Study on the Effectiveness of Microbubbles in the Cleaning of Milk Residue using a Nikuni Pump

Manuel Alejandro Padilla Chevez

Escuela Agrícola Panamericana, Zamorano
Honduras
November 2020

ZAMORANO
FOOD SCIENCE AND TECHNOLOGY MAJOR

Study on the Effectiveness of Microbubbles in the Cleaning of Milk Residue using a Nikuni Pump

Special graduation project presented as partial requirement to obtain a Bachelor of Science degree in Food Science and Technology

Presented by:

Manuel Alejandro Padilla Chevez

Zamorano, Honduras
November 2020

Study on the Effectiveness of Microbubbles in the Cleaning of Milk Residue using a Nikuni Pump

Manuel Alejandro Padilla Chevez

Abstract. Dairy products play an important role in the human diet. Due to this, cow milk must undergo high-temperature treatments to provide a safer product. This high-temperature treatment also known as pasteurization promotes the accumulation of proteins and minerals on heat exchangers that are known as fouling. In the dairy industry rapid fouling in heating equipment is responsible for more than 50% of the production cost. Microbubbles can become a green cleaning agent. In this study, the effect of temperature and rotation using Microbubble- Infused Water using a Nikuni Pump and pure water in cow milk fouling behavior were studied by a Spinning Disk Apparatus (SDA). The SDA is capable of controlling two principal factors, temperature and rotation. The experiment consisted on two phases, the first one was to evaluate the effect of temperature. The effect of temperature was evaluated using three different temperatures at the same rotation speed: 25, 40 and 55 °C @ 80 RPM; meanwhile, the effect of rotation was evaluated using three rotation speed at the same temperature: 50, 65, and 80 RPM at 25 °C. In the first phase fouling loss and fouling loss percentage increased as the temperature increased in both Microbubble-Infused Water and pure water. In the second phase, rotation speed had no significant difference in the fouling loss and fouling loss percentage in both Microbubble-Infused Water and pure water. In the second phase, the rotation speed had no significant difference in the fouling loss.

Key words: Dairy, fouling, green technology, microbubble-infused water.

Resumen. Los productos lácteos juegan un papel importante en la dieta humana. La leche de vaca debe someterse a tratamientos de alta temperatura para proporcionar un producto más seguro. Este tratamiento de alta temperatura también conocido como pasteurización promueve la acumulación de proteínas y minerales en los intercambiadores de calor. Los residuos de leche en los intercambiadores de calor son responsables de más del 50% del costo de producción. Las microburbujas pueden convertirse en un agente de limpieza ecológico que pueden ayudar a reducir este costo. En este estudio, se estudió el efecto de la temperatura y la rotación usando agua infundida con microburbujas usando una bomba Nikuni y agua pura en el comportamiento del residuo de la leche de vaca mediante un aparato de disco giratorio (SDA). El experimento consistió en dos fases, la primera fue evaluar el efecto de la temperatura. El efecto de la temperatura se evaluó utilizando tres temperaturas diferentes a la misma velocidad de rotación: 25, 40 y 55 °C @ 80 RPM; mientras tanto, el efecto de la rotación se evaluó utilizando tres velocidades de rotación a la misma temperatura: 50, 65 y 80 RPM @ 25 °C. La pérdida de residuo de leche y el porcentaje de pérdida de residuo de leche aumentaron a medida que aumentaba la temperatura en ambos tratamientos. La velocidad de rotación no tuvo una diferencia significativa en la pérdida de residuo de leche y el porcentaje de pérdida de residuo de leche en ambos tratamientos.

Palabras clave: Agua con infusión de Microburbujas, lácteos, residuo de leche, tecnología verde

TABLE OF CONTENTS

LIST OF TABLES AND FIGURES

Tables Page

Figures Page

1. INTRODUCTION

Nowadays, with the growth of industrial development, industrial water usage and wastewater generation significantly increased. At the global level, the water usage ratios are 69% for agriculture, 12% for municipal and 19% for industry (AQUASTAT - FAO's Global Information System on Water and Agriculture 2020). In the food industry, cleaning is a critical process, since manufacturers must reduce bacterial load and eliminate pathogens in food processing line to meet the strict hygiene standards.

