Design and evaluation of microbubble-based cleaning process of membranes fouled by oily wastewater

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Abstract. As an alternative for the conventional clean-in-place (CIP) process for microfiltration membranes, a novel cleaning technology based on microbubbles has been recently available. Microbubbles are known for their ability to generate pressure waves through shrinking and subsequent self-collapsing phenomenon which causes foulant removal. In addition, they have hydrophobic surfaces which have the potential to attract organic substances like oil. In the present study, the effectiveness of microbubbles in palm oil foulant removal from the membranes in the first two stages of the CIP cleaning process was investigated and compared with the conventional process, which uses water and sodium hydroxide (NaOH) as cleaning agents. Vinylidene Polyfluoride (PVDF) microfiltration membranes of 0.1 μm pore size were fouled with an oil-in-water emulsion (O/W) made of palm oil. The emulsion was recirculated by a laboratory scale filtration system at 7 L/min and 155 kPa of transmembrane pressure (TMP). A 75% flux recovery was observed after the application of microbubbles in the first two steps of the CIP process compared to the 22% flux recovery obtained with the control without microbubbles. The use of microbubbles for membrane cleaning purposes proved to restore the membrane flux to a better performance level. The results of this study suggest that the use of microbubbles is more efficient in foulant detachment compared to the conventional cleaning process.

Keywords: Clean-in-place, cross-flow filtration, microfiltration.

Resumen. Como una alternativa al sistema convencional de limpieza *in situ* (CIP) para membranas de microfiltración, se desarrolló una tecnología de limpieza novedosa basada en el uso microburbujas como agentes de limpieza. Las microburbujas son conocidas por su habilidad de generar ondas de presión a través del fenomeno de colapso por reducción en diámetro que causa la remoción de la suciedad de la membrana, además, poseen propiedades hidrofóbicas lo que les permite interactuar con componentes hidrofóbicos adheriendolos a su superficie. En el presente estudio, se investigó la eficacia de las microburbujas para remover la capa de aceite de las membranas en las primeras dos etapas del proceso de limpieza *In Situ* (CIP) comparandolo con el proceso convencional el cual utiliza agua e hidróxido de sodio (NaOH) como agentes de limpieza. Membranas de microfiltración de Polifloruro de Vinilideno (PVDF) de 0.1 μm tamaño de poro fueron ensuciadas con una emulsión de aceite en agua (O/W). La emulsión se recirculó por un sistema de filtración a escala de laboratorio a 7 Lpm y a una presión de transmembrana (TMP) de 155 kPa. Se observó un 75% de recuperación del flujo despues de la aplicación de microburbujas en las primeras dos pasos del proceso CIP comparado con un 22% de recuperacion de flujo que se obtuvo con el control, en el cual no se utilizó microburbujas. El uso de microburbujas con fines de limpieza de membranas demostró restaurar el flujo de la membrana a un mejor nivel de rendimiento. Los resultados de este estudio sugieren que el uso de microburbujas es más eficiente en la remoción de suciedad en comparación con el proceso de limpieza convencional.

Palabras clave: Filtración por flujo cruzado, limpieza *In situ*, microfiltración.

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1. INTRODUCTION

Nowadays, with the industrial development, there is an increment in the amount of wastewater production, especially in the food industry. According to Liu *et al*. 2013, oily and organic emulsions are the most harmful pollutants to the environment because they can affect drinking water and groundwater resources, and endanger human health. Therefore, the environmental protection agencies and the governments of many countries, require that all industrial wastewater, generated in plant during food processing, must be treated before discharge, to minimize its impacts on the environment.

The greatest amount of wastewater is produced during the cleaning and sanitizing processes of the food manufacturing plants. Nonetheless, cleaning and sanitization are two very important steps in food processing to ensure the food quality and the food safety. Since all food destined to human consumption has to be safe, all the equipment in food processing plants must be cleaned and sanitized after each batch of production (Varzakas 2015), making it difficult to reduce the wastewater production.

Wastewater can be treated by membrane filtration to separate different waste substances produced in food processing. Currently, wastewater treatment processes are carried out through membrane filtration processes. There are many types of filtration systems such as microfiltration, nanofiltration, ultrafiltration and reverse osmosis (Cheryan 1998). Compared to dead-end filtration, cross-flow microfiltration systems are more widely used for oily wastewater treatment (Liu *et al*. 2013). However, although membrane filtration is very effective in wastewater treatment, oily deposits can accumulate on the membrane (i.e., fouling) and reduce the filtration efficiency over time, hence the membrane must be cleaned regularly to ensure its performance.

The membrane cleaning process is performed by a Clean-In-Place (CIP) protocol using water and chemical detergents (i.e., cleaning agents) to remove the fouling layer from the surface of the membrane (Memisi *et al*. 2015). However, the main disadvantages of this process is high water and chemical consumption, which can generate a great amounts of wastewater in each cleaning operation (Guo *et al*. 2012). Alkaline compounds such as sodium hydroxide (NaOH) are used for removal of the organic phase and strong acidic compounds like hydrocloric acid (HCl) are used for the removal of the inorganic phase of the fouling layer. The use of chemicals, as a cleaning agents, for membrane cleaning purposes, is extremely polluting to the environment, because the resulting waters after the cleaning process are difficult to treat (Garmsiri *et al*. 2017).

Another important disadvantage of the CIP cleaning process is the high energy consumption because the cleaning cycle uses hot water in the intermediate stages between each cleaning step to remove the excess of chemical detergents. Due to the fact that the cleaning process is carried out frequently, the high consumption of water and energy are reflected as a negative impact to the environment (Pettigrew *et al*. 2015).

One of the trends in the food industry in the 21st century is the increasing demand for green technologies, in order to minimize the negative impacts to the environment that food processing plants generates (Hima *et al*. 2007). Nowadays, new products are being developed with eco-friendly technologies with the aim of reducing their carbon footprint and wastewater production. In recent years, microbubbles have achieved great attention due to their wide range of applications in different fields of science and technology (Agarwal *et al*. 2011). In the field of wastewater treatment technologies, the application of microbubbles has shown promising results as green cleaning agents (Agarwal *et al*. 2012).

Microbubbles are bubbles generated in water with a diameter less than 50 µm, which have three main properties, (1) large surface area, (2) hydrophobic surface that can adsorb organic molecules such as fat, and (3) an extended recidence time in solution (Miyamoto *et al*. 2017). Due to this properties, microbubbles can be considered as cleaning agents, therefore, they can be applied to a membrane cleaning processes in order to reduce the fouling layer from the membrane surface. Nowadays, three possible mechanisms by which microbubbles can reduce the fouling layer from the membrane are being studied, (1) foulant detachment due to the shear stress generated by injecting air in the form of microbubbles (Watabe *et al*. 2016), (2) adsorption of foulant on microbubbles surface due to hydrophobic interactions (Miyamoto *et al*. 2017), and (3) foulant detachment by self-collapse of microbubbles. (Takahashi *et al*. 2003, 2007; Agarwal *et al*. 2012).

Based on these properties of the microbubbles, the present study included the following objectives:

- Design a bench-scale cross-flow microfiltration system that includes a Microbubble Generator Device.
- Evaluate the cleaning efficacy of the microbubbles as cleaning agents in the pre-rinse step and pre-rinse & alkali steps of the CIP process for membrane cleaning.
- Compare the cleaning efficiency of microbubbles as cleaning agents, with the conventional membrane cleaning process that uses alkaline chemical detergents.

2. MATERIALS AND METHODS

Location.
This study was conducted in the Food Process Sustainability Laboratory in the Food Science Department of Purdue University, West Lafayette IN, United States.

Phase 1. Design and assembly of a bench-scale filtration system.
A bench-scale cross-flow microfiltration system was designed and assembled for the treatment of the oil-in-water emulsion (O/W), simulating oily wastewater treatment, and adapted to introduce the microbubbles solution, which is generated by a Microbubble Generator Device (MGD), as a cleaning agent during the clean in place process (CIP) cleaning process.

The base design of the system is composed of a cross-flow cell made of acrylic material, in which flat sheet membranes were placed, a feed tank of 10 liters capacity, a positive displacement pump connected to a 1 HP motor, which is controlled by a motor driver (VFD). The purpose of adding a motor driver to the system is to have a better control of the transmembrane pressure (TMP) of the system during the experiments, since it is an important factor in the treatment of oily waters. The entire system is connected through a flexible tubing of 3/8 inch of internal diameter and the permeate output that uses flexible tubing of 1/8 inch of internal diameter.

