectively, so it can be concluded that they undergo similar changes in color over the duration of the 12-month storage.

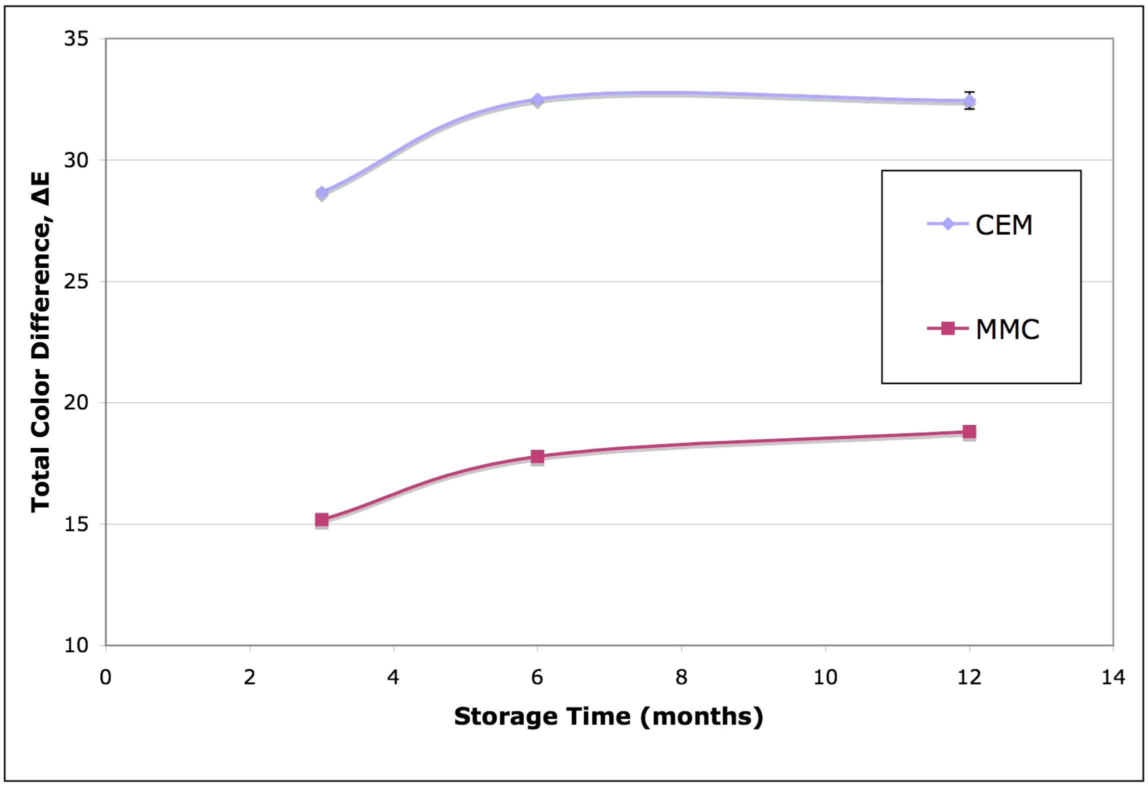


*Figure 5. Changes in the total color difference, ΔE, of Commercial Evaporated Milk (CEM) and Microfiltered Milk Concentrate (MMC; Sept. 8, 2005 run) over the duration of 12-month storage (error bars indicate the standard deviation from 3 replicates)*

The changes in the L, a, b values are shown in Figure 6. The changes in the L values for both the CEM and MMC did not change very much during the 12-month storage period. The a and b values, on the other hand, increased over time for both indicating the development of more red and yellow character for both products. These increases were very similar for both CEM and MMC over the 12-month period.

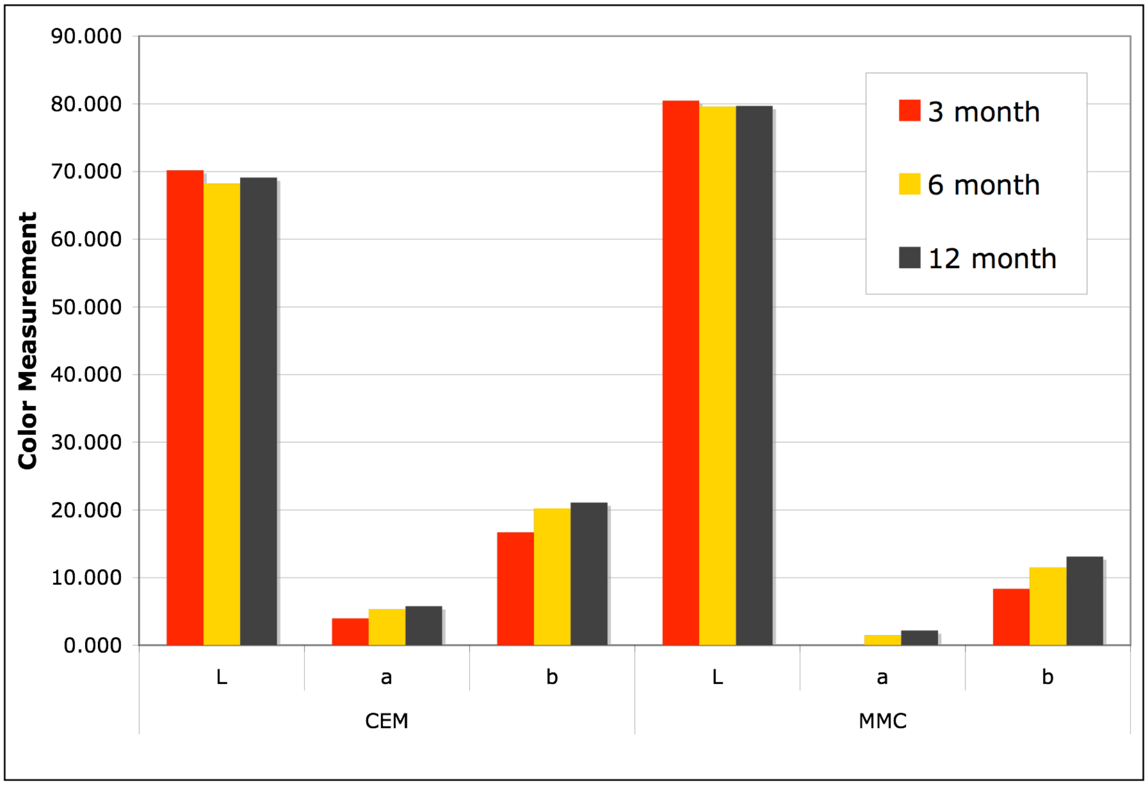


*Figure 6. Changes in L, a, b values of Commercial Evaporated Milk (CEM) and Microfiltered Milk Concentrate (MMC; Sept. 8, 2005 run) over 12-month storage*