Cleaning-in-place (CIP) is widely used in food processing industry to remove product residuals, deposits and microbes that remain in processing line (Kylee R. Goode et al. 2013). CIP is an important process to maintain production efficiency and ensure product quality and safety. During CIP, water and chemical solutions are circulated in the processing equipment and pipelines, its operation is generally fully automated for large-scale production processes. Different type of CIP processes are used in food and pharmaceutical industries such as 3, 5 or 7-step CIP processes product recovery which includes the following stages are, i) Pre-rinse, ii) Detergent recirculation, iii) Intermediate rinse, iv) Second detergent recirculation, v) Intermediate rinse, vi) Disinfection, vii) Final rinse (Dhage and Dhage 2016). Each step is operated at specific conditions, including time, flow rate, temperature and chemical concentration, to ensure a repeatable level of cleanness is being achieved.

Milk and its products play important roles in human diet. Over the last three decades, world`s milk production has increased by more than 58%, from 522 million tons in 1987 to 828 million tons in 2017 (FAOSTAT 2020). At the global level, cow milk has the largest production is most widely consumed by human (Faye and Konuspayeva 2012).For a safer milk product with longer shelf life, thermal treatment (pasteurization) on milk is required, which, however, promotes accumulation of proteins and minerals on heat exchangers, which is known as fouling. Fouling is a severe problem in food processing industry, for example, in dairy industry, the rapid fouling in heating equipment is responsible for 80% of the production cost (Asset *et al.* 2005).

Milk is a complex biological fluid and contains several substances (Bansal and Chen 2006). Milk fouling is classified into two categories depending on the temperature at which milk is processed. Type A (protein) fouling usually takes place at a temperature between 75 and 110 ºC, Type B (mineral) fouling occurs at temperatures above 110 ºC (Burton 1968).

Although cleaning is required for milk processing, current CIP operation with intensive uses of alkaline and acid can negatively impact production costs and environmental footprint (Zouaghi *et al.* 2018), hence finding alternative approaches is necessary. Microbubbles with diameters less than 50 μm have been applied in many industrial processes due to their unique interfacial physicochemical properties (Ashutosh Agarwal et al. 2012): (i) large surface area, (ii) hydrophobic surface that can absorb organic molecules such as fat, (iii) extended residence time in liquid, and (iv) fast dissolution rate and generation of free radicals (Makoto Miyamoto et al. 2007). These properties make microbubbles an alternative, green cleaning agent. A CIP process incorporated el with microbubbles is expected to enhance its cleaning efficiency and reduce the associated environmental footprint. This study included the following objectives:

- Determine the percentage of elimination of fouling using microbubble-infused water (MIW) as cleaning agent using a Nikuni Pump for milk fouling on heat transfer surfaces at the pre-rinsing stage in comparison with pure water.

- Evaluate the effect of microbubble-infused water using a Nikuni Pump in the cleaning process in the milk fouling using different temperature maintaining the change in temperature (ΔT).

- Evaluate the effect of different rotation speed of the Spinning Disk Apparatus (SDA) on the cleaning process in the milk fouling.

2. MATERIALS AND METHODS

Research location

This study was conducted in the Food Process Sustainability Laboratory, Department of Food Science at Purdue University in West Lafayette IN, United States. Statistical Analysis was performed at Zamorano University, in Honduras, Central America.

Raw materials

Milk fouling deposits were used for cleaning tests in this study. Milk was prepared from whole milk powder purchased from Great American Spice Co. (Fort Wayne, IN, USA). Milk powder was mixed with water at a ratio of 3:7 (w/v) under magnetic agitation at room temperature until all powder was completely dissolved, resulting in milk sample of 20% solid content. In this case the ratio was decided due to the type A fouling which takes places at temperature between 75 °C and 110 °C, and their composition is 50-70% protein (H. Burton 1968; Visser and Jeurnink 1997).

Milk fouling deposits

Milk deposits were formed on stainless steel surfaces for subsequent cleaning. A stainless-steel disc with a diameter of 89 mm was placed on a hot plate (Thermo Scientific, Waltham, MA, USA) and heated to 100 ºC. Three millimeters of milk sample were pipetted at the center of the heated disc, which spread and formed a circular deposit of 62-mm diameter. After 10 min, the same procedure was repeated for two more times, resulting in the final deposit formed by 9 mL of milk sample in total. The fouled disc was then placed in a vacuum oven overnight at 50 ºC and −0.05 MPa for complete drying, and the mass of the dry deposit was measured (Zhang *et al.* 2020).

