

Controlling the Mineral Content of Sweet Whey Powder in an Industrial Setting

A Senior Project

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I. Abstract

The objective of this project was to reduce the final ash content of sweet whey powder. The data for this project was collected over a two-week period, from July 13th to July 27th 2010 in a dairy plant in the Mid-West of the United States. The data is composed of particle size test, ash test, and pH test. The conclusions of this study are that multiple pre-heat treatment tank changes contribute to mineral content by requiring more buffering than usual. The low pH of this pre-heat liquid forces a need for more buffering. Particle segregation may play a role in increasing the final mineral content, however more analysis needs to be done to determine the extent of this contribution. Finally, the increase in ash content may be due to seasonal variations in the cows environment and feed composition.

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V. Introduction

Cheese has come a long way from its humble beginnings during the agricultural revolution to the modern era of dairy product processing. Cheese is the major product that comes from the milk produced in California. The utilization of milk for class 4b products (Cheese, other than cottage cheese) was 39.4% of California's total production for the months of January through June 2010, according to the California Department of Food and Agriculture's mid-year review. All the cheese produced also has an equally large stream of whey that comes along with it. Much of this liquid whey is fractionated, concentrated and dried in some manner and sold as an ingredient to make products such as bread, candy, and other processed foods. If the liquid sweet whey is out of the targeted range of the many parameters (see Appendix 1) that are set by the Code of Federal Regulations (CFR) 21CFR184.1979, it is sold as an ingredient for animal feed at a significant economic loss. One of these variables, mineral content (commonly referred to ash) is the focus of this study.

VI. Literature Review

A. The Cheese Make Process and a Brief History of Whey Processing

Cheese was discovered between 3,000 and 8,000 years ago, most likely in the Fertile Crescent. This region between the Tigris and Euphrates rivers stretches from southern Turkey to the Mediterranean coast. It is where the agricultural revolution, or the domestication of plants and animals, took place (Fox and McSweeney, 2004). The discrepancy in the time around which cheese was actually discovered maybe due to these records being graphic, and not written. Also it would have taken time for this discovery to gain enough importance and popularity in people's lives that they considered it important enough to record. The story goes that a traveler placed some milk in a satchel made from a domesticated animal's stomach before a long trek. When the traveler stopped to have some of the milk stored in the satchel, it had curdled and become cheese (Smithers et al., 2008). It took centuries of learning about cheese making until the process could become industrialized. The first cheese plant in the United States was built in Rome, New York in 1891 (Farkye, 2009). Since then the dairy industry has adopted many specialized pieces of equipment to improve the safety, speed and control with which cheese is produced. It is important to note however that those basic principles that were discovered so long ago are still at the foundation of today's cheese making. Cheese production involves adding starter cultures (starters) to raw or pasteurized milk, either whole or reduced fat milk, that has been heated to their prime growing temperature, usually between 89°F and 100°F. Shortly after the starters are added, calcium chloride is also added. At this point it is also possible to add annatto

to give the cheese a yellow color, such as that seen in Cheddar. Once the starters have begun to grow, rennet is added to coagulate the milk. Rennet is a specialized enzyme that cleaves the milk protein casein, specifically the κ (kappa)-casein, at its Phe105-Met106 bond to form two peptides, para- κ -casein (bonds 1-105) and (glyco)marcopeptide (bonds 106-169). The cleaving of the kappa casein destabilizes the caseins structure, and exposes the highly calcium sensitive alpha-s1 (α_{s1}), alpha-s2 (α_{s2}) and beta (β) caseins, to the calcium. These proteins react by forming a gel. The extra calcium provided by the calcium chloride that was added earlier in the process further strengthens this gel and speeds up the coagulation of the milk. The gel is cut with curd knives within about 30 minutes of the rennet being added, and then the process of syneresis begins. Syneresis is the process by which whey is expelled from the cheese curd. After the freshly cut curd is allowed to rest for about 5 minutes, cooking the curd begins and continues for roughly an hour, depending on the type of cheese. Then the whey is drained from the curds, and both are moved on to further processing in their respective areas.