The system is composed of five lines; (1) concentrate feed line in which high pressure flex tubing was used, and the other four lines; (2) bypass line, (3) concentrate return line, (4) permeate output line and (5) drain line low pressure flex tubing, was used. Needle valves were placed in the bypass and concentrate feed lines for a better control of the feed flow rate. In the rest of the system, gate valves were placed to be able to condition the system during fouling tests and subsequently modify the system for cleaning tests. For permeate collection, a 3,000 ml capacity beacker was used, and, for the measurement of the permeate mass an analytical balance was used.

The system was modified, through the addition of valves in the lines, to allow the entry of the microbubble solution during the cleaning tests. The Microbubble Generator Device (MGD) is an annex system to the cross-flow microfiltration system, which was used to generate a microbubble solution that was introduced to the system during the cleaning tests. This system is composed of the MGD, and, a tank of 190 liters capacity. Table 1 shows the parts and equipment that compound the cross-flow microfiltration system.

Table 1. Specifications of the component parts of the bench-scale filtration system.

No.	Part	Description
1	Conical feed tank	10 L max capacity, stainless steel.
2	Feed pump	Poly-Diaphragm pump model DP-43-P, 1Hp, Max 650 rpm, max 218 PSI, max 5 GPM @ max PSI. ¾´ inlet& ½´outlet.
3	Motor	1 Hp, 1760 RPM, 3PH, 60 Hz. Model BALD-CEM3546.
4	Motor driver	AC 1PH, 220V, 50/60Hz. ATO model GK3000-2S0015G.
5	Flow meter	Blue-White F-550, max 2 GPM.
6	Drain valve (tank)	1´ OD, stainless steel.
7	Bypass control valve	3/8´ Needle valve, stainless steel
8	Concentrate pressure gauge	Max 400 PSI.
9	CF042 Cell	Acrylic cell model CF042, max 400 PSI.
10	Concentrate control valve&gauge assembly	3/8´ Stainless steel needle valve & pressure gauge.
11	Scale for measuring permeate mass	Analytical balance for mass measure, max 10 Kg.
12	permeate collector	Beaker, 3000 mL capacity.
13	Pressure relief valve	3/8´ 200 PSI max pressure.
14	Concentrate feed line	3/8´ high pressure rigid tubing.
15	Bypass line	3/8´ low pressure flex tubing.
16	Concentrate return line	3/8´ low pressure flex tubing.
17	Permeate output line	1/8´ low pressure rigid tubing.
18	Drain line	3/8´ low pressure flex tubing.
19	Pressure relief line	3/8´ low pressure flex tubing.
20	Control valves	3/8´ OD, in-line plastic valves.
21	Microbubble Generator Device (MGD)	Nikuni pump A3:C22 17 LPM water flow rate, 1.3 NL/min air flow rate, model KTM20ND

Determination of the operating parameters. Before running the tests, a system characterization was performed using flat sheet microfiltration membranes (MF) to establish the operating parameters of the system for the experiment; inlet pressure (IP), outlet pressure (OP), trans membrane pressure (TMP), feed flow rate (QF) and the oil-in-water solution treatment time. Table 2 shows the characteristics of the micorfiltration membrane used for the tests.

Table 2. Flat sheet microfiltration membrane features.

Model	Feed	Applications	pH range @ 25 ºC	Flux ($L/m^2/h$) /kPa	Pore size/ MWCO	Polymer
V0.1	Industrial/ Dairy	Fat/microbial Removal, Protein Fractionation	1−11	402−431 /138	0.1 µm	PVDF

Flux = flow rate per unit area. MWCO = Molecular Weight Cut-Off. PVDF = Vinylidene Polyfluoride.
Source: (Sterlitech Corporation 2019) adapted by the author.

The microfiltration membrane is made of Vinylidene Polyfluoride (PVDF), a chemically inert polymer and resistant to sudden changes in pH (Synder 2019). The membrane fouling time was established based on the minimum membrane operating flux of 402 $L/m^2/h$ @ 138 kPa TMP, operation below this point indicates that the membrane is completely fouled. Equation 1 was used to calculate the TMP.

$$TMP = \frac{IP+OP}{2} \quad [1]$$

Where:
TMP= Trans membrane pressure.
IP= Inlet pressure.
OP= Outlet pressure.

Phase 2. Experimental setup & operation.

Experimental design and statistical analysis. The investigation was carried out using a Completely Randomized Design (CRD) with two replicates for the control and three replicates for the treatments using microbubbles. Data obtained from all the tests was evaluated for significance by an analysis of variance (ANOVA) using SAS® software version 9.4, and when the effect of the factors was significant ($P < 0.05$), the Duncan test was applied at 95% probability.

Table 3 shows the description of the treatments for evaluating the effect of the cleaning protocol on the membrane performance at pre-rinse step. For the membrane performance after each treatment, the results are presented in percentage of flux recovery.

Table 3. Description of the treatments to evaluate the cleaning efficiency of microbubbles at pre-rinse step.

Treatment	Description
1	Cleaning with microbubbles
2	Cleaning with the conventional protocol

Table 4 shows the description of the treatments for evaluating the efect of the cleaning protocol on the membrane performance pre-rinse & alkali steps. For the membrane performance after treatment, the results are presented in percentage of flux recovery.

Table 4. Description of the treatments to evaluate the cleaning efficiency of microbubbles at pre-rinse & alkali steps.

Treatment	Description
1	Cleaning with microbubbles
2	Cleaning with the conventional protocol.

Membrane preconditioning process. For the preconditioning of the microfiltration membranes, RO water (deionized water) was recirculated in the system. This process was only performed on completely new membrans. It is also performed on used membranes that have not been used for more than 24 hours. The purpose of preconditioning was to hydrate the microfiltration membranes before being used. The microfiltration membranes were placed inside the cross-flow filtration cell, the feed tank was filled with RO water and allowed to run until the permeate flow became stable, the estimated total time was 60 minutes working at 155 kPa transmembrane pressure (TMP).

Membrane fouling process.

Preparation of the oil in water emulsion. An oil-in-water (O/W) emulsion (model oily wastewater) was prepared as follows:

- Mixing 10 g of palm oil, 1,000 g of distillate water and 1 g of surfactant Tween 20® (polyoxyethylene (20) sorbitan monolaurate) at 10,000 rpm using a laboratory homogenizer (Ultra-Turrax® T-50, IKA Labortechnik, Germany) for 50 minutes.

- Dilute the mixture by adding 9,000 g of distillated water for the final oil/water concentration of 0.1 wt% (Garmsiri *et al*. 2017).

- Mix at 10,000 rpm for 5 more minutes to homogenize the emulsion.

Oil in water emulsion treatment. For the membrane fouling, an oil-in-water emulsion (0.1 wt%) was recirculated in the system at 155 kPa transmembrane pressure (TMP), and feed flow rate of 7 L/min for 135 min, simulating oily wastewater treatment. Figure 1 shows the operation of the cross-flow microfiltration system, blue pipes show the oily wastewater treatment cycle, white pipes were not used in this stage of the experiment.