Spinning disc apparatus (SDA)

The cleaning tests were carried out using a Spinning Disc Apparatus (SDA). A detailed description of the apparatus is given in (Zhang *et al.* 2019). In brief, the SDA consists of a cylindrical can that is immersed in a reservoir of test solution, which in this study is either RO water or MIW. The cylindrical can rotates at varying speeds, which is driven by a stepper motor. A glycol/water recirculation system was used to regulate the temperature of the can. The temperature of the can is a variable of the cleaning tests, which is measured by T-type thermocouples connected to a datalogger. Zhang also indicated that the SDA is a reliable tool to simulate the actual flow conditions which arise in milk pasteurization.

Microbubble-infused water

A Nikuni microbubble pump (Nikuni America, Mt. Prospect, IL, USA) was used to generate the microbubbles in water. The pump is connected to a flowmeter to regulate the flow rate of MIW at 0.5 L/min.

The MIW was characterized by Chung *et al* (2019) using the Particle Counter PC3400 (Chemtrac, Norcross, GA, USA), the bubble size distribution (Figure 1) showed that the microbubbles generated were smaller than 20 µm. (Lee *et al.* 2015) reported that microbubbles smaller than 10 µm are more stable in liquid. Stabilizing microbubbles in a solution increase its cleaning efficacy, because microbubbles have higher tendency to attach to fouling deposits instead of coalescing to each other (Temesgen *et al.* 2017).

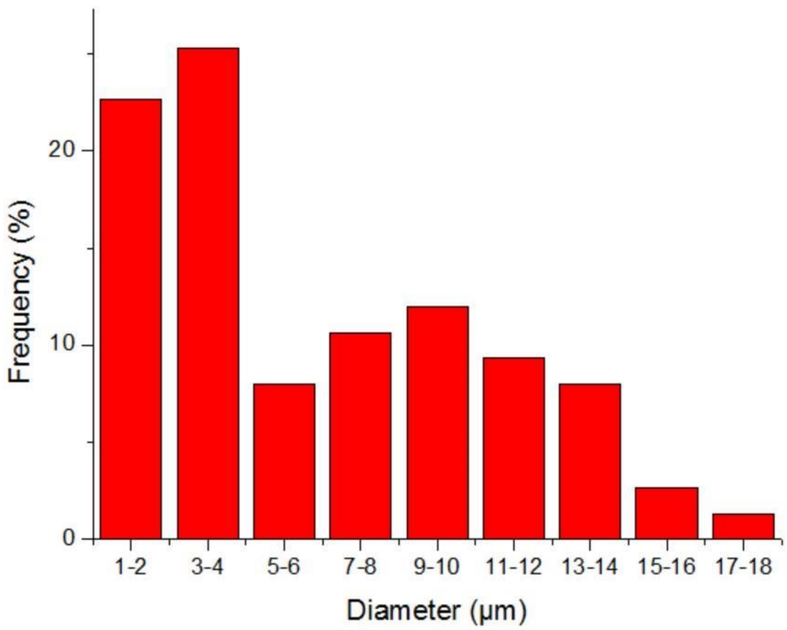


Figure 1 Size distribution of microbubble in water (Chung *et al.* 2019)

Cleaning test

The disc with dry deposit was soaked in water for 30 min to obtain a soft deposit to mimic the fouling obtained at the heat exchanger walls. Then, the disc was attached to the SDA for cleaning using either MIW or water as the control for 20 min. The cleaning was conducted at temperatures of 25, 40 and 55 ºC and the rotational speed of the SDA cylinder were 50, 65 and 80 rpm. After cleaning, the disc was detached from the SDA and placed in the vacuum oven overnight to dry the deposit residue at the same temperature and pressure. The mass of the dry deposit residue was then measured to determine the cleaning efficiency.

Experimental design and statistical analysis

The experimental design of this study followed Completely Randomized Design (CRD) with a factorial arrangement 2 × 3 The data obtained from all the tests was evaluated for significance by the analysis of variance (ANOVA) using the SAS® software version 9.6. When the effect of the variable was significant ($P < 0.05$), post-hoc Tukey test was applied at 95% confidence interval. The statistical analysis was done by ANOVA to determine the effect of the temperature (Table 1) and rotation speed (Table 2). In order to have a more precise and to reduce variation in the percentage analysis methods of transformations of data was used. In this case Arcsine was used to change the percentage in temperature percentage and rotation percentage. A residual analysis was done to all data in order to find any outliers.

Table 1. Factorial design of temperature at 80 RPM.

Treatments	**Temperature (°C)**
	25
Microbubble-infused water	40
	55
	35
	50
Pure Water	55

Rpm: revolutions per minute of the Spinning Disk Apparatus.