The changes in apparent viscosity over time for the CEM and MMC can be seen in Figure 7. The 30°C measurement was taken since it most closely resembles room temperature, the temperature that both products would be stored and served. Both the CEM and MMC products showed significant changes ($P < 0.05$) in apparent viscosity over the 12-month storage period (refer to Appendix, Tables 10-11 for statistical tests). In comparison the MMC showed much less change in viscosity (net increase of 0.08 cP) over the 12-month period in comparison to the CEM sample, which had a net increase of 0.69 cP.

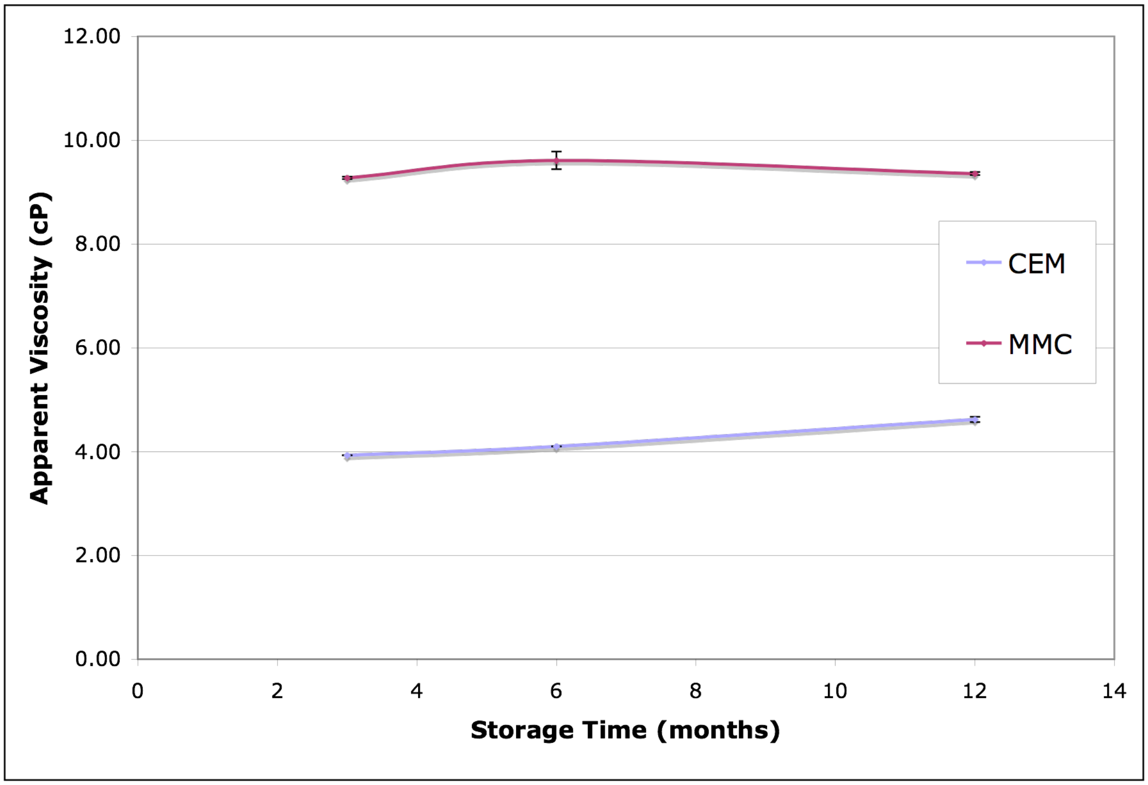


*Figure 7. Changes in apparent viscosity (cP) at 30°C and 183.4 $s^{-1}$ shear rate for Commercial Evaporated Milk (CEM) and Microfiltered Milk Concentrate (MMC; Sept. 8, 2005 run) over the 12-month storage (error bars signify the standard deviation from 3 replicates)*

The changes in apparent viscosity over the 12-month storage period for the MMC are shown in Figure 8 (also refer to Appendix, Table 12 for tabulated values). The apparent viscosities of the MMC samples increase steadily over time. The samples measured at 20°C showed the greatest change over the 12-month period. The samples measured at 30 and 40°C were similar and showing a slow but steady rise in apparent viscosity over time.