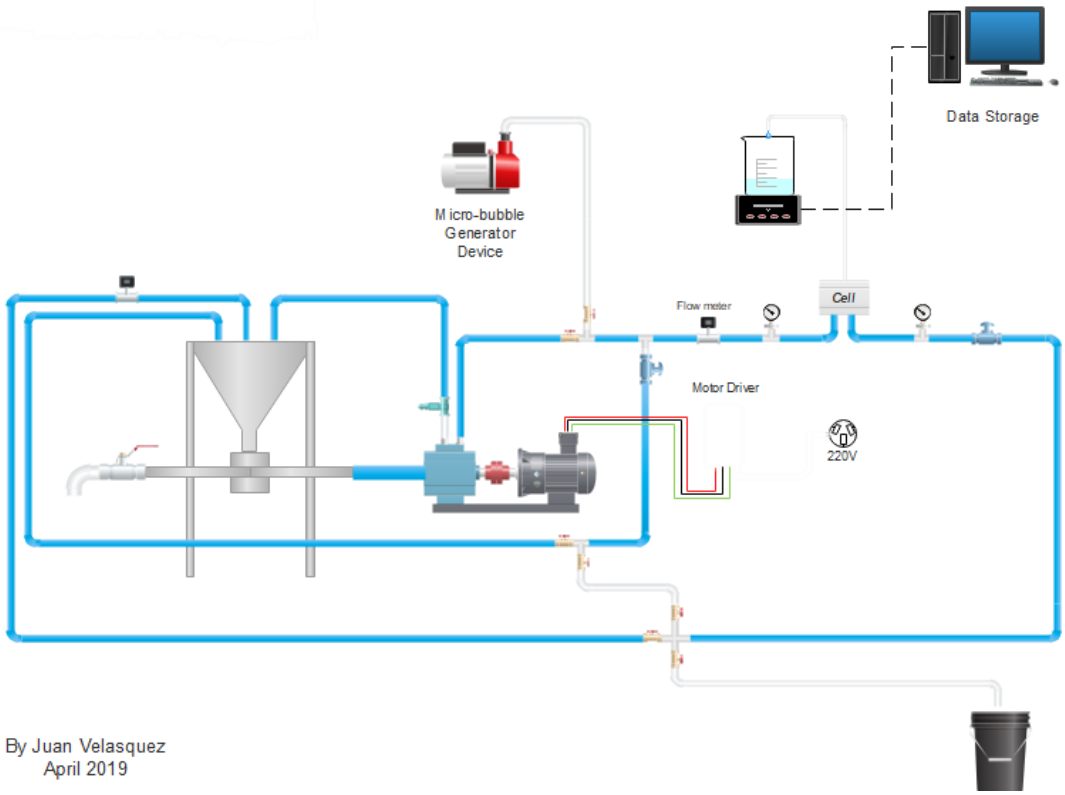

Figure 1. Schematic flow diagram of the oily wastewater treatment cycle for membrane fouling.

Membrane cleaning process.

For membrane cleaning evaluation it was divided in two experiments, first one, was the evaluation after cleaning at pre-rinse step (Table 3), and the second one which was the evaluation after cleaning in the pre-rinse and alkali steps (Table 4). Each experiment had its own control treatment wich was the conventional cleaning process.

Conventional cleaning process. For this stage of the experiment, the cleaning process was based on the cleaning protocol recommended by the membrane manufaturer (Sterlitech Corporation 2019). This protocol simulates the Clean-In-Place (CIP) cleaning of membranes process. Table 5 shows the CIP protocol for fouled membranes, and it is based on chemical usage.

Table 5. Clean In Place (CIP) steps for conventional membrane cleaning process.

#	Step	T (°C)	CFV (m/s)	TMP (kPa)	Time (min)
1	Pre-rinse (water)	25±2	1.29	155.132	10
2	Alkali (0.05% NaOH)	40±2	1.29	155.132	20
3	Intermediate rinse (water)	25±2	1.29	155.132	10
4	Acid (0.1% HCl)	40±2	1.29	155.132	20
5	Final rinse (water)	25±2	1.29	155.132	10

T = Temperature. CFV = Cross flow velocity. TMP = Trans membrane pressure.
Source: (Sterlitech Corporation 2019), adapted by the author.

Alkali step uses Sodium Hydroxide (NaOH) to remove the organic foulant, acid step uses strong acidic compounds like Hydrochloric Acid (HCl) to remove non-organic materials, and, the rinse steps use fresh water to remove the excess of chemicals from the membrane surface. Equation 2 was used to calculate the cross flow velocity (CFV).

$$CFV = \frac{QF}{Acs} \quad [2]$$

Where:
CFV= Cross Flow Velocity (m/s)
QF= Feed flow rate (m^3/s)
Acs= Feed chanel cross sectional area (m^2)

Table 6 shows the experimental steps that was followed to evaluate the effect of the conventional cleaning protocol after cleaning at the pre-rinse step. As observed the conventional protocol only uses water at this step, and this was the control for comparison with the other prtotocol which uses a microbubble solution instead of water in this step of the CIP.

Table 6. Experimental steps for membrane cleaning at pre-rinse step using the conventional protocol.

#	Step	CFV (m/s)	TMP (kPa)	Time (min)
1	[¥]Membrane preconditioning (RO water)	1.29	155.132	60
2	Oily wastewater treatment	1.29	155.132	135
3	Membrane cleaning stage (CIP)			
	Pre-rinse (water)	1.29	155.132	10
4	After cleaning (RO water)	1.29	155.132	60

[¥]Preconditioning step with Reverse Osmosis water only applies to a fresh membranes.
CFV = Cross flow velocity.
TMP = Transmembrane pressure.

Table 7 shows the experimental steps that was followed to evaluate the effect of the conventional cleaning protocol after cleaning at the pre-rinse & alkali steps. As observed the conventional protocol only uses water in the pre-rinse step and an alkali solution with sodium hydroxide (0.05% NaOH) in the second step. This was the control treatment for comparison with the other cleaning protocol which uses a microbubble solution instead of water and alkali solutions in this steps of the CIP.

Table 7. Experimental steps for membrane cleaning at pre-rinse & alkali steps using the conventional protocol.

#	Step	CFV (m/s)	TMP (kPa)	Time (min)
1	¥Membrane preconditioning (RO water)	1.29	155.132	60
2	Oily wastewater treatment	1.29	155.132	135
3	Membrane cleaning stages (CIP)			
	Pre-rinse (water)	1.29	155.132	10
	Alkali (NaOH 0.05%)	1.29	155.132	20
4	After cleaning (RO water)	1.29	155.132	60

¥Preconditioning step with Reverse Osmosis water only applies to a fresh membranes.
CFV = Cross flow velocity.
TMP = Transmembrane pressure.

Cleaning with microbubbles.

Microbubbles solution preparation. For the generation of a stable solution of microbubbles, the Microbubbles Generator Device (MGD) was used. This device operates with an air flow rate of 3 L/min, and takes 15 minutes to generate a microbubbles stable solution of 190 liters according to previous studies carried out by Chung 2018. After that, the solution was introduced to the membrane filtration system at the steps of the CIP process established in the experimenal protocols. Table 8 shows the experimental steps that was followed to evaluate the use of microbubbles, as cleaning agents, in the pre-rinse step. As observed water was replaced for the use of microbubbles in this step of the CIP process.

Table 8. Experimental steps for membrane cleaning using microbubbles at pre-rinse step.

#	Step	CFV (m/s)	TMP (kPa)	Time (min)
1	¥Membrane preconditioning (RO water)	1.29	155.132	60
2	Oily wastewater treatment	1.29	155.132	135
3	Membrane cleaning stages (CIP)			
	Pre-rinse (Microbubbles)	1.29	155.132	10
4	After treatment (RO water)	1.29	155.132	60

¥Preconditioning step with Reverse Osmosis water only applies to a fresh membranes.
CVF = Cross flow velocity.
TMP = Transmembrane pressure.

Table 9 shows the experimental steps that was followed to evaluate the use of microbubbles, as cleaning agents, in the pre-rinse & alkali steps. As can be observed water and alkali solution was replaced for the use of microbubbles solution in those steps of the CIP process.

Table 9. Experimental steps for membrane cleaning using microbubbles at pre-rinse & alkali steps.

#	Step	CFV (m/s)	TMP (kPa)	Time (min)
1	¥Membrane preconditioning (RO water)	1.29	155.132	60
2	Oily wastewater treatment	1.29	155.132	135
3	Membrane cleaning stages (CIP)			
	Pre-rinse (Microbubbles)	1.29	155.132	10
	Alkali (Microbubbles)	1.29	155.132	20
4	After cleaning (RO water)	1.29	155.132	60

¥Preconditioning step with Reverse Osmosis water only applies to a fresh membranes.
CFV = Cross flow velocity.
TMP = Transmembrane pressure.

Figure 2 shows the CIP cleaning cycle by introducing the microbubbles as cleaning agents, blue pipe shows the cleaning cycle, white pipes were not used in this stage.