Whey shares its discovery with cheese, however it experienced an entirely different history. Much of the whey produced in the earliest times of cheese production was dumped into rivers, sewers and even the ocean (Smithers et al., 2008). Basically the cheapest method of disposal was used and as cheese making grew to utilize more of the milk supply, the issue of whey disposal also grew. According to Smithers et al. (2008), it has only been in the last 60 years or so that whey has gone from “[the] gutter-to-gold”. Before the 1950’s, many authors wrote about whey being used for medical purposes, and as a ‘cure’ for ailments (Smithers et al., 2008; Zollikofer, 1974). Many

factors including, technological advances, increased scientific knowledge, and tighter environmental regulations caused the major shift in whey utilization that now represents a normal operation in the dairy industry today.

B. Basic whey processing

There are two basic types of whey, acid whey and sweet whey. Acid whey is typically made by direct acidification of milk to produce cheese. Sweet whey is whey produced using the enzyme rennet. There are key differences in the composition of sweet whey and acid whey; those are summarized in Table 1 (Jimenez-Flores, 2010).

Table 1. Acid whey vs. Sweet whey.

Product	% Lactose	% Protein	% Salt Components	Fat	Water	pH
Dry Acid Whey	65.5	12.3	11.0	1.0	4.3	~4.6
Dry Sweet Whey	73.5	12.9	8.0	1.1	4.5	>6.1

From the cheese making room, the whey is usually pumped into storage tanks or silos; these vessels act as balance tanks to keep the processing equipment further down the line operating as much as possible. The first step the whey usually goes through is clarification, which is separating out any cheese curds and fines that might have been carried over during pumping from the cheese room. Then, the whey is passed through a separator to remove fat (as whey cream) in the whey. The defatted whey is pumped to a pH adjustment tank where an acid and a base are added to buffer the solution. This pH adjustment is done to help keep as many minerals in solution as possible, preventing fouling of the evaporator, which reduces efficiency and cuts down on run times. As soon as the whey has its pH adjusted, it is sent to another holding tank that again keeps the machinery fed with a flow of product at all times.

The next step is evaporation, or the partial removal of water; this is usually achieved by heating the milk indirectly with steam and under vacuum (Gekas and Antelli, 2002). Evaporation is done under vacuum to decrease the energy consumption of the process and also to reduce any heat damage that the product may endure. The steam used to heat the first pass of the product, or effect, is used to create a vacuum in the subsequent effects and thereby reduce the temperature required to boil the product. This method of creating vacuum is referred to as Thermal Vapor Recompression or TVR. Another method of creating a vacuum in an evaporator is using the thermal principal and an electrically driven fan to recompress the vapor and achieve a higher level of vacuum, and a lower boiling point of the product (Gekas and Antelli, 2002). This method of evaporation is referred to as Mechanical Vapor Recompression or MVR. The evaporator I was working with was a falling film seven-effect thermal vapor recompression (TVR) machine. In a falling film machine, the product is pumped to the top and falls down the tubes where evaporation occurs.

The separated silo feeds the evaporator. During this step the whey is heat treated to kill most microorganisms and concentrated. Once the whey is evaporated to the total solids target, it is placed in a crystallization silo where it is cooled and agitated gently. This crystallization targets lactose, and is done to reduce Millard browning when the whey is run through the dryer. While the crystallizer is filling, calcium hydroxide or lime is added to adjust the final pH. After 12 to 16 hours of crystallization, the condensed whey is fed into the dryer where more water is removed and it comes out at as a powder. The dryer used to process the whey I was working with was a nozzle atomizer spray dryer with a filter-mat. An atomizer delivers a fine mist of product to the

drying chamber, where the water removal occurs. This is just one type of dryer and other dryers used in dairy processing are: Drum Dryers, usually used for more viscous products, spray driers with rotary atomizers, and fluid beds (Refstrup, 2002). Fluid beds use a 'bed' of air to finish out the drying process and convey powder, while the filter mat dryer type uses a belt to convey the powder. The dried whey is typically conveyed to a powder holding silo and then is placed in bags and shipped to customers.

