Federal-State Marketing Improvement Program
Final Performance Report
For the Period of [September 30, 2013 – September 29, 2016]

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Recipient Name: **University of Minnesota**
Project Title: **Adding Value to Acid Whey Residues from Dairy Processing: Technical Feasibility and Market Opportunity Analysis Grant**
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An Outline of the Issue or Problem: Only one quarter of U.S. milk production is consumed as fluid milk. The rest is processed into dairy products such as butter, yogurt, cheese or milk powders. Over the last three decades, dairy farmers have used dairy check-off program to invest in fundamental and applied dairy product research that added value to residues of dairy processing, such as liquid sweet whey, a waste stream of cheese production. A recent rise in consumer demand for Greek yogurt has amplified the long-standing problem of *acid (i.e. "sour")* whey, which is a byproduct of removing water from yogurt to achieve Greek-style thickness. While value was added to sweet whey by concentrating whey proteins, acid whey is not as rich in whey proteins because they are mostly retained in yogurt. Furthermore, due to its composition, acid whey is difficult to dry and handle. As such, this bystream is currently considered as waste, adding little value to dairy producers, and disposal problems to dairy processors. This project examined if additional marketing opportunities can be created for acid whey by converting it to soluble dietary fiber.

Goals and Objectives: The first objective was to conduct a full technical analysis with the goal of identifying yield-maximizing process for converting acid whey to dietary fiber. A second objective, conditional on successful completion on the first objective, was to identify market size and necessary preconditions that indigestible carbohydrates produced from acid whey would have to satisfy to be competitive on the dietary fiber market.

Acid whey was produced by manufacturing yogurt, followed by centrifugation to remove whey. Proteins were removed from the acid whey stream as their presence would lead to side reactions that would lower the yield of fiber. The proteins were heat-precipitated and removed from the whey by centrifugation. The deproteinized whey was concentrated using either a Genevac vacuum centrifuge, a Buchi Rotovap, or a glass vacuum-pan condensor. We then crystallized the lactose by holding the concentrated material in a refrigerator for several days, and centrifuged out the lactose crystals. Washing steps were also investigated to remove acid and minerals. We dried the concentrated whey by drum drying, or drying in aluminum pans in a gravimetric oven. The results of this clean up process are described below.

Additional activities to evaluate factors limiting the polymerization of lactose to dietary fiber were the development of a benchtop method to polymerize, and evaluating the effect of minerals and moisture on polymerization. We used sweet-whey permeate as the material to evaluate these effects (contains minerals like acid whey, but not as much lactic acid). The benchtop method involved using a MARS6 microwave digestor. We developed a protocol to polymerize lactose, glucose and citric acid blends, and then investigated how calcium and moisture affected the polymer yield.

The above described process did not result in marketable dietary fiber product so instead of market analysis we performed additional activities to evaluate to evaluate factors limiting the polymerization of lactose to dietary fiber. To that end we developed a benchtop method to polymerize, and evaluate the effect of minerals and moisture on polymerization. We used sweet-whey permeate as the material to evaluate these effects (contains minerals like acid whey, but not as much lactic acid). The benchtop method involved using a MARS6 microwave digestor. We developed a protocol to polymerize lactose, glucose and citric acid blends, and then investigated how calcium and moisture affected the polymer yield.

Contribution of Project Partners:

Agropur, Inc., LaCrosse, WI supplied acid whey powder, and whey permeate.

Idaho Milk Products, Jerome, ID supplied IdaPro permeate powder that was used in an extrusion trial

CEM Corporation provided technical assistance for troubleshooting the MARS6 Microwave digestion system.

Results, Conclusions, and Lessons Learned:

Technical Summary:

Dried acid whey was extruded on a Buhler twin-screw extruder as a pre-trial experiment to understand if extensive processing of acid whey will be required in order to polymerize the lactose in the material to dietary fiber. The result of this trial is that it was determined (by chromatography) that no significant polymerization occurred.

Additionally, the dried acid whey contained too much acid and protein, which resulted in a black product, and corroded the interior of the extruder and caused approximately $40,000 in damage. As result, clean-up procedures for acid whey were investigated, and a small scale lab process to evaluate the effect of moisture, protein and minerals on polymerization was developed.

Heat treatment and washing both reduced the amount of protein and reduced the amount of brown color evident. Heat treatment also improved the run-ability on a drum dryer. Heat treatment greatly reduced the amount of nitrogen in the samples. It did not seem to greatly affect the minerals though.

Figure 1. Appearance of pilot-scale acid whey after clean-up and drying.