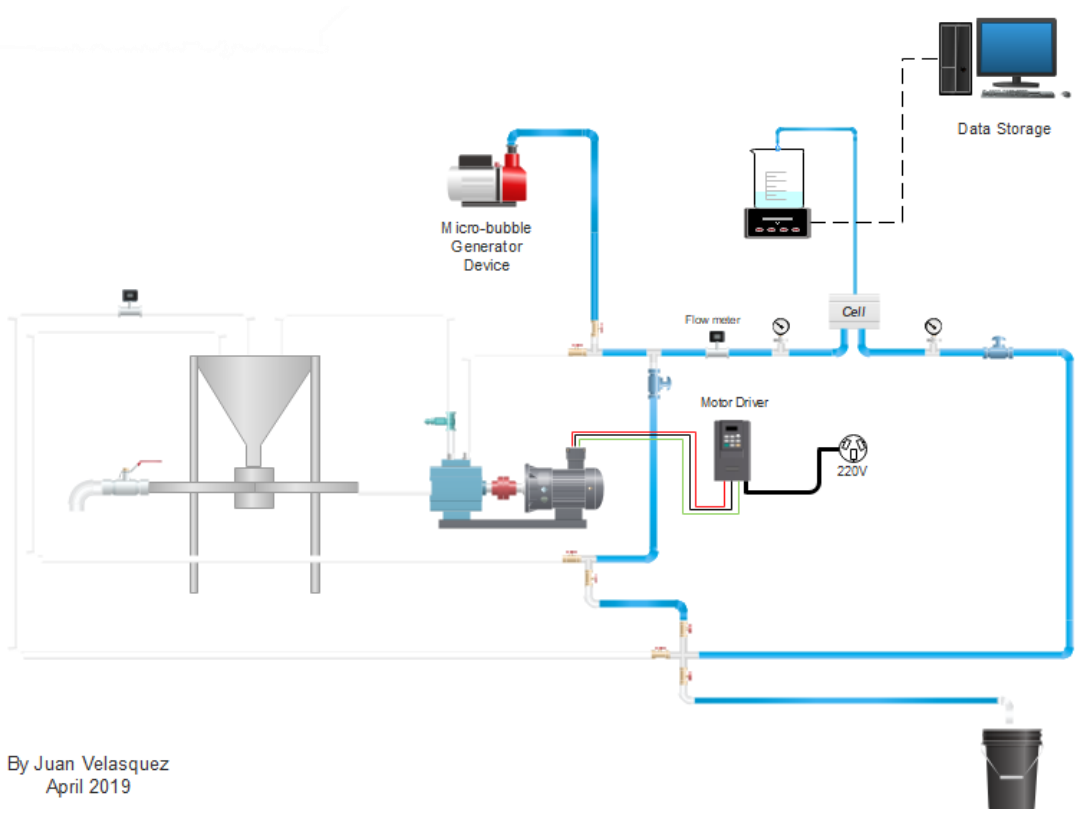

Figure 2. Introduction of microbubble-infused water for membrane cleaning.

After clening stage. After each cleaning test, reverse osmosis water (deionized water) was recirculated at 155 kPa TMP for 60 min to calculate the flux recovery (*FR)* of the membrane.

Variables and equations. The filtration system reports the weight of the permeate (*p*) over time (*t*), therefore, it is necessary to calculate the permeate flow (P_f) and the membrane flux (*F*). Equation 3 shows that P_f is calculated dividing *p* by the density of the solution (ρ) as a function of the elapsed time (*t*). From equation 4, *F* is obtained, which results from the division of P_f by the specific area of the membrane (*A*).

To calculate the membrane performance after each treatment it is necessary to calculate the membrane flux recovery (*FR*). Equation 5 shows that *FR* results from the division of the difference of flux after cleaning (F_a) and flux before cleaning (F_b), by the difference of initial flux (I_f) and flux before cleaning (F_b) multiplied by 100. (Kazemimoghadam y Mohammadi 2007)

$$P_f = \frac{p/\rho}{t} \quad [3]$$

$$F = \frac{P_f}{A} \quad [4]$$

$$FR = \left[\frac{F_a - F_b}{I_f - F_b}\right] \times 100 \quad [5]$$

Where:
F= Membrane flux ($L/m^2/h$)
P_f= Permeate flow rate (L/h)
A= Membrane specific area (m^2)
p= Permeate weight (g)
ρ= Density of the solution (g/cm^3)
t= Time (min)
FR= Flux recovery (%)
F_a= Flux after cleaning ($L/m^2/h$)
F_b= Flux before cleaning ($L/m^2/h$)
I_f= Initial flux (pre-conditioned new membrane) ($L/m^2/h$)

3. RESULTS AND DISCUSSION

Phase 1. Bench-scale cross-flow microfiltration system.

The base design of the system was taken from Sterlitech Corporation USA, and adpted in order to introduce microbubbles during the cleaning tests. The cross-flow microfiltration system was designed and developed following specifications reported by Mueller *et al*. 1997 & Mirsaeedghazi *et al*. 2010 in the description of their experiments using filtration systems. A user manual was developed in order to describe the correct manipulation and adjustment of the system before perform an experiment. The system, basically, is composed of a cross-flow filtration cell made of acrylic material in which the flat sheet microfiltration membranes are placed, a pump with a capacity up to 19 L/min, a conical feed tank of 10 liters capacity and an analytical balance to quantify the permeate mass. Pressure gauges and flow meter were added to the system to control the operating parameters. Figure 3 shows the flow of the bench-scale microfiltration system and its component parts.

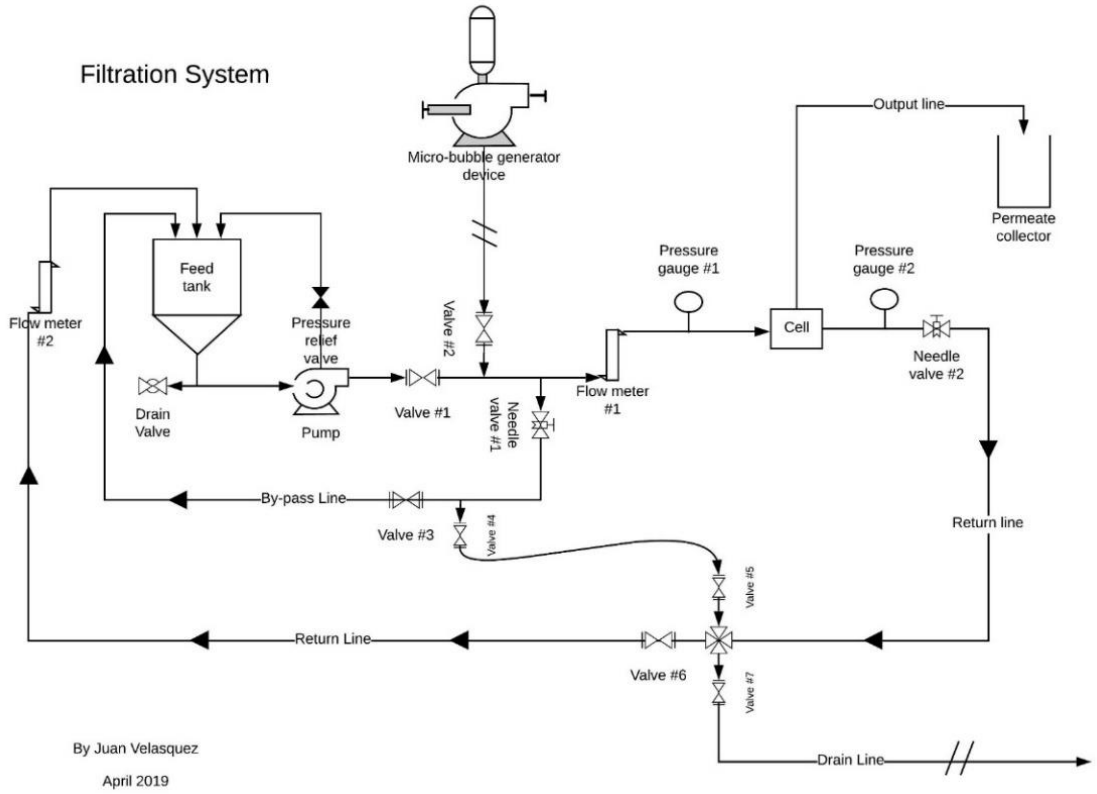

Figure 3. Bench-scale cross-flow microfiltration system schematic flow diagram.

Operating parameters.

The operating parameters of the system were established based on the specifications of the microfiltration membranes (Table 2). Since the feed flow rate is directly related to the pressure, it was set at 7 liters per minute to maintain a constant transmembrane pressure (TMP) of 155 kPa. Table 10 describes the operating parameters of the cross-flow microfiltration system that were used for the experiments.

Table 10. Microfiltration system operation parameters.

Parameter	Condition
Feed flow rate	7 L/min
Inlet pressure (IP)	172 kpa
Outlet pressure (OP)	138 kPa
Transmembrane pressure (TMP)	155 kPa

Phase 2. Experimental procedure.

Oil in water emulsion. According to the characterization of the oil-in-water emulsion carried out by Garmsiri *et al*. 2017, the size diameter of the oil droplets are in a range between 0.1 µm to 0.201 µm. Therefore, microfiltration membranes of 0.1 pore size were used for the treatment of the oil in water emulsion.