C. Overview of Milk Minerals

Minerals represent a very small portion of what is known as milk on a wet basis, .07% on average (Fox, 2009). The top five most abundant minerals in milk are: potassium, calcium, chloride, phosphorus, and sodium (Cashman, 2006). These minerals are only a few of the more than 20 essential minerals milk has to offer, and many of these become part of the whey stream. Each of these minerals plays an important role in how milk functions as a product during processing and when it's digested by humans and other animals. Potassium is the most abundant mineral in milk and whey; this was also true for the sweet whey I worked with. Potassium is a very important mineral in humans that has been shown to reduce blood pressure. It may also slow and possibly prevent renal disease, and lowers urinary calcium excretion. Certain types of potassium may also prevent diabetes (He et al., 2008). Finally, it may also decrease the likelihood of experiencing a cardiac arrhythmia (He et al., 2008). Calcium is also a very important mineral that dairy foods have a great deal of. Calcium intake may be complemented with potassium intake to increase its retention in your body (He et al., 2008). Dairy products, being high in both potassium and calcium, are ideal sources of calcium that is easily absorbed into the body. Thanks to the wide and ever increasing number of dairy

products available, it is easier for people to get their recommended daily intake of calcium. Chloride is an essential anion and it also plays an instrumental role in keeping fluid levels in our body's where they should be (Cashman, 2002). Chloride also contributes to the control of the electrolyte balance in our bodies (Cashman, 2002). Finally, phosphorus can be obtained through dairy products as well. In fact, milk and dairy products can contribute as much as 30 to 45% of the total phosphorus intake of people in western countries (Cashman, 2002). Phosphorus plays a pivotal role in metabolism, and, according Cashman (2002) to it also is an "essential component of many biological molecules, including lipids, proteins, carbohydrates, and nucleic acids."

D. Minerals in Whey and Their Significance

The minerals in whey are only a small part of the liquid whey and are overshadowed by the whey's larger constituents of: lactose, casein and whey proteins, and water. The principal minerals in whey are the same as in milk. They are as follows: potassium, calcium, chloride, phosphorus, and sodium (Cashman, 2006). These minerals serve many of the same purposes summarized in the milk minerals section and they end up in a wide variety of foods and beverages. Sweet whey has a number of functional properties, such as browning, bulking, foaming, and water binding (Banavara et al., 2003). These properties are all affected by the mineral content of the powder. Knowing the mineral contents of whey is important in their use as food ingredients and in understanding their contribution to the nutritional quality of foods (Wong, 1978). This is still true today and it has become even easier to know the approximate mineral content of many products thanks to advances in technology, such a near infrared spectrometers

and inline product analyzers that give real-time results of what is happening with a product.

VII. Objective

The main objective of this project was to reduce the final ash content of sweet whey through process adjustments in an industrial setting. This is a large undertaking, and the first one of its kind that I've ever done. In order to finish this project on time with the tight timetable I was dealing with, my team and I defined a few key areas to look at. The key areas we decided to focus on were: the addition of wash water to the whey stream, pH adjustment prior to evaporation, and the segregation of particles in the storage silos.