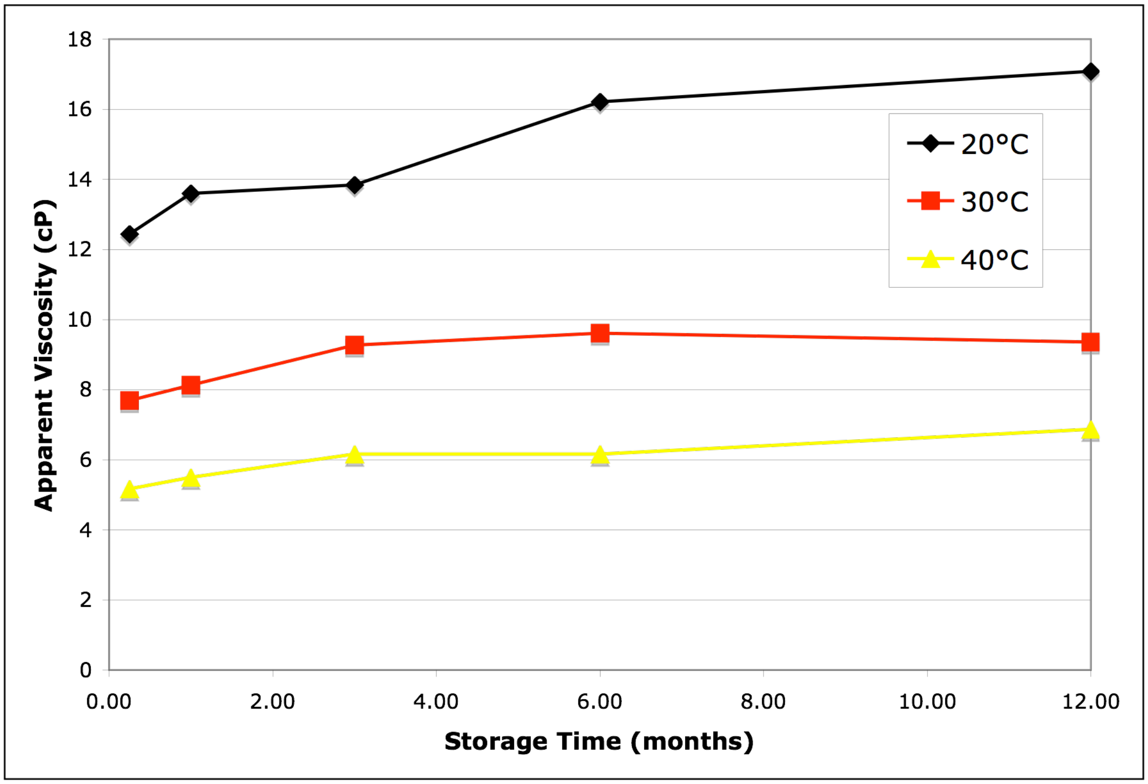


*Figure 8. Changes in apparent viscosity (cP) at 183.4 $s^{-1}$ shear rate and 20, 30, and 40°C for Microfiltered Milk Concentrate (MMC; all four runs) from 1-week to 12-month storage*

The changes in the K (flow constant) and n (flow behavior index) over the 12-month storage period can be seen in Tables 4 and 5, respectively. The K value remains relatively constant for the MMC samples though they do experience a slight decrease at the 6-month storage mark. The K for the CEM sample increases steadily over time however it does slow down between the 6-month and 12-month storage period.

*Table 4. Changes in K (flow constant; Pa•s$^n$) ± the standard deviation (for 3 replicates) at 30°C of Commercial Evaporated Milk (CEM) and Microfiltered Milk Concentrate (MMC; Sept. 8, 2005) samples over the 12-month storage period*

| Storage Time | CEM | MMC |
|---|---|---|
| 3 month | 2.77 ± *0.01* | 12.87 ± *0.06* |
| 6 month | 4.36 ± *0.02* | 10.83 ± *0.70* |
| 12 month | 4.61 ± *0.24* | 13.00 ± *0.46* |

The n for the CEM sample approaches 1 as storage time increases indicating that it will behave like a Newtonian liquid as confirmed in previous research (Vélez-Ruiz et al., 1998). MMC sample continues to have n < 1, indicating that it will continue to be slightly pseudoplastic.

*Table 5. Changes in n (flow behavior index) ± the standard deviation (for 3 replicates) at 30°C of Commercial Evaporated Milk (CEM) and Microfiltered Milk Concentrate (MMC; Sept. 8, 2005) samples over the 12-month storage period*

| Storage Time | CEM | MMC |
|---|---|---|
| 3 month | 1.07 ± *0.00* | 0.94 ± *0.00* |
| 6 month | 0.99 ± *0.00* | 0.98 ± *0.01* |
| 12 month | 1.00 ± *0.01* | 0.94 ± *0.01* |

## Conclusions

The unique properties of the novel microfiltered milk concentrate (MMC) produced in this study allow it to serve a variety of applications. The MMC can be a possible alternative to commercial evaporated milk (CEM) especially for those who dislike the darker color and “cooked” flavor of the current product. The lactose and whey proteins were greatly depleted during the microfiltration (MF) concentration process thereby limiting Maillard browning and protein denaturation. This allowed for the better retention of the white color and fresh flavor of the HTST pasteurized skim milk. There were some rheological differences between the CEM and MMC in which the MMC had a greater viscosity and was more pseudoplastic in nature. All in all, the changes in color, viscosity, and flow behavior of the MMC product were comparable to CEM throughout the duraton of the 12-month storage.

Another possible application for this MMC product is its use as a high-protein dietary supplement due to its increased protein content from the MF concentration of casein protein. The unique chemical profile of this product offers nutritive benefits that current concentrated dairy products are not able to. The MMC would be particularly useful for the populations that do not have access to fresh milk and refrigeration. Since this product is highly concentrated, it can also deliver nutrients more per serving.

These are only a few suggestions in the wide gamut of possibilities that this unique product can offer. More research is needed to further characterize this novel product especially in its sensory quality.

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## Appendix

There is 1.37% (w/w) whey protein in 8x MFR (Ardisson-Korat et al., 2004).

½ (% whey protein in 8x MFR) = % whey protein in MMC

½ (1.37%) = 0.69%

*Figure 1. Calculation of % whey protein in Microfiltered Milk Concentrate (MMC)*

*Table 1. $F_o$ calculations for Microfiltered Milk Concentrate (MMC; Jul. 14, 2005 run)*

| Time (min) | Temp (F) | Target Temp | (250-T)/18 | log-1[(250-T)/18] | F0/min |
|---|---|---|---|---|---|
| 0 | 99.7 | 250.0 | 8.350 | 223872113.9 | 0.000 |
| 2 | 204.6 | 250.0 | 2.522 | 332.8298139 | 0.003 |
| 3 | 222.0 | 250.0 | 1.556 | 35.93813664 | 0.028 |
| 6 | 247.8 | 250.0 | 0.122 | 1.325019355 | 0.755 |
| 7 | 250.2 | 250.0 | -0.011 | 0.974740226 | 1.026 |
| 9 | 250.8 | 250.0 | -0.044 | 0.902725178 | 1.108 |
| 13 | 250.8 | 250.0 | -0.044 | 0.902725178 | 1.108 |
| 15 | 250.8 | 250.0 | -0.044 | 0.902725178 | 1.108 |
| 17 | 142.5 | 250.0 | 5.972 | 938041.8666 | 0.000 |
| 19 | 109.0 | 250.0 | 7.833 | 68129206.91 | 0.000 |
| 20 | 108.1 | 250.0 | 7.883 | 76442227.43 | 0.000 |
| 22 | 107.7 | 250.0 | 7.906 | 80455466.17 | 0.000 |
| 30 | 104.0 | 250.0 | 8.111 | 129154966.5 | 0.000 |
| 32 | 103.3 | 250.0 | 8.150 | 141253754.5 | 0.000 |