Table 2. Factorial design of rotation speed at 25°C.

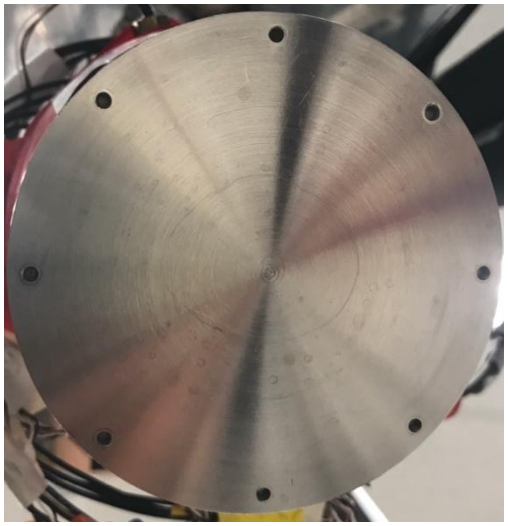
Treatments	**RPM**
	50
Microbubble-infused water	65
	80
	50
	65
Pure Water	80

Rpm: revolutions per minute of the Spinning Disk Apparatus

3. RESULTS AND DISCUSSION

The formation of fouling deposits has been widely investigated in the last decade. Cleaning of this deposits can present a huge cost in the dairy operations where whey proteins are present (Smithers 2015). Researches consider that unfolded β-lactoglobulin (β-lg) which is the main protein in whey is the principal cause of promoting the deposits of unwanted solids in the surfaces of heat exchangers (Yang *et al*. 2018).Figure 1 shows pictures of the SDA disk before (1a) and after forming the milk fouling (1b), in which the paste is completely attached to the disk. As is expected the deposits are attributed to the β-lg.

The fouling present on the disk is considered a Type A fouling since this type of fouling takes place between 75 and 110 °C as describe by Bansal and Cheng (2006), these deposits are white, soft and spongy as can be observe on Figure 2b. The white color of the fouling is due to whey protein. β -Lactoglobulin (β -Lg) and α-lactalbumin (α-La) are the two main proteins in whey protein. Nevertheless, the dominant protein in heat induced fouling is β-lg.



A **B**

Figure 2. Pictures of clean SDA disk (A) and disk with fouling deposits (B).

Effect of temperature on cleaning

The fouling loss on the disks and the percentage loss of fouling was evaluated in this part. Table 3 shows the probability of the factors temperature and treatment. The loss and loss percentage in the temperature percentage analysis had a significant difference ($P < 0.05$) in which 55 °C is different from 25 and 40 °C as seen in Table 4.

Table 3. Probability of the factors and interactions for treatment (type of water) and temperature.

Source	**PR > F**
Temperature	0.0005
Treatment	0.0710
Temperature × Treatment	0.0326

Pr. Probability less then 0.05 show a significant difference.

Table 4. Loss percentage means and standard deviation (SD) for different temperatures at 80 rpm.

Temperature	**Loss % ± SD**
25 °C	25.17± 3.86^{A}
40 °C	29.68 ± 10.27^{A}
55 °C	40.85 ± 2.86^{B}
CV (%)	18.98

A–B Means with different letters in the column are statistically different (P < 0.05).
CV(%):Coefficient of Variation.
Rpm: revolutions per minute of the Spinning Disk Apparatus.

Temperature itself had a significant difference but it is well known that increasing the temperature of the CIP can decrease the cleaning time of different kind of deposits (Goode *et al.* 2013). In this experiment it followed that trend in which at a higher temperature there was a greater fouling loss. Incrementing the temperature of the solution of microbubbles helps to increase the grease removal which means that raising the temperature of the cleaning solution has a positive impact on microbubble cleaning (Miyamoto *et al.* 2007). The cleaning temperature of 35 and 50 °C had a much greater cleaning capacity than 20 °C. high temperature could weaken the structure of membrane fouling layer, expand the membrane pores and facilitate foulants removal. (Zhang *et al.* 2017). Microbubble-Infused Water and Pure Water Treatments on this analysis do not show a significant difference (P < 0.05) as seen in Table 5.

Table 5. Loss percentage means and standard deviation (SD) for different treatments at different temperature at 80 rpm.

Treatment	**Loss % ± SD**
Microbubble-infused water	34.32 ± 8.08^{A}
Pure Water	29.49 ± 9.99^{A}
CV (%)	28.71%

A–B Means with different letters in each column are statistically different (P < 0.05).
CV(%): Coefficient of Variation.
* Temperature are 25,40 and 55 °C.
Rpm: Revolutions per minute of the Spinning Disk Apparatus.