A. Wash Water addition to the whey stream

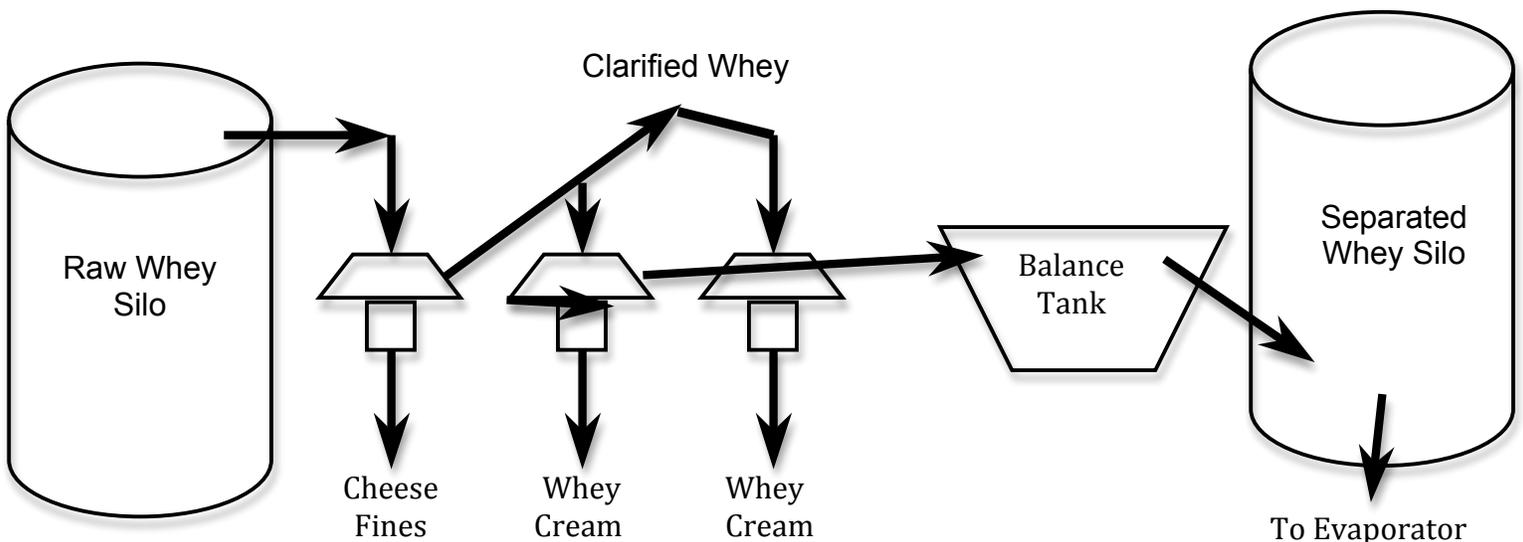
Wash water is simply the water added to the cheese to heat it up and prepare it for the pasta filata (stretching) process. Pasta filata means plastic curd, and is named such after a particular part of the process (McNaughton, 2002). The pasta filata process involves pouring warm water over the cheese curd at the right time and then kneading the cheese, much like you would knead bread. This is done to align the proteins in the cheese, such as mozzarella, to give it the characteristic stretching ability. This addition of water to the cheese curd washes away additional whey from the curd. When this water and whey mixture is added to the pure whey stream it may compound the amount of minerals in the liquid whey. This water solution is typically very low in pH, and means more adjustment to the pH of the liquid whey. This brings us to the next variable, pH adjustment prior to evaporation.

B. pH adjustment prior to evaporation

The pH scale is based on the hydrogen ion concentration of the item being tested. (Tro, 2006) The whey is drawn from the vats at a pH around 5.2-5.8, depending on the

product. The whey is then comingled in a silo and then it starts its journey. (See Fig 1.) To allow the highest possible volume of product through the system, the pH of the liquid whey is treated with an acid and a base to reduce fowling. Fowling is the buildup a film on the evaporation surface that leads to a loss of efficiency. If the pH wasn't adjusted to >5.9 the minerals in the whey would precipitate out and reduce the efficiency of the system. This is a critical point where there may be potential to reduce the buffering solution added to the liquid whey stream, therefore reducing the mineral content.

Figure 1. Liquid whey flow diagram



C. Particle segregation in the storage silos

Generally after the condensed sweet whey is dried, that sweet whey powder is stored in silos until it can be packaged. In the plant I was at, they were getting powder with high ash content when the level in these silos was high (usually $>60\%$ full). This condition usually occurred when they were approaching the end of a weeks run. Due to the

segregation of the bins having the potential to affect the final ash content of the product, I wanted to find out exactly how much some different particle sizes contribute to the ash content of the dried sweet whey.