***Total $F_0$ = 12.186 minutes***

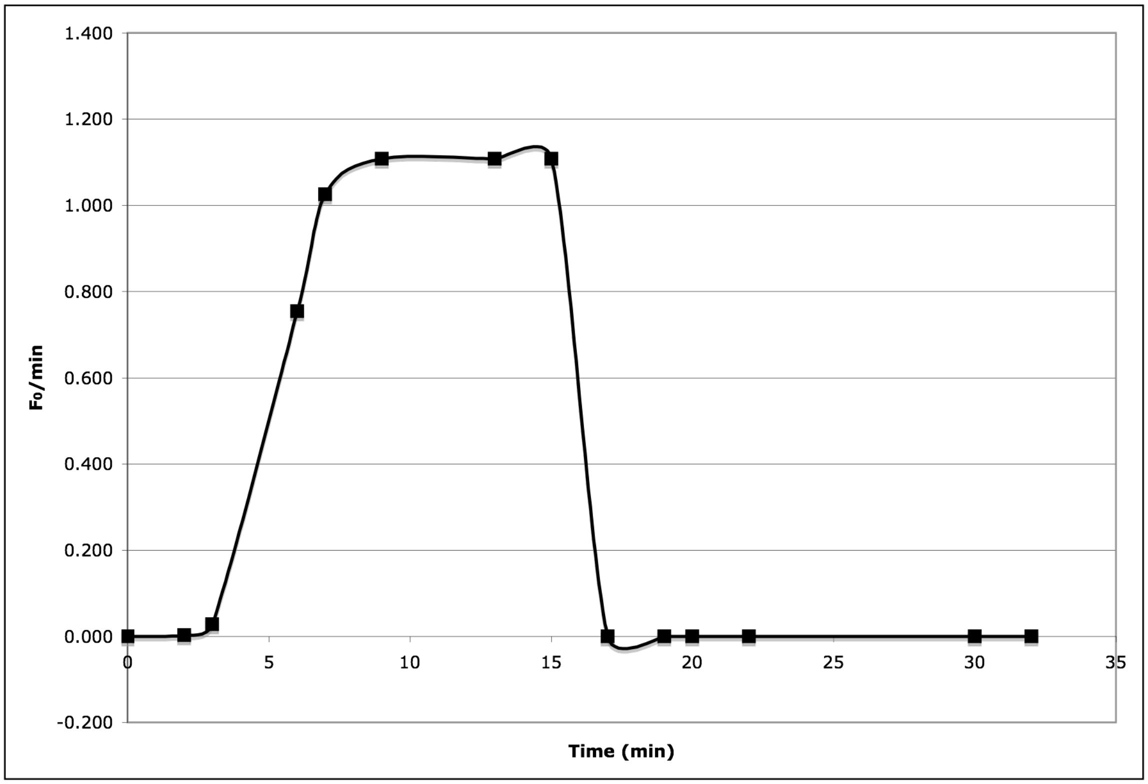


*Figure 1. Graph of $F_0$/min versus Time for Microfiltered Milk Concentrate (MMC; Jul 14, 2005 run)*

*Table 2. $F_o$ calculations for Microfiltered Milk Concentrate (MMC; Aug. 9, 2005 run)*

| Time (min) | Temp (F) | Target Temp | (250-T)/18 | log-1[(250-T)/18] | F0/min |
|---|---|---|---|---|---|
| 0 | 85.1 | 250.0 | 9.161 | 1449142559 | 0.000 |
| 1 | 111.9 | 250.0 | 7.672 | 47013460.82 | 0.000 |
| 2 | 150.9 | 250.0 | 5.506 | 320298.9799 | 0.000 |
| 3 | 182.7 | 250.0 | 3.739 | 5481.367102 | 0.000 |
| 4 | 208.0 | 250.0 | 2.333 | 215.443469 | 0.005 |
| 5 | 224.3 | 250.0 | 1.428 | 26.77797784 | 0.037 |
| 6 | 232.6 | 250.0 | 0.967 | 9.261187281 | 0.108 |
| 7 | 239.3 | 250.0 | 0.594 | 3.930469626 | 0.254 |
| 8 | 246.6 | 250.0 | 0.189 | 1.544859148 | 0.647 |
| 9 | 247.5 | 250.0 | 0.139 | 1.376857165 | 0.726 |
| 10 | 248.6 | 250.0 | 0.078 | 1.196128333 | 0.836 |
| 11 | 249.4 | 250.0 | 0.033 | 1.079775162 | 0.926 |
| 12 | 249.6 | 250.0 | 0.022 | 1.052500285 | 0.950 |
| 13 | 249.8 | 250.0 | 0.011 | 1.025914365 | 0.975 |
| 14 | 250.0 | 250.0 | 0.000 | 1 | 1.000 |
| 15 | 250.1 | 250.0 | -0.006 | 0.987289332 | 1.013 |
| 16 | 250.4 | 250.0 | -0.022 | 0.950118507 | 1.053 |
| 17 | 250.3 | 250.0 | -0.017 | 0.962350626 | 1.039 |
| 18 | 250.3 | 250.0 | -0.017 | 0.962350626 | 1.039 |
| 19 | 250.3 | 250.0 | -0.017 | 0.962350626 | 1.039 |
| 20 | 250.4 | 250.0 | -0.022 | 0.950118507 | 1.053 |
| 21 | 250.5 | 250.0 | -0.028 | 0.938041867 | 1.066 |
| 22 | 250.4 | 250.0 | -0.022 | 0.950118507 | 1.053 |
| 23 | 250.4 | 250.0 | -0.022 | 0.950118507 | 1.053 |
| 24 | 230.6 | 250.0 | 1.078 | 11.96128333 | 0.084 |
| 25 | 177.1 | 250.0 | 4.050 | 11220.18454 | 0.000 |
| 26 | 148.0 | 250.0 | 5.667 | 464158.8834 | 0.000 |
| 27 | 124.7 | 250.0 | 6.961 | 9143471.407 | 0.000 |
| 28 | 113.1 | 250.0 | 7.606 | 40323252.52 | 0.000 |
| 29 | 105.4 | 250.0 | 8.033 | 107977516.2 | 0.000 |
| 30 | 104.5 | 250.0 | 8.083 | 121152765.9 | 0.000 |
| 31 | 103.0 | 250.0 | 8.167 | 146779926.8 | 0.000 |
| 32 | 101.4 | 250.0 | 8.256 | 180117352.8 | 0.000 |
| 33 | 99.1 | 250.0 | 8.383 | 241731548.1 | 0.000 |
| 34 | 95.1 | 250.0 | 8.606 | 403232525.2 | 0.000 |
| 35 | 94.7 | 250.0 | 8.628 | 424402347.8 | 0.000 |