Membrane fouling by an oil in water emulsion. According to Tummons *et al*. 2016, during the treatment of oil-in-water emulsions through a cross-flow microfiltration system, there are three characteristic stages of membrane fouling: (1) oil droplet attachment and clustering, (2) oil droplet deformation, and (3) oil droplet coalescence. Based on these three stages, oil droplets of different sizes are created from the oil droplets of the emulsion during the membrane fouling stage. These oil droplets are attached in the membrane surface creating a fouling layer and thus reducing the permeate flow rate. In the third stage there is a fouling removal due to the drag force of the crossflow, this can be an important phenomenon which can enhance the cleaning efficacy of microbubbles during the membrane cleaning stage.

Microbubbles solution. A bench-scale Microbubble Generator Device (MGD) was used to create a stable solution of microbubbles. According to the characterization carried out by Chung 2018 the generated microbubbles have a diameter less than 20 µm. Lee *et al*. 2015 reported that microbubbles with diameters smaller than 10 µm are more stable in solution. Stabilization in solution increases the efficacy of microbubbles for cleaning purposes, because they have greater stability and therefore the coalescence rate is lower (Temesgen *et al*. 2017). Figure 4 shows the distribution of the microbubbles according to its diameter size.

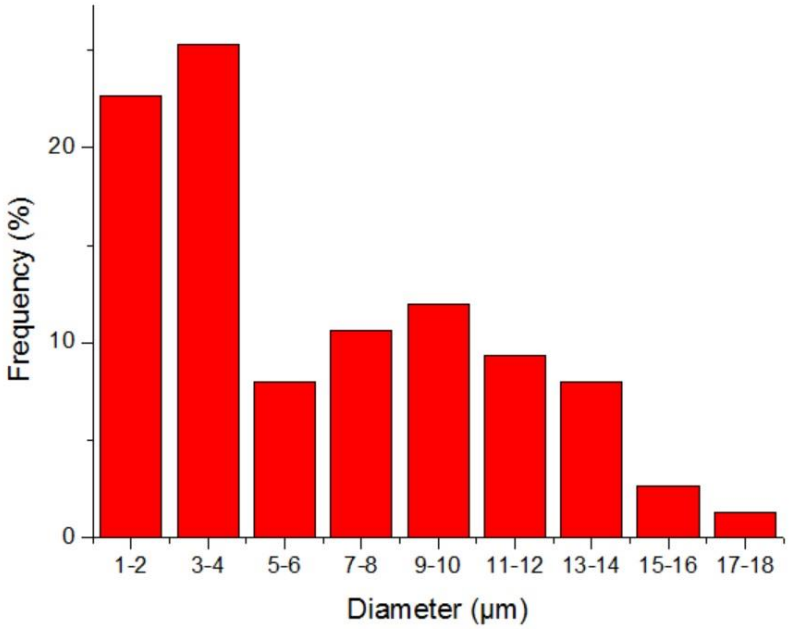

Figure 4. Microbubbles size characterization.
Source: Chung 2018.

As observed in Figure 4, the MGD generates microbubbles with a diameter less than 20 µm, and around 50% of the microbubbles are in a diameter range of 1-4 µm. So, this means that the microbubbles in solution are stable and the coalescence rate is lower. Stability in solution are an important parameter because it is necessary to keep the microbubble integrity from the production unit (MGD) to the place were it will be used (membrane).

Using microbubbles to reduce membrane fouling.

It is assumed that the efficacy of microbubbles in membrane defouling is based on the combination of three possible mecanisms that have been studied recently. (1) Foulant detachment due to the shear stress by injecting air in the form of microbubbles (Watabe *et al*. 2016), (2) adsorption of foulant on microbubbles surface (Miyamoto *et al*. 2017), and (3) foulant detachment by self-collapse of microbubbles. (Takahashi *et al*. 2003, 2007; Agarwal *et al*. 2012).

Table 11 shows the results of the flux obtained at each step of the experimental protocol, initial flux, flux before cleaning and flux after the cleaning process respectivelly, only at the pre-rinse stage. Based on the results of flux of each step of the experiment, the flux recovery was calculated using equation 5. This parameter is given in percentage, and, it was used as an indicator for the efficacy of the cleaning protocol applied in the first step of the CIP process.

Table 11. Membrane flux (L/m^2/h) at each stage of the experimental protocol for cleaning at pre-rinse step.

Treatment	Initial	Before cleaning	After cleaning
	Mean ± S.D.	**Mean ± S.D.**	**Mean ± S.D.**
Microbubbles	1845.36 ± 57.06^{A}	249.96 ± 12.52^{B}	351.37 ± 1.57^{B}
Conventional	1845.45 ± 56.02^{A}	341.37 ± 3.91^{A}	390.87 ± 3.75^{A}
%C.V.	3.07	3.65	0.68

$^{A-B}$Means with different capital letters in each column are statistically different ($P < 0.05$).
S.D. = Standard deviation.
%C.V. = Coefficient of variation.

As observed in Table 11, there were no significant statistical differences in the initial flux of the membranes used for both treatments, indicating that both experiments were performed with membranes that had the same initial flux. The flux before the cleaning process was statistically different for both treatments, indicating that there was a greater level of fouling in the membranes used for the cleaning treatments using microbubbles. The flux after cleaning was statistically different, indicating that the flux of the membrane after the cleaning process was higher in the treatments using the conventional cleaning protocol. Although it is observed that the flux after the cleaning process was higher in the treatment with conventional cleaning, it cannot be concluded that it was the best cleaning treatment because membranes with a lower degree of fouling were used for this experiment. For the evaluation of the efficiency of cleaning treatments, equation 5 was used to calculate the percentage of flux recovery after the application of each cleaning treatment.

Table 12 shows the results obtained from each step of the experimental protocol, initial flux, flux before cleaning, and flux after cleaning in the pre-rinse & alkali steps respectively. Based on the flux results of each step of the experiment, the flux recovery was calculated using equation 5. This parameter was used as an indicator of the efficiency of the applied cleaning protocol.

Table 12. Membrane flux (L/m^2/h) at each stage of the experimental protocol for cleaning at pre-rinse & alkali steps.

Treatment	Initial	Before cleaning	After cleaning
	Mean ± S.D.	**Mean ± S.D.**	**Mean ± S.D.**
Microbubbles	1844.99 ± 48.29^{A}	224.57 ± 40.64^{B}	1451.32 ± 5.20^{A}
Conventional	1829.94 ± 14.50^{A}	333.40 ± 3.42^{A}	663.92 ± 8.65^{B}
%C.V.	2.19	12.4	0.57

$^{A-B}$Means with different capital letters in each column are statistically different ($P < 0.05$).
S.D. = Standard deviation.
%C.V. = Coefficient of variation

As observed in Table 12, there were no significant statistical differences in the initial flux of the membranes used for both treatments, indicating that both experiments were performed with membranes that had the same initial flux. The flux before the cleaning process was statistically different for both treatments, indicating that there was a greater level of fouling in the membranes used for the cleaning treatments using microbubbles. The flux after cleaning was statistically different, indicating that the flux of the membrane after the cleaning process was higher in the treatments using microbubbles. Although it is observed that the flux after the cleaning process with microbubbles was higher, it cannot be concluded that it was the best cleaning treatment because it is necessary to calculate the flux recovery after the cleaning process. For the evaluation of the efficiency of the cleaning treatments, equation 5 was used to calculate the percentage of flux recovery after the application of each cleaning treatment.

Comparison between cleaning protocols.

The efficacy of microbubbles as fouling removal agents was tested in the first two stages of the CIP process (pre-rinse and alkali steps) for cleaning of microfiltration membranes fouled by oily wastewater treatment in a cross-flow filtration system. In previous studies it was found that the introduction of microbubbles solution during the pre-rinse and alkali steps of the CIP cleaning process restores the membrane performance to a better performance level than using the conventional membrane cleaning process which uses large amounts of water and excessive use of chemical detergents to restore the membrane performance.

Introduction of microbubbles into the pre-rinse step of the CIP process. The results obtained from the introduction of microbubbles during the pre-rinse step of the CIP process are shown in Figure 5. The control treatment was the use of water in the pre-rinse step, which is the conventional membrane cleaning process, and the treatments are the introduction of microbubbles in the same step of the CIP process.

In order to measure the effectiveness of the microbubbles as cleaning agents, the flux recovery was calculated after each treatment, and it was found that the cleaning process using microbubbles was more effective in removing the oil layer in the pre-rinse step of the CIP process. Harun & Zimmerman 2018 found a similar effect when they applied microbubbles during the cleaning process of a membrane fouled by surface seawater, they found that after microbubbling into the membrane, the flux was restored to a level similar to the original flux, indicating that microbubbles performed a cleaning effect on the membrane.