VIII. Materials and Methods

A. Laboratory Procedures

The first lab procedure I did was an ash test. The ash content of a product refers to the inorganic residue remaining after either ignition or complete combustion of the organic matter in a foodstuff. The elements in ash are converted from their original forms to oxides or carbonates, and the organic salts are lost to oxidation during the intense heat treatment undergone in the procedure (Fox, 2009). I processed samples that were collected at the halfway point of drying a crystallizer tank, so as to catch a clean representative sample. There are a few tools needed to perform the ash test, this list started with an Analytical Balance capable of weighing a sample to the nearest of 0.0001 gram. Porcelain Crucibles were needed to hold the samples during the high heat treatments endured during the test and a stainless steel spatula. Also required was an Atmospheric oven heated to 100°C for pre-treating the crucibles and an electric thermostatically controlled muffle furnace capable of reaching and sustaining temperatures of 540°F to 550°F for many hours. To complete the pre-ashing procedure, I used a Bunsen burner, crucible tongs, a watch glass and a desiccator with desiccant. To perform the ash test on powder I first made sure the crucibles were clean, dry, and had an identification number on the bottom of them. To ensure they were dry, I stored them in an atmospheric oven at 100°C before use; furthermore I heated the crucibles over a Bunsen burner for 2 minutes to help ensure any residual material that may have been present was removed. After letting the crucibles cool for 10 minutes in the desiccator, I weighed up the vessel empty and recorded weight 1 to the 0.0001 gram. Weight 2 was recorded after I added exactly, or as close as the scale would allow, 2

grams, accurate to the 0.0001 gram. At this point I turned the muffle furnace on so it can pre-heat and I started carbonizing my samples. Carbonizing the samples was done by holding the crucible over the Bunsen burner until the sample was black, if any white or grey appeared I would have to throw that sample out. While the samples were still warm from carbonization, they were placed in a 550°F muffle furnace to ash for 2-3 hours, or until the sample was white-grey. After the time had elapsed I placed the samples in the dessicator for 15 minutes to cool, then I recorded weight 3 and discarded the sample. The ash content on the as is basis was calculated by the following equation: $((W3 - W1)/(W2 - W1)) \times 100$.

The ash testing procedure for fluid samples required all of the same materials as the powder test, except the spatula was exchanged for a small pipette. To perform the ash test on fluid samples I first made sure the crucibles were clean, dry, and had an identification number on the bottom of them. To ensure they were dry, I stored them in an atmospheric oven at 100°C before use; furthermore I heated the crucibles over a Bunsen burner for 2 minutes to help ensure any residual material that may have been present was removed. After letting the crucibles cool for 10 minutes in the desiccator, I weighed up the empty vessel and recorded weight 1 to the 0.0001 gram. Weight 2 was recorded after I added between 3 and 5 grams of sample, accurate to the 0.0001 gram. The samples were placed in the atmospheric oven for 1 hour at 100°C to evaporate some of the water off and reduce boiling over. After the hour elapsed I turned the muffle furnace on so it can pre-heat and I started carbonizing my samples. Carbonizing the samples was done by holding the crucible over the Bunsen burner until the sample was black, if any white or grey appeared I would have to throw that sample out. While the

samples were still warm from carbonization, they were placed in a 550°F muffle furnace to ash for 3-4 hours, or until the sample was white-grey. After the time had elapsed I placed the samples in the dessicator for 15 minutes to cool, then I recorded weight 3 and discarded the sample. The ash content on the as is basis was calculated by the following equation: $((W3-W1)/(W2-W1)) \times 100$. The dry basis calculation is: $100/(100-\% \text{ Moisture}) \times \text{as is ash}$. In addition to testing powdered samples, I also tested the following: raw liquid whey, separated liquid whey, condensed liquid whey, and the wash water (pre-heat liquid).