***Total $F_0$ = 15.955***

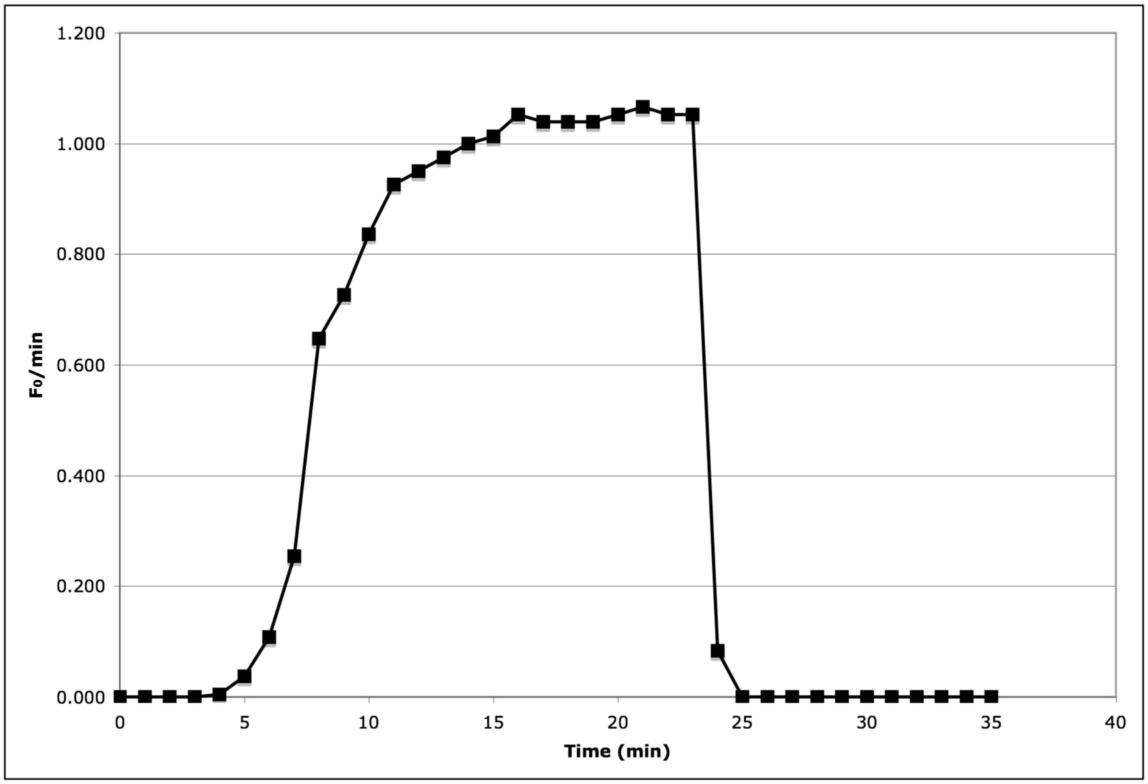


*Figure 3. Graph of $F_0$/min versus Time for Microfiltered Milk Concentrate (MMC; Aug. 9, 2005 run)*

*Table 3. $F_o$ calculations for Microfiltered Milk Concentrate (MMC; Sept. 2, 2005 run)*

| Time (min) | Temp (F) | Target Temp | (250-T)/18 | log-1[(250-T)/18] | F0/min |
|---|---|---|---|---|---|
| 0 | 100.1 | 250.0 | 8.328 | 212705038.6 | 0.000 |
| 1 | 100.1 | 250.0 | 8.328 | 212705038.6 | 0.000 |
| 2 | 110.2 | 250.0 | 7.767 | 58434141.34 | 0.000 |
| 3 | 131.9 | 250.0 | 6.561 | 3640081.531 | 0.000 |
| 4 | 156.8 | 250.0 | 5.178 | 150583.6354 | 0.000 |
| 5 | 181.4 | 250.0 | 3.811 | 6473.082037 | 0.000 |
| 6 | 199.2 | 250.0 | 2.822 | 664.0827851 | 0.002 |
| 7 | 206.1 | 250.0 | 2.439 | 274.7191214 | 0.004 |
| 8 | 209.1 | 250.0 | 2.272 | 187.1639586 | 0.005 |
| 9 | 215.7 | 250.0 | 1.906 | 80.45546617 | 0.012 |
| 10 | 223.4 | 250.0 | 1.478 | 30.0453853 | 0.033 |
| 11 | 228.4 | 250.0 | 1.200 | 15.84893192 | 0.063 |
| 12 | 233.4 | 250.0 | 0.922 | 8.360306937 | 0.120 |
| 13 | 237.6 | 250.0 | 0.689 | 4.885273572 | 0.205 |
| 14 | 240.9 | 250.0 | 0.506 | 3.202989799 | 0.312 |
| 15 | 244.1 | 250.0 | 0.328 | 2.127050386 | 0.470 |
| 16 | 247.0 | 250.0 | 0.167 | 1.467799268 | 0.681 |
| 17 | 249.5 | 250.0 | 0.028 | 1.066050499 | 0.938 |
| 18 | 250.6 | 250.0 | -0.033 | 0.926118728 | 1.080 |
| 19 | 250.4 | 250.0 | -0.022 | 0.950118507 | 1.053 |
| 20 | 250.6 | 250.0 | -0.033 | 0.926118728 | 1.080 |
| 21 | 250.6 | 250.0 | -0.033 | 0.926118728 | 1.080 |
| 22 | 250.8 | 250.0 | -0.044 | 0.902725178 | 1.108 |
| 23 | 250.8 | 250.0 | -0.044 | 0.902725178 | 1.108 |
| 24 | 251.1 | 250.0 | -0.061 | 0.86873814 | 1.151 |
| 25 | 251.3 | 250.0 | -0.072 | 0.846794011 | 1.181 |
| 26 | 251.5 | 250.0 | -0.083 | 0.825404185 | 1.212 |
| 27 | 241.7 | 250.0 | 0.461 | 2.891419537 | 0.346 |
| 28 | 217.0 | 250.0 | 1.833 | 68.12920691 | 0.015 |
| 29 | 189.4 | 250.0 | 3.367 | 2326.305067 | 0.000 |
| 30 | 166.7 | 250.0 | 4.628 | 42440.23478 | 0.000 |
| 32 | 151.1 | 250.0 | 5.494 | 312208.2999 | 0.000 |
| 33 | 131.7 | 250.0 | 6.572 | 3734411.934 | 0.000 |
| 34 | 124.1 | 250.0 | 6.994 | 9872893.322 | 0.000 |