Microbubble treatments that are combined with other methods for example effervescent cleaning reagents can significantly improve cleaning performance over conventional methods (Chesters *et al*. 2015). Microbubbles are effective for reversible fouling and are not effective for irreversible fouling by the natural organic matter adsorption. (Watabe *et al.* 2016). Also, protein concentration decreases when exposed with microbubble over long period of time which is one of the reasons why microbubble could control membrane fouling which can suggest more time in cleaning process with microbubble (Lee *et al.* 2015).

Effect of different rotation speeds on the cleaning process

The rotational speed used in the analysis where 80, 50 and 65 rpm. The part evaluated was the fouling loss on disk and the percentage loss of fouling due to different rotations and treatments. The probability of the factors and interactions for the type of water and rotation can be seen in Table 6.

Table 6. Probability of the factors and interactions for type of water and rotation.

Source	PR > F
Rotation	0.0019
Treatment	0.4342
Rotation × Treatment	0.2962

Pr. Probability less then 0.05 show a significant difference.

The treatments itself had no significant difference ($P < 0.05$) in the fouling loss as seen in Table 7. However, there is a significant difference ($P < 0.05$) between the cleaning at 65 rpm and the other rotational speeds (80 and 50 rpm) as seen in Table 8.

Table7. Loss percentage means and standard deviation (SD) for different rotations at 25 °C.

Rotation	Loss % ± SD
80 RPM	25.17 ± 3.86^{A}
50 RPM	22.08 ± 1.95^{A}
65 RPM	17.88 ± 1.83^{B}
CV (%)	11.5%
Pr	< 0.0005

$^{A-B}$ Means with different letters in the column are statistically different ($P < 0.05$).
CV(%):Coefficient of Variation.
Rpm: revolutions per minute of the Spinning Disk Apparatus.

Table 8. Loss percentage means and standard deviation (SD) for different treatment at different rotations at 25 °C.

Treatment	Loss % ± SD
Microbubble-infused water	22.22 ± 4.62^{A}
Pure Water	21.30 ± 3.46^{A}
CV (%)	18.55%

$^{A-B}$ Means with different letters in the column are statistically different ($P < 0.05$).
CV(%) = Coefficient of Variation.
* Rotations are 50, 65 and 80 rpm.

The 65 rpm had a less fouling loss in comparison to the 80 and 50 rpm. However, there was a high coefficient of variation in the 65rpm test which explains why there is a significant difference in the loss and loss % of fouling. Higher revolutions per minute improved the efficiency of removing casein micelles and whey protein layer (Zhang *et al.* 2017). Nevertheless, in Zhang experiments the rpm used where from 500 to 2500 rpm.

4. CONCLUSIONS

- The introduction of the microbubbles as a cleaning agent for the elimination of fouling at the pre rising step show no significant difference in comparison to using pure water.
- As temperature increases the fouling loss on both MIW and pure water increased.
- Rotation has less effect on the amount of cleaning on the milk residue in relation with the change in temperature.

5. RECOMMENDATIONS

- Use different detergents solutions in addition to the microbubble-infused water in comparison to pure water.
- Find an optimization of maximum cleaning temperature and cost for the cleaning.
- Reproduce the experiment with higher rpm to see if it has a higher effect on the percentage of cleaning.
- Evaluate the efficiency of using MIW as cleaning agent in the aspect of water usage.

6. REFERENCES

Agarwal, A., Xu, H., Ng, W. J., & Liu, Y. (2012). Biofilm detachment by self-collapsing air microbubbles: a potential chemical-free cleaning technology for membrane biofouling. J. Mater. Chem. 22:2203–2207. en. doi:10.1039/C1JM14439A.

AQUASTAT - FAO's Global Information System on Water and Agriculture. 2020. [place unknown]: [publisher unknown]; [updated 2020 Aug 3; accessed 2020 Sep 14]. http://www.fao.org/aquastat/en/.

Asselt A., Vissers M., Smit F., de Jong P. 2005. In-line control of foul-ing. In: Proceedings of Heat Exchanger Fouling and Cleaning: Challenges and Opportunities. Engineer-ing Conferences International; Kloster Irsee, Germany.

Bansal, B., & Chen, X. D. (2006). A Critical Review of Milk Fouling in Heat Exchangers. Comprehensive Reviews in Food Science and Food Safety. 5:27–33. en. doi:10.1111/j.1541-4337.2006.tb00080.x.