For the fluid samples I realized it was necessary to obtain the moisture to be able to calculate the dry basis ash percentage. For this analysis I used a Lab Wave 2000 microwave analyzer. This analyzer started with a known weight of a sample, then it used microwaves to evaporate the water in sample. It then reweighed the sample and subtracted the known beginning weight from the end weight and gave the moisture content. The materials needed for moisture analysis were a microwave analyzer, two paper pads for the analyzer and a disposable pipette. For free flowing products I agitated them by shaking in a one-foot arc for 30 seconds, placed the appropriate amount of sample on one of the pads in the analyzer that was on the right channel for the sample I was testing. I then covered the sample with the other pad, closed the door and hit run. For the condensed samples required I was required to heat them in a 93°F water bath for 10 minutes and then blend the heated sample on high for 1 minute. 10 grams of blended sample was then diluted with 30 grams of warm water and agitated in a one-foot arc for 30 seconds until mixed. The testing procedure for condensed samples was the same as non-condensed, other than the machine channels. To attempt to

determine how much particle size contributed to ash content, I also separated samples by particle size using a rotap machine and a series of 6 screens. A Rotap machine holds the screens in a guide and pounds the top of the sifting screens, much like a hammer would, to separate particles out by particle size. I analyzed the ash content of each size of particle and report the data in table 2. For the particle analysis test, I used the following screen sizes: 30, 50, 60, 70, 100, and 200. Below the 200 screen there was a pan to catch particles smaller than any of the screens. The screen size refers to the identification number on the front of the screen. I would place 225 grams of powder on the 30-screen, which would be the top. I choose 225 grams because it would provide me with enough powder in the coarser screens that I could run the ash test. After ensuring I had clean dry sieve screens to work with, I would place the screens on the pan in the following order, starting from the bottom and working my way up: 200, 100, 70, 60, 50, 30. I would then place the lid on the screen column, swing down the mechanical arm and allow the machine to run for 10 minutes. After the ten minutes had elapsed, I would weigh the amount of powder retained in each of the screens and the pan. Once I had accurately recorded the weight, I would place each sample in to a whirl pack bag with the date of the sample, the sample source, the weight and the screen size the sample was collected from. Then when I had time, I would go back a run an ash test on 2 grams of each of the different screen sizes for each particle size.

IX. Results and Discussion

A. Results

There was no observed difference between the amount of wash water used and the final ash content of the sweet whey powder. The number of wash water tank changes in a day did make a difference though. Due to the typically low pH of the wash water, 4.92 from an average of 16 samples, it required more buffering to reach the pH target for evaporation. The average of the results of the particle size test is reported in table 2. Figure 2 illustrates the distribution of the whey powder in the screens and the pan. This was calculated by dividing the amount of powder in any given screen by the total sample tested.

Table 2. Particle Size Test Results.

Screen Size	Percentage of Powder in Screen
30	0.14%
40	0%
50	3.62%
60	4.31%
70	8.15%
100	24.00%
200	41.06%
Pan (>200)	18.73%

Figure 2. Graph of Particle Size.