***Total $F_0$ = 13.257***

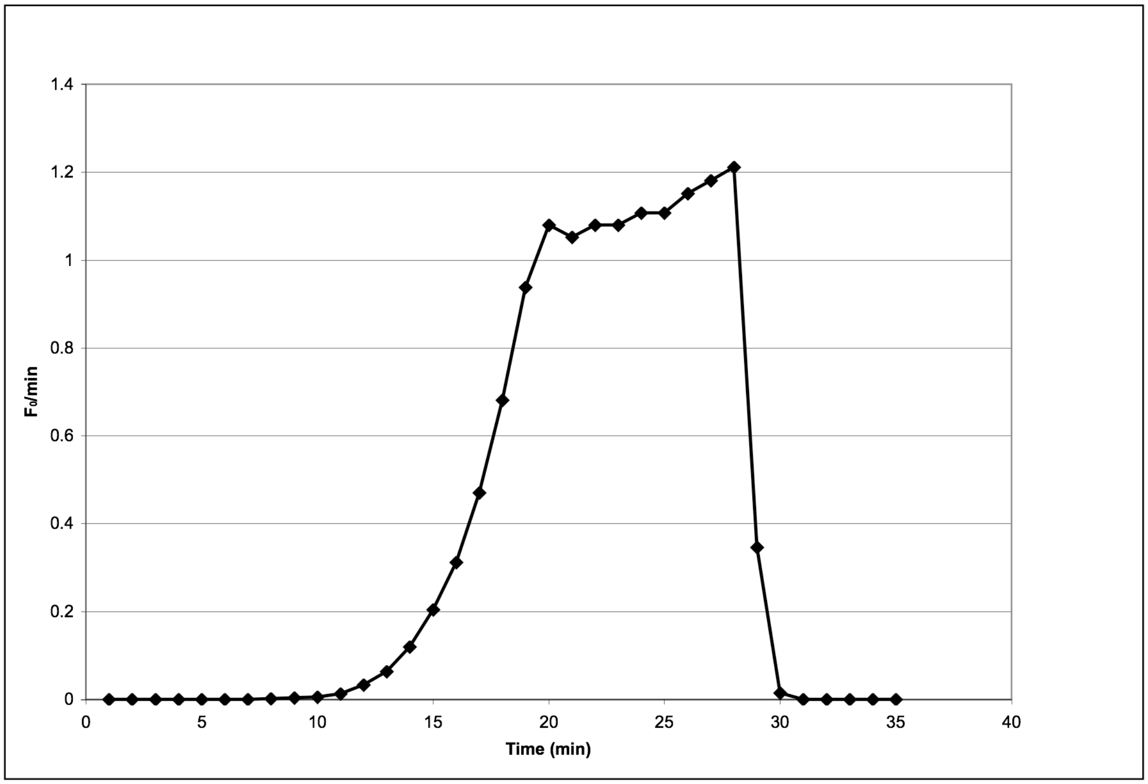


*Figure 4. Graph of $F_0$/min versus Time for Microfiltered Milk Concentrate (MMC; Sept. 2, 2005)*

*Table 4. $F_0$ calculations for Microfiltered Milk Concentrate (MMC; Feb. 8, 2007)*