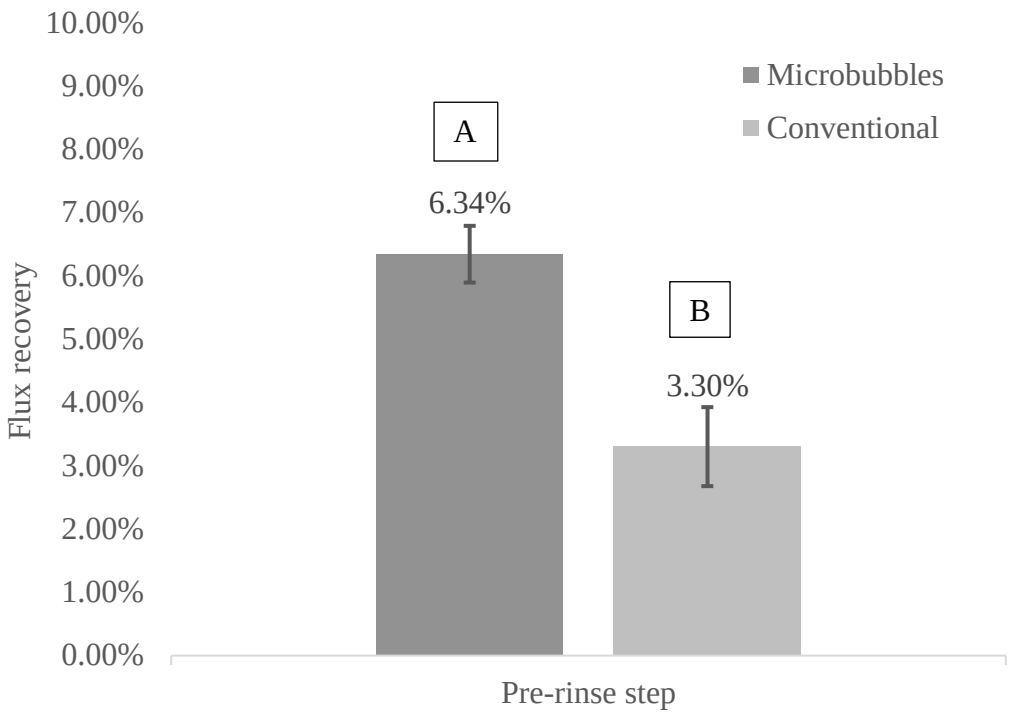

Figure 5. Cleaning efficiency of microbubbles based on percentage of membrane flux recovery after cleaning at pre-rinse step, compared to the conventional cleaning process (10% C.V.).
[A-B]Means with different letter are statistically different ($P < 0.05$).
%C.V. = Coefficient of variation.

As observed in Figure 5, there were significant statistical differences ($P < 0.05$) between both cleaning protocols, indicating that the use of microbubbles as cleaning agents was more efficient in cleaning the membrane during the pre-rinse step compared to the conventional cleaning process. Cleaning with microbubbles may remove the fouling from the membrane when they come in contact with it due to shear stress generated by injecting air in the form of microbubbles to the surface of the membrane (Watabe *et al*. 2016).

Figure 5 shows that using microbubbles, as a cleaning agents, replacing water during the first step (pre-rinse) of the CIP process, had a better efficiency than the conventional CIP process, restoring the membrane performance aproximately 3% more during the first step of the cleaning protocol. Since the microbubbles have properties of hydrophobic interaction they have the ability to adsorb hydrophobic foulant onto their surface and consequently reduce the oil layer of the membrane. Miyamoto *et al*. 2017 have reported the use of microbubbles for the degreasing of surfaces through the phenomenon of adsorption. So, it is assumed that the cleaning effect of the microbubbles was because the hydrophobic interactions with the oil in the membrane surface.

Although microbubbles had a positive effect on membrane cleaning, the application of microbubbles only at this step of the CIP process has no greater effect compared to the application of microbubbles in the first two steps (pre-rinse & alkali). This can be verified by observing Figure 6, in which it can be seen that there is a minimum reduction of the oil layer in the membrane after 10 minutes of microbubbling (pre-rinse step) and there is no visual difference with the membrane cleaned using water (control).

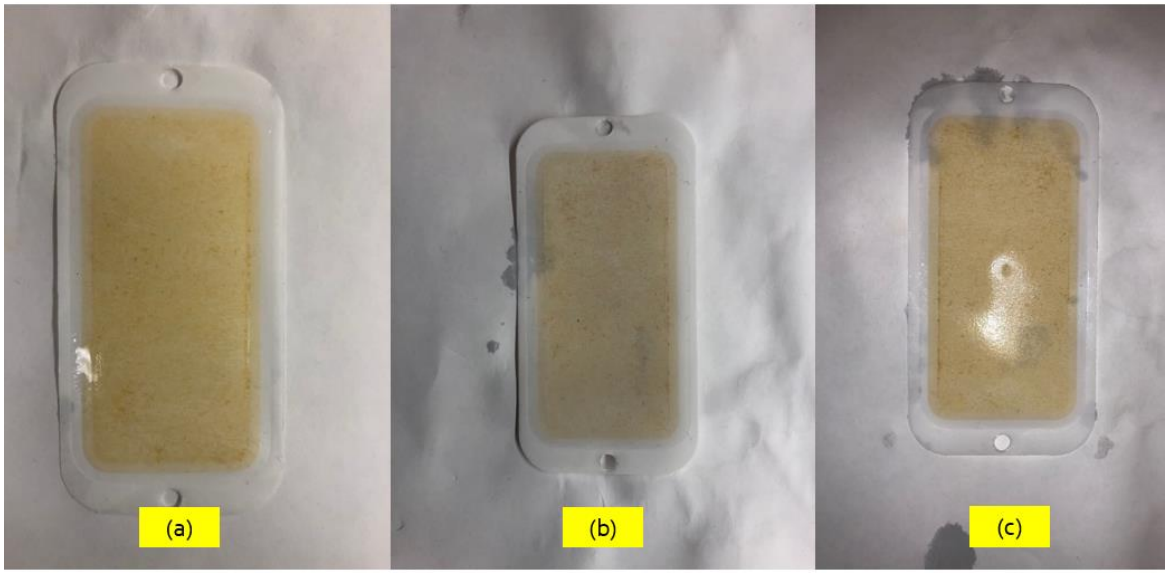

Figure 6. Photography of fouled membrane (a), after microbubbling into pre-rinse step (b), and after cleaning with water (control) at pre-rinse step (c) .

Introduction of microbubbles in the pre-rinse & alkali steps of the CIP process. The results obtained from the introduction of microbubbles during the pre-rinse & alkali steps of the CIP process are shown in Figure 7. The control treatment was the use of water in the pre-rinse step and a 0.5% Sodium Hydroxide (NaOH) solution in the alkali step of the CIP process, which is the conventional cleaning process, and the comparison treatments were the introduction of microbubbles in those same steps replacing the water and chemical detergent. In order to measure the effectiveness of the microbubbles as cleaning agents, the flux recovery was calculated after each treatment, and it was found that the cleaning process using microbubbles was more effective in fouling removal from the surface of the membrane.

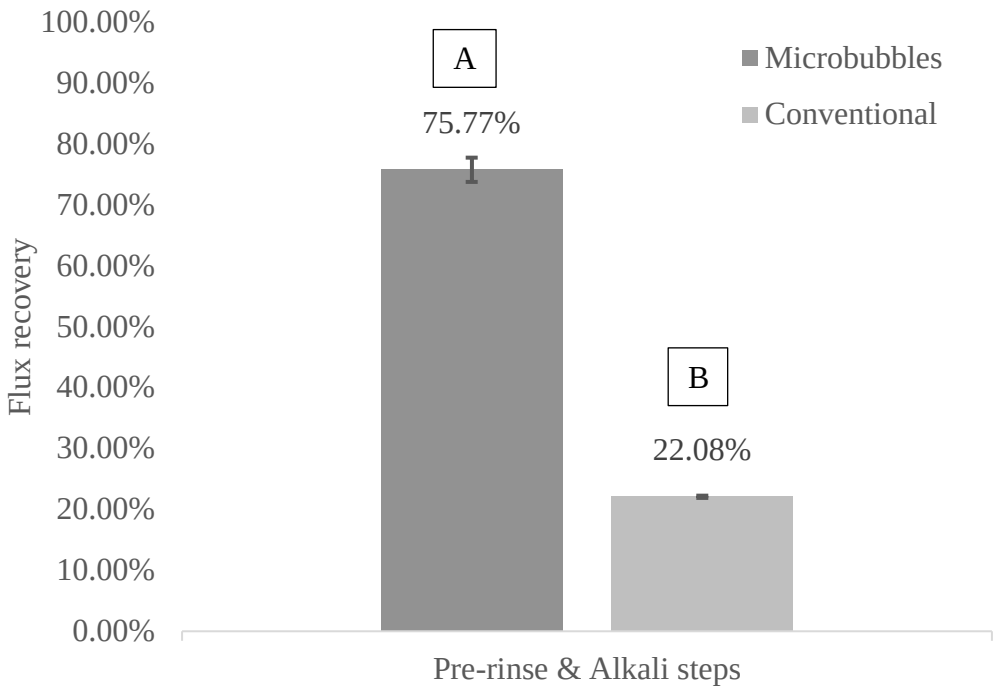

Figure 7. Cleaning efficiency of microbubbles based on percentage of membrane flux recovery after cleaning at pre-rinse & alkali steps, compared to the conventional cleaning process (2.99% C.V).
[A-B]Means with different letter are statistically different ($P < 0.05$).
%C.V. = Coefficient of variation.