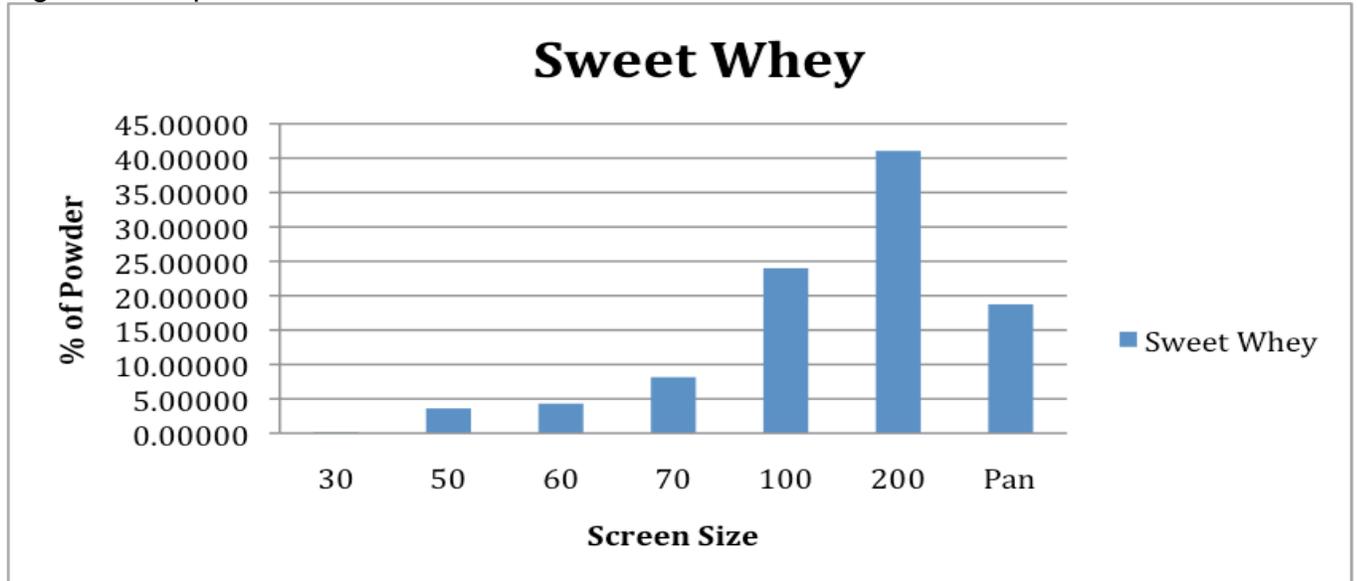


Table 3 shows the average ash content of all the powder I broke up by particle size.

This is the ash percentage for the as is basis. Screen Size 50 is an average of 7 samples and the rest are an average of 18 samples.

Table 3. Ash Content by Particle Size.

Screen Size	Ash %
50	7.08%
60	6.18%
70	5.51%
100	5.96%
200	9.48%
Pan (>200)	12.45%

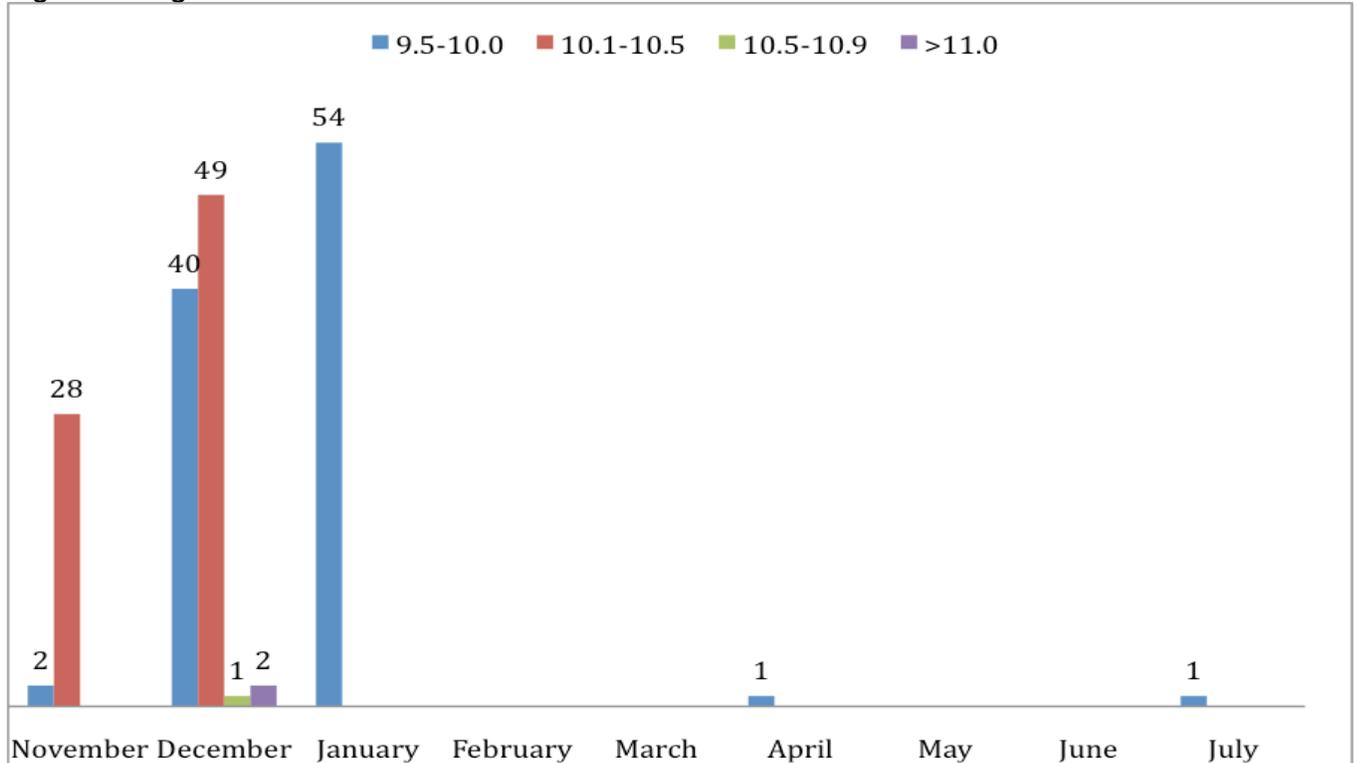
Table 4 shows the average of 16 samples per each category. The ash is reported in the dry basis form to allow it to be compared to the powder ash results.