| Time (min) | Temp (F) | Target Temp | (250-T)/18 | log-1[(250-T)/18] | F0/min |
|---|---|---|---|---|---|
| 0.00 | 78.97 | 250.0 | 9.502 | 3174436665 | 0.000 |
| 1.00 | 79.05 | 250.0 | 9.497 | 3142116058 | 0.000 |
| 2.00 | 91.85 | 250.0 | 8.786 | 611098350 | 0.000 |
| 3.00 | 146.85 | 250.0 | 5.731 | 537719.2145 | 0.000 |
| 4.00 | 188.14 | 250.0 | 3.437 | 2733.170135 | 0.000 |
| 5.00 | 200.23 | 250.0 | 2.765 | 582.1032178 | 0.002 |
| 6.00 | 206.25 | 250.0 | 2.431 | 269.4980056 | 0.004 |
| 7.00 | 208.92 | 250.0 | 2.282 | 191.5235673 | 0.005 |
| 8.00 | 210.07 | 250.0 | 2.218 | 165.3230212 | 0.006 |
| 9.00 | 229.44 | 250.0 | 1.142 | 13.87465593 | 0.072 |
| 10.00 | 241.76 | 250.0 | 0.458 | 2.869312021 | 0.349 |
| 11.00 | 246.26 | 250.0 | 0.208 | 1.613532724 | 0.620 |
| 12.00 | 248.82 | 250.0 | 0.066 | 1.162935306 | 0.860 |
| 13.00 | 249.77 | 250.0 | 0.013 | 1.029859022 | 0.971 |
| 14.00 | 250.41 | 250.0 | -0.023 | 0.94890388 | 1.054 |
| 15.00 | *250.74* | 250.0 | -0.041 | 0.909680508 | 1.099 |
| 16.00 | *251.00* | 250.0 | -0.056 | 0.879922544 | 1.136 |
| 17.00 | *251.19* | 250.0 | -0.066 | 0.858793777 | 1.164 |
| 18.00 | *251.23* | 250.0 | -0.068 | 0.854410677 | 1.170 |
| 19.00 | *251.29* | 250.0 | -0.072 | 0.847877935 | 1.179 |
| 20.00 | *251.32* | 250.0 | -0.073 | 0.844630319 | 1.184 |
| 21.00 | *251.36* | 250.0 | -0.076 | 0.840319505 | 1.190 |
| 22.00 | *251.41* | 250.0 | -0.078 | 0.834961915 | 1.198 |
| 23.00 | *251.39* | 250.0 | -0.077 | 0.83710084 | 1.195 |
| 24.00 | 249.44 | 250.0 | 0.031 | 1.07426422 | 0.931 |
| 25.00 | 198.29 | 250.0 | 2.873 | 746.066909 | 0.001 |
| 26.00 | 168.92 | 250.0 | 4.504 | 31948.05655 | 0.000 |
| 27.00 | 146.72 | 250.0 | 5.738 | 546736.1343 | 0.000 |
| 28.00 | 127.71 | 250.0 | 6.794 | 6221410.945 | 0.000 |
| 29.00 | 115.34 | 250.0 | 7.481 | 30276879.41 | 0.000 |
| 30.00 | 106.35 | 250.0 | 7.981 | 95621500.76 | 0.000 |
| 31.00 | 98.33 | 250.0 | 8.426 | 266754104.8 | 0.000 |
| 32.00 | 91.64 | 250.0 | 8.798 | 627737072.6 | 0.000 |
| 33.00 | 86.67 | 250.0 | 9.074 | 1185465417 | 0.000 |
| 34.00 | 82.86 | 250.0 | 9.286 | 1929992208 | 0.000 |
| 35.00 | 79.31 | 250.0 | 9.483 | 3039329447 | 0.000 |

***Total $F_0$ = 15.391***

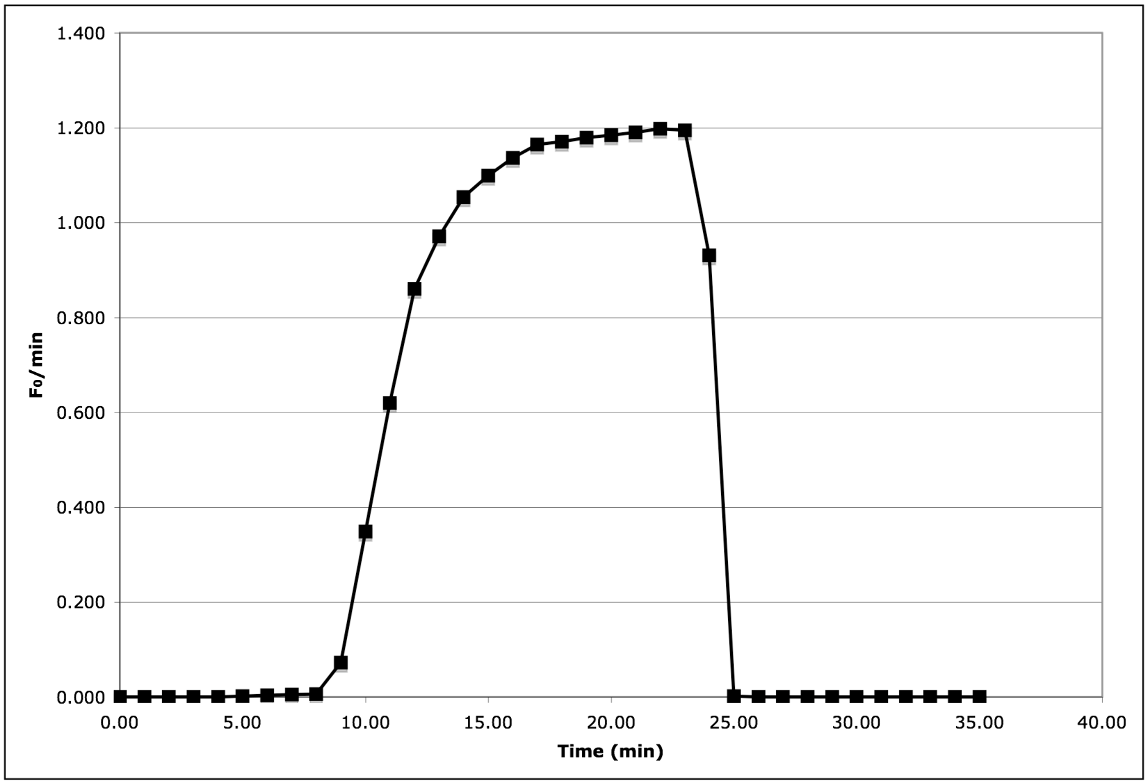


*Figure 5. Graph of $F_0$.min versus Time for Microfiltered Milk Concentrate (MMC; Feb. 8, 2007)*

$$\Delta E = \sqrt{(\Delta L^2) + (\Delta a^2) + (\Delta b^2)}$$
$$\Delta E = \sqrt{(70.205 - 93.542)^2 + (3.968 + 0.263)^2 + (16.690 - 0.5893)^2}$$
$$\Delta E = \sqrt{(-23.340)^2 + (4.230)^2 + (16.100)^2}$$
$$\Delta E = \sqrt{544.76 + 17.89 + 259.21}$$
$$\Delta E = \sqrt{821.86}$$
$$\Delta E = 28.67$$

*Figure 6. Sample calculation for ΔE using the L, a, b values from the 3-month storage sample of Commercial Evaporated Milk (CEM)*