Burton H. 1968. Section G. Deposits from whole milk in heat treatment plant—a review and discussion. Journal of Dairy Research. 35:317–330. doi:10.1017/S0022029900019038

Chesters, S. P., Armstrong, M. W., & Fazel, M. (2014). Microbubble RO membrane cleaning reduces fouling on WWRO plant. Desalination and Water Treatment. 55:2900–2908. doi:10.1080/19443994.2014.940225.

Dhage B., Dhage A. (2016). Automation of CIP Process in dairy industries using programmable controllers and SCADA. In: 2016 International Conference on Automatic Control and Dynamic Optimization Techniques (ICACDOT). [place unknown]: [publisher unknown]; p. 318–323. https://www.researchgate.net/publication/315512491_Automation_of_CIP_Process_in_dairy_industries_using_programmable_controllers_and_SCADA.

Faye B, Konuspayeva G. 2012. The sustainability challenge to the dairy sector – The growing importance of non-cattle milk production worldwide. International Dairy Journal.24:50–56. doi:10.1016/j.idairyj.2011.12.011.

Food and Agriculture Organization (FAOSTAT). 2020. FAO's Global Information System on Dairy Production FAO; [updated 2020 Sep 3; accessed 2020 Sep 14]. http://www.fao.org/faostat/en/#data/QL.

Goode, K. R., Asteriadou, K., Robbins, P. T., & Fryer, P. J. (2013).Fouling and Cleaning Studies in the Food and Beverage Industry Classified by Cleaning Type. Comprehensive Reviews in Food Science and Food Safety.12:121–143. doi:10.1111/1541-4337.12000.

Lee E., Y., Kim H, Jang. A. 2015. Influence of microbubble in physical cleaning of MF membrane process for wastewater reuse. Environ Sci Pollut Res. 22:8451–8459. en. doi:10.1007/s11356-014-3928-y.

Miyamoto M, Ueyama S, Hinomoto N, Saitoh T, Maekawa S, Hirotsuji J. 2017. Degreasing of Solid Surfaces by Microbubble Cleaning. Japanese Journal of Applied Physics.46:1236–1243. doi:10.1143/JJAP.46.1236.

Smithers GW. 2015. Whey-ing up the options – Yesterday, today and tomorrow. International Dairy Journal.48:2–14. doi:10.1016/j.idairyj.2015.01.011.

Temesgen T, Bui TT, Han M, Kim T-i, Park H. 2017. Micro and nanobubble technologies as a new horizon for water-treatment techniques: A review. Advances in Colloid and Interface Science. 246:40–51. doi:10.1016/j.cis.2017.06.011.

Visser, J., & Jeurnink, T. J. M. (1997). Fouling of heat exchangers in the dairy industry. Experimental Thermal and Fluid Science.14:407–424. doi:10.1016/S0894-1777(96)00142-2.

Watabe, T., Matsuyama, K., Takahashi, T., & Matsuyama, H. (2014). Use of microbubbles to reduce membrane fouling during water filtration. Desalination and Water Treatment. 57:3820–3826. doi:10.1080/19443994.2014.991946.

Yang W, Li D, Chen XD, Mercadé-Prieto R. 2018. Effect of calcium on the fouling of whey protein isolate on stainless steel using QCM-D. Chemical Engineering Science.177:501–508. doi:10.1016/j.ces.2017.12.004.

Zhang BY, Lu J, Huang J-Y. 2019. Effect of sugar on the fouling behavior of whey protein. Food and Bioproducts Processing.113:2–9. doi:10.1016/j.fbp.2018.10.002.

Zhang BY, Xu S, Villalobos-Santeli JA, Huang J-Y. 2020. Fouling characterization of camel milk with comparison to bovine milk. Journal of Food Engineering. 285:110085. doi:10.1016/j.jfoodeng.2020.110085.

Zhang W, Ding L, Jaffrin MY, Tang B. 2017. Membrane cleaning assisted by high shear stress for restoring ultrafiltration membranes fouled by dairy wastewater. Chemical Engineering Journal.325:457–465. doi:10.1016/j.cej.2017.05.076.

Zouaghi S, Six T, Nuns N, Simon P, Bellayer S, Moradi S, Hatzikiriakos SG, André C, Delaplace G, Jimenez M. 2018. Influence of stainless steel surface properties on whey protein fouling under industrial processing conditions. Journal of Food Engineering.228:38–49. doi:10.1016/j.jfoodeng.2018.02.009.