As observed in Figure 7, there were significant statistical differences ($P < 0.05$) between both cleaning protocols, indicating that micorbubbles have a greater cleaning efficiency when applied in the pre-rinse & alkali steps of the CIP process respectively. The use of microbubbles as cleaning agents during the first and second steps of the CIP process restored the membrane performance to a 75% compared with the control which was only 22% of flux recovery.

These results agree with those obtained by Agarwal *et al*. (2012), they found that after one hour of microbubbling there was an 88% efficiency in the biofilm detachment from a nylon membrane surface, compared to 1 hour using a solution of sodium hypochlorite (NaOCl) (0.5%) in which only 10% was reduced of the fouling layer. It was further demonstrated that microbubbling is much more efficient than chemical cleaning in terms of fouling removal.

Also, these results coincide with the results reported by Watabe *et al*. (2016), they established that the longer the exposure of the membrane to microbubble treatment is, at a constant cross flow velocity (CFV), the fouling layer reduces considerably. In this case there were 30 minutes of microbubbling, which comprise 10 minutes of the pre-rinse step and 20 minutes of the alkali step. So, it is assumed that the cleaning effect of the microbubbles was better in this experiment because there were more exposure time of the

membrane to the microbubbles solution, compared to the first experiment which was only 10 minutes exposure.

The result of this experiment can be verified visually by observing Figure 8, which shows the fully fouled membrane (a), the membrane after applying the microbubbles (b), and the membrane after clean with water in the pre-rinse step and sodium hidroxide at alkali step (c). As observed, there is no presence of oil layer in the membrane surface when applied microbubbles (b), compared to the membrane cleaned with the control (c), which shows a remanent of oil layer in the surface.

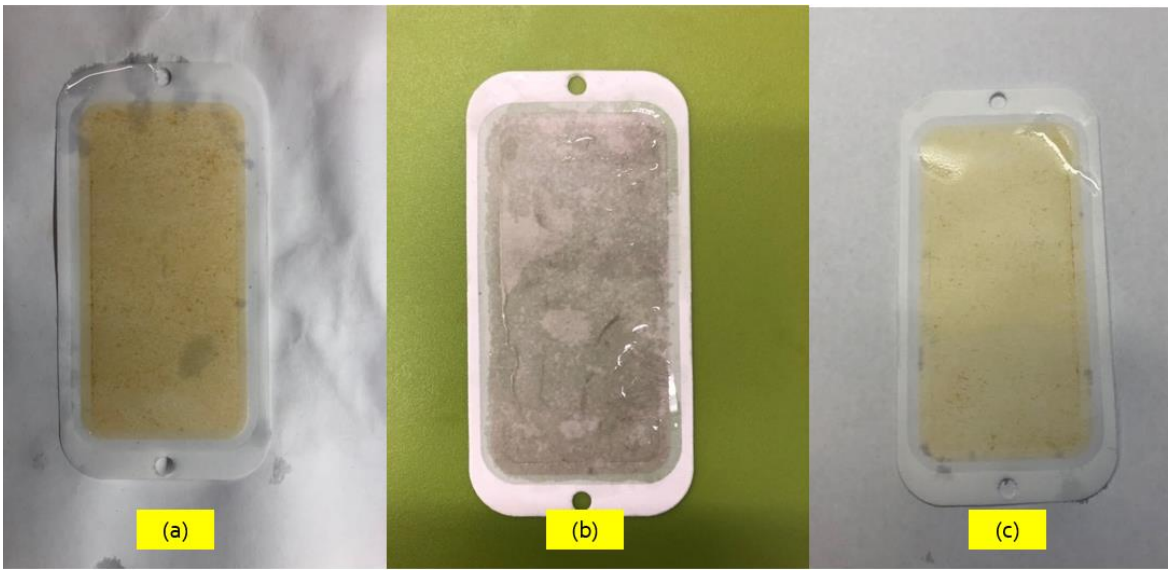

Figure 8. Photography of fouled membrane (a), after microbubbling into pre-rinse & alkali steps (b), and after cleaning with water and sodium hydroxide (control) at pre-rinse & alkali steps respectively (c).

Due to the fact that microbubbles have a high internal pressure, which increases as their diameter reduce, they can generate pressure waves when they collapse and therefore can eventually blow away the foulant from the membrane surface. According to the Young–Laplace equation, the pressure inside a 1 μm bubble is about 390 kPa (equivalent to a force of 3.9 kN cm^2) at 298 K, which is almost four times the atmospheric pressure (Takahashi *et al*. 2007). In addition, the generated pressure waves can burst nearby microbubbles, thus generating a chain reaction, which may increase the efficiency of microbubbles in removing the fouling layer (Takahashi *et al*. 2003; Agarwal *et al*. 2012). Therefore, it is assumed that the effect of microbubbles self-collapse helped to reduce the fouling layer from the membrane.

4. CONCLUSIONS

- A bench scale cross-flow microfiltration system was designed to include the introduction of microbubble solutions during the membrane cleaning process.

- The introduction of the microbubbles as cleaning agents during the pre-rinse & alkali steps in the CIP process proved to restore the membrane flux.

- The results of this study suggest that the use of microbubbles is more efficient in foulant detachment from the membrane surface, compared to the conventional membrane cleaning processes, restoring the membrane flux to a greater performance level.

5. RECOMENDATIONS

- Replace the analogue balance of the microfiltration system for an electronic analytical balance able to connect to a software, thus, the measurement of the permeate mass over time is programmable, to ovoid human error during permeate mass measurement.

- Perform a microscopic inspection of the membranes after cleaning with microbubbles to detect possible damage to the structure of the PVDF type membranes due to the effects of the cavitation phenomenon caused by the self-collapse of the microbubbles during the cleaning process.

- Evaluate the effect of the temperature and transmembrane pressure (TMP) on the process of cleaning membranes with microbubbles in water and make a correlation between both parameters.

- Perform a Life Cycle Assessment (LCA) for the Microbubbles Generator Device (MGD) in order to evaluate the environmental impact of the production of large quantities of microbubbles solution for membrane cleaning purposes.