*Table 5. T-test results for apparent viscosity (cP) comparison of 3-month storage samples of Commercial Evaporated Milk (CEM) and Microfiltered Milk Concentrate (MMC; Sept. 14, 2005 run)*

| | *CEM* | *MMC* |
|---|---|---|
| Mean | 4.493703704 | 9.795185185 |
| Variance | 1.89895679 | 11.56118086 |
| Observations | 9 | 9 |
| Pearson Correlation | 0.973308332 | |
| Hypothesized Mean Difference | 0 | |
| df | 8 | |
| t Stat | -7.635073761 | |
| P(T<=t) one-tail | 3.05073E-05 | |
| t Critical one-tail | 1.859548033 | |
| P(T<=t) two-tail | 6.10145E-05 | |
| t Critical two-tail | 2.306004133 | |

*Table 6. T-test results for effect of temperature on apparent viscosity (cP) in a 3-month storage sample of Commercial Evaporated Milk (CEM)*

| | *20°C* | *40°C* |
|---|---|---|
| Mean | 6.305 | 3.316666667 |
| Variance | 5E-05 | 0.000355556 |
| Observations | 2 | 2 |
| Pearson Correlation | -1 | |
| Hypothesized Mean Difference | 0 | |
| df | 1 | |
| t Stat | 163 | |
| P(T<=t) one-tail | 0.001952797 | |
| t Critical one-tail | 6.313751514 | |
| P(T<=t) two-tail | 0.003905594 | |
| t Critical two-tail | 12.70620473 | |

*Table 7. T-test results for effect of temperature on apparent viscosity (cP) in a 3-month storage sample of Microfiltered Milk Concentrate (MMC; Sept. 14, 2005 run)*

| | *20°C* | *40°C* |
|---|---|---|
| Mean | 13.71166667 | 6.103333333 |
| Variance | 0.034672222 | 0.005688889 |
| Observations | 2 | 2 |
| Pearson Correlation | 1 | |
| Hypothesized Mean Difference | 0 | |
| Df | 1 | |
| t Stat | 97.12765957 | |
| P(T<=t) one-tail | 0.003277116 | |
| t Critical one-tail | 6.313751514 | |
| P(T<=t) two-tail | 0.006554233 | |
| t Critical two-tail | 12.70620473 | |

*Table 8. T-test results for effect of storage time (months) on the ∆E value of Commercial Evaporated Milk (CEM)*

| | *3 month* | *12 month* |
|---|---|---|
| Mean | 28.66624797 | 32.42834382 |
| Variance | 0.004323235 | 0.160535028 |
| Observations | 4 | 4 |
| Pearson Correlation | -0.619983093 | |
| Hypothesized Mean Difference | 0 | |
| Df | 3 | |
| t Stat | -16.92970384 | |
| P(T<=t) one-tail | 0.000224422 | |
| t Critical one-tail | 2.353363435 | |
| P(T<=t) two-tail | 0.000448843 | |
| t Critical two-tail | 3.182446305 | |

*Table 9. T-test results for effect of storage time (months) on the $\Delta E$ value of Microfiltered Milk Concentrate (MMC; Sept. 8, 2005 run)*

| | *3 month* | *12 month* |
|---|---|---|
| Mean | 15.18393584 | 18.81261123 |
| Variance | 0.004279479 | 7.41297E-05 |
| Observations | 4 | 4 |
| Pearson Correlation | -0.12854262 | |
| Hypothesized Mean Difference | 0 | |
| df | 3 | |
| t Stat | -108.2053256 | |
| P(T<=t) one-tail | 8.70084E-07 | |
| t Critical one-tail | 2.353363435 | |
| P(T<=t) two-tail | 1.74017E-06 | |
| t Critical two-tail | 3.182446305 | |

*Table 10. T-test results for effect of storage time (months) on the apparent viscosity (cP) of Commercial Evaporated Milk (CEM)*

| | *3 month* | *12 month* |
|---|---|---|
| Mean | 3.93 | 4.623333333 |
| Variance | 0 | 0.002133333 |
| Observations | 3 | 3 |
| Pearson Correlation | | |
| Hypothesized Mean Difference | 0 | |
| df | 2 | |
| t Stat | -26 | |
| P(T<=t) one-tail | 0.000738008 | |
| t Critical one-tail | 2.91998558 | |
| P(T<=t) two-tail | 0.001476016 | |
| t Critical two-tail | 4.30265273 | |

*Table 11. T-test results for effect of storage time (months) on the apparent viscosity (cP) of Microfiltered Milk Concentrate (MMC; Sept. 8, 2005 run)*

| | *3 month* | *12 month* |
|---|---|---|
| Mean | 9.273333333 | 9.356666667 |
| Variance | 0.000533333 | 0.000833333 |
| Observations | 3 | 3 |
| Pearson Correlation | 1 | |
| Hypothesized Mean Difference | 0 | |
| df | 2 | |
| t Stat | -25 | |
| P(T<=t) one-tail | 0.000798085 | |
| t Critical one-tail | 2.91998558 | |
| P(T<=t) two-tail | 0.00159617 | |
| t Critical two-tail | 4.30265273 | |

*Table 12. Average of measured apparent viscosities (cP) for Microfiltered Milk Concentrate (MMC; all for runs) based on temperature (°C), shear rate ($s^{-1}$), and storage time*

| | | Shear Rate | | |
|---|---|---|---|---|
| Temperature | *Storage Time* | **122.3 s¹** | **183.4 s¹** | **244.6 s¹** |
| **20°C** | **1 week** | 12.48 | 12.44 | 12.27 |
| | **1 month** | 13.97 | 13.60 | 13.16 |
| | **3 month** | 14.40 | 13.84 | 13.58 |
| | **6 month** | 16.74 | 16.21 | 15.79 |
| | **12 month** | 17.73 | 17.08 | 16.63 |
| | | | | |
| **30°C** | **1 week** | 7.76 | 7.69 | 7.63 |
| | **1 month** | 8.40 | 8.13 | 7.94 |
| | **3 month** | 9.52 | 9.27 | 9.07 |
| | **6 month** | 9.77 | 9.61 | 9.51 |
| | **12 month** | 9.56 | 9.36 | 9.17 |
| | | | | |
| **40°C** | **1 week** | 5.22 | 5.17 | 5.19 |
| | **1 month** | 5.53 | 5.50 | 5.44 |
| | **3 month** | 6.27 | 6.16 | 6.05 |
| | **6 month** | 6.21 | 6.16 | 6.10 |
| | **12 month** | 7.02 | 6.87 | 6.76 |