Treatment		Processing Description	Qualitative Description	Appearance (after grinding)
Heat Treatment	Wash			
Heat treated (Replicate 2)	None	Very sticky, stuck to drum dryer blade, caramel-consistency	Dark brown, large glassy pieces	
	Once	Slightly sticky, flaked off of drum dryer into small clumps	Light tan powder, some clumps	
	Twice	Dried nicely, flaked off the drum dryer into a powder	White powder, few clumps	
No heat treatment (Replicate 1)	None	Very sticky, stuck to drum dryer blade, caused oil buildup on drums, caramel like consistency,	Brown, large pieces, pliable	
	Once	Somewhat sticky, built up on drum dryer blade, caused some oil buildup, crumbly consistency	Dark tan, large clumps with some powder	
	Twice	Slightly sticky, flaked off of drum dryer into small clumps, slight oil buildup	Light tan powder with some clumps	

We also developed a method to polymerize lactose/glucose/citric acid in a MARS6 microwave assisted reaction system (CEM Corporation). Issues were encountered with achieving the polymerization in the microwave. During the extrusion process, moisture was not required to polymerize the lactose and, in fact, we believe increased moisture could lead to hydrolysis instead of polymerization. However, microwave heating is the result of the frictional heat generated by the vibration of polar molecules (such as water). Not having a polar solvent in the reaction vessels led to inconsistent heating between tubes and the inability to achieve our target temperature in most cases. We investigated adding a small percentage of water (<1% wt/wt) and were able to achieve polymerization and consistent heating with pure lactose. An example chromatogram is shown in Figure 2. Another interesting observation is that the temperature of the reaction necessary to achieve polymerization was lower (140C in the microwave instead of 185C during extrusion). This is most likely due to being in sealed tubes that generate pressure during heating, whereas we were running the extruder without a die-plate and not achieving pressures greater than atmospheric pressure.

After developing the experimental protocols for bench-top polymerization, we investigated the effect of added moisture (Figure 3, Table 3), and of minerals (Table 4), on the ability to polymerize lactose. Moisture addition did not have a detrimental effect on fiber yield until quite a bit of moisture was added. This is interesting in that it could be that lactose containing coproducts could be polymerized in an extruder without having to be dried first. This has not been investigated, however. Calcium did have a detrimental effect at all levels added. We investigated the effect of increasing the citric acid catalyst when calcium was present and determined that higher concentrations improved the fiber yield (Table 5).

We evaluated polymerization of whey permeate instead of acid whey. It is mainly lactose and minerals. Unlike acid whey, it does not have as much nitrogen, or the high levels of lactic acid. We used this as a surrogate to the acid whey, assuming we can at some point clean up the acid whey itself to this level to run on our extruder and not cause damage. Using the benchtop method, we found that adding higher concentrations of citric acid catalyst did increase fiber yield, however the yield was low (<4%) when compared to pure lactose (>35%). Extruding permeate with increased citric acid to counteract the high mineral content resulted in some polymerization. Previous runs with permeate did not result in polymerization when when 4% citric acid was used. Using 10% citric acid with permeate resulted in a fiber yield of approximately 4%. Much lower than what we achieve when using pure lactose. This low yield directly correlated with what we achieved by the benchtop method, so further work to improve yields on permeate and acid whey can proceed on the bench prior to expensive extrusion trials.

- What unanticipated challenges were encountered?
  It is very difficult to clean the lactic acid out of acid whey, and the cleaned up samples still had quite a bit left. We were unable to run the cleaned-up acid whey on the extruder again because we were not confident that we could run it without doing further damage to our equipment.
- What unexpected positive results occurred? We developed a benchtop method to try to understand what things are inhibiting polymerization. We still do not have enough answers generated to move forward with acid whey as the primary lactose source.
- "If I had it to do over, I would have…" I would have written the project for permeate, or mother liquor from lactose processing specifically, and tried to clean that product up to improve fiber yield. Minerals and nitrogen are factors that I think are inhibiting, and it is worth looking at cost effect options to allow for polymerization. Acid whey will require too much energy to clean up to make it a viable lactose source for this process.
- What advice do you have for others taking on a similar project work? Don't put acid whey in an extruder without first removing the lactic acid. Also, develop benchtop methods prior to running on expensive processing equipment.

Evaluation:

We set out to solve the problem of acid whey by proposing to convert lactose in acid whey to indigestible dietary fiber. This can be measured by the degree of polymerization of lactose in treated samples. We evaluated the degree of lactose polymerization in a variety of scenarios described above. Our results indicate that acid whey will require too much energy to clean up to make it a viable lactose source for processing into dietary fiber.

Current or Future Benefits/Recommendations for Future Research:

We recommend investigating whey permeate, or 'mother liquor' from lactose processing as a feed for creating dietary fiber from lactose. Acid whey will likely not be solved using this method.

Project Beneficiaries: Given the research-heavy nature of the project, and extensive technical troubleshooting we performed, the primary beneficiaries were students employed on the project:

Two graduate students graduated with Master's degrees while working on this project

An undergraduate completed her undergraduate research thesis developing the lab method of clean-up and drying method for acid whey, and presented her results at the undergraduate research symposium. She is now enrolled in a graduate program at Kansas State.

Indirect beneficiaries are other dairy researchers who can build upon insights developed in this grant to pursue further research in this area. At present state, we do not forsee direct industry beneficiaries.

Additional Information:

Posters:

Kuechel. A. and T.C. Schoenfuss. 2017. The effect of calcium phosphate on lactose polymerization to soluble fiber. IFT National Convention

Kuechel, A. and T.C. Schoenfuss 2016. Development of a benchtop method to polymerize lactose to soluble fiber. American Dairy Science Association National Convention.

Tebben, L. 2016. Clean up of acid whey: Effect of heating and washing on by-product of Greek yogurt production. UMTC Undergraduate Research Presentation
https://conservancy.umn.edu/handle/11299/181113