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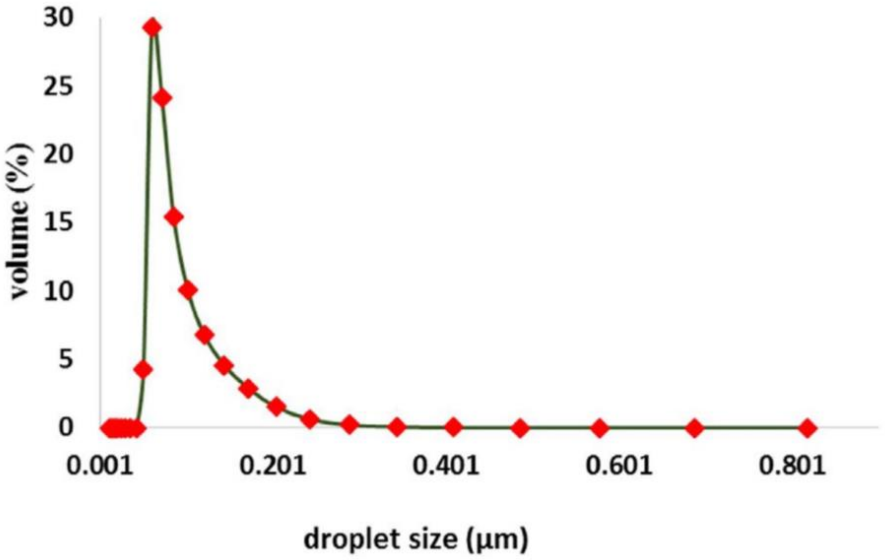
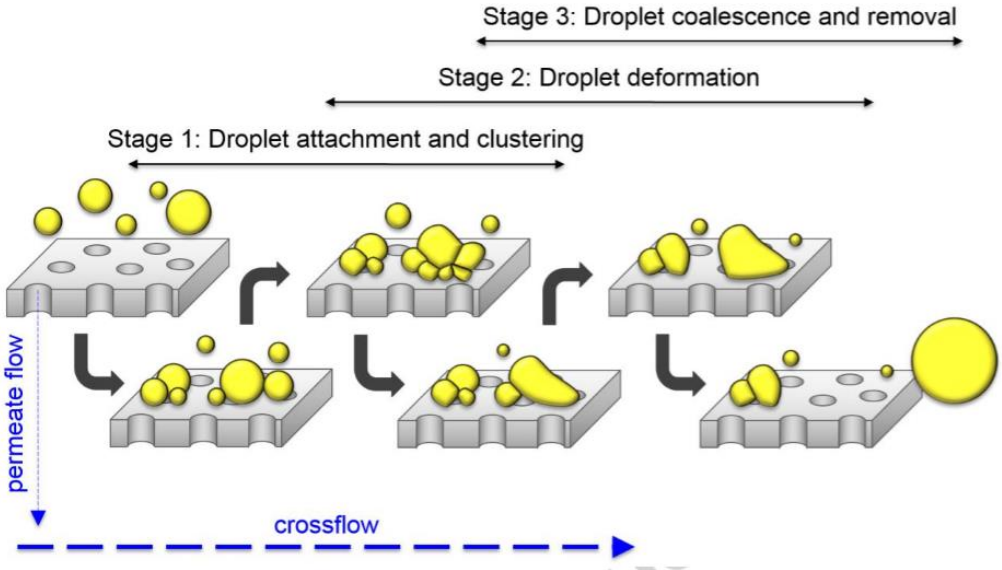
7. APPENDICES

Appendix 1. Droplet size distribution of the oil in water emulsion.



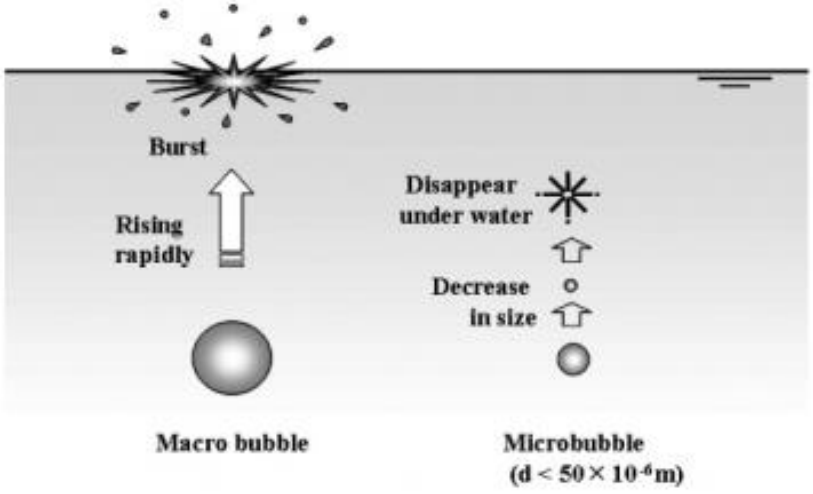
Source: (Garmsiri *et al*. 2017)

Appendix 2. Stages of membrane fouling during oily wastewater treatment.



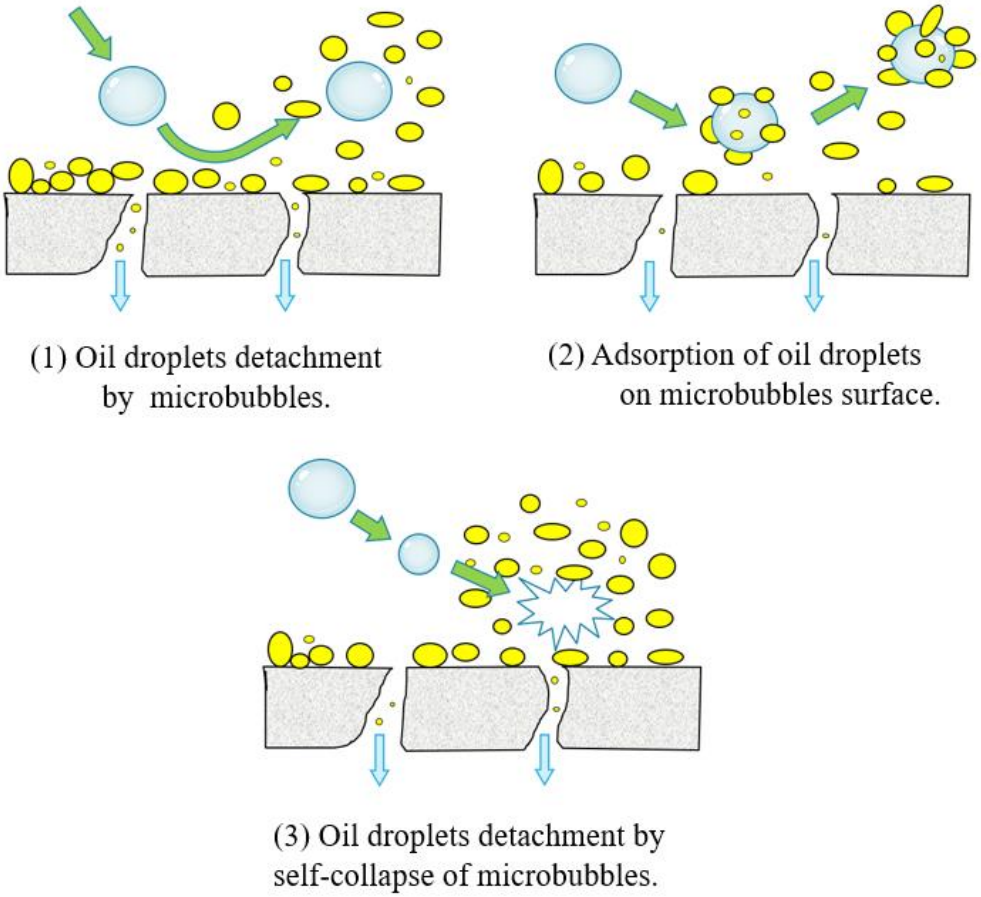
Source: (Tummons *et al*. 2016)

Appendix 3. Microbubble behavior.

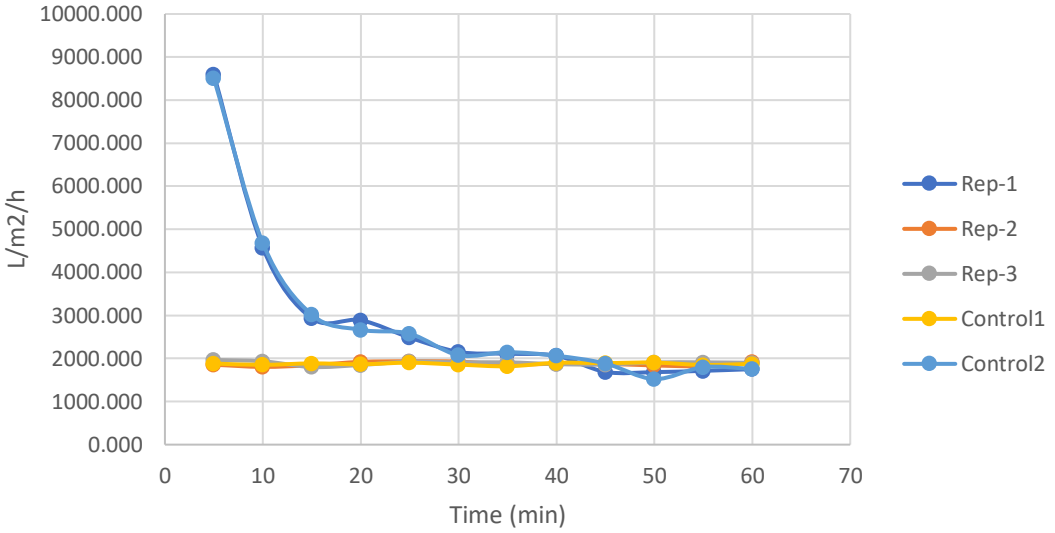


Source: Takahashi *et al*. 2007