Table 4. Average Ash for Fluid samples on the Dry Basis.

Product	Ash %
Unseparated Whey	8.27%
Separated Whey	7.98%
Pre-heat Liquid	9.62%
Crystallizer Tanks	8.79%

Figure 3 shows the number of pallets that were down graded from grade A sweet whey to animal feed during the fiscal year to date that I was working.

Figure 3. High Ash Levels YTD.



B. Discussion

The low pH of the wash water would contribute to the mineral content of the whey because a greater amount of buffering solution would have to be added to the whey stream to achieve the desired change in pH. The distribution of the particle sizes of the powder is interesting. It illustrates the large proportion of fine particles that are high in ash. If these fine particles were to build up in a silo that didn't get agitated enough these fine particles would definitely increase the final ash content of the product. Also the fluctuation in ash content seems to occur more frequently around the winter months. This data is ambiguous however because the drop off in high ash powder coincides with a reduction in the number of times the preheat tank was changed in a day.

X. Conclusion

The data in this study points to a need to keep the number of wash water tank changes in a day low. The data suggests that the low pH of the solution indirectly contributes to the mineral content of the end product. This indirect contribution is the increase in the buffering solutions needed to adjust the Ph prior to evaporation. The addition of an acid and a base at the pH adjustment stage of the process is an important control point. A pH monitoring system that is tied into supply pumps to more accurately adjust this pH could be a viable solution to help conserve ingredients, and avoid over adding minerals to the product. Finally, the final product mineral content may vary if the bin is not properly agitated. The current agitation system works well in low bin levels, however when the bin levels are high the agitation system does not work as well. More research needs to be done in this area to draw better conclusions and to formulate a plan of action. Finally, the increase in ash content may be due to seasonal variations in the cows environment and feed composition, but a more extensive study of the incoming milk must be done to determine if this hypothesis is valid.

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Appendix 1.

Code of Federal Regulations Title 21 Section 184.1979- Definition of Sweet Whey.

“Whey obtained from a procedure in which there is insignificant conversion of lactose to lactic acid is known as sweet whey. Sweet whey has a maximum titratable acidity of not more than 0.16 percent, calculated as lactic acid, and an alkalinity of ash of not more than 225 milliliters of 0.1N hydrochloric acid per 100 grams. The acidity of whey, sweet or acid, may be adjusted by the addition of safe and suitable pH-adjusting ingredients.”

In addition to this, the whey must have the following: 1. A protein content between 10% and 15%, 2. A fat content between 0.2% and 2.0%, 3. An ash content between 7% and 14%, 4. A lactose content between 61% and 75%, and 5. A moisture content between 1% and 8%. Please refer to 21CFR184.1979 for a complete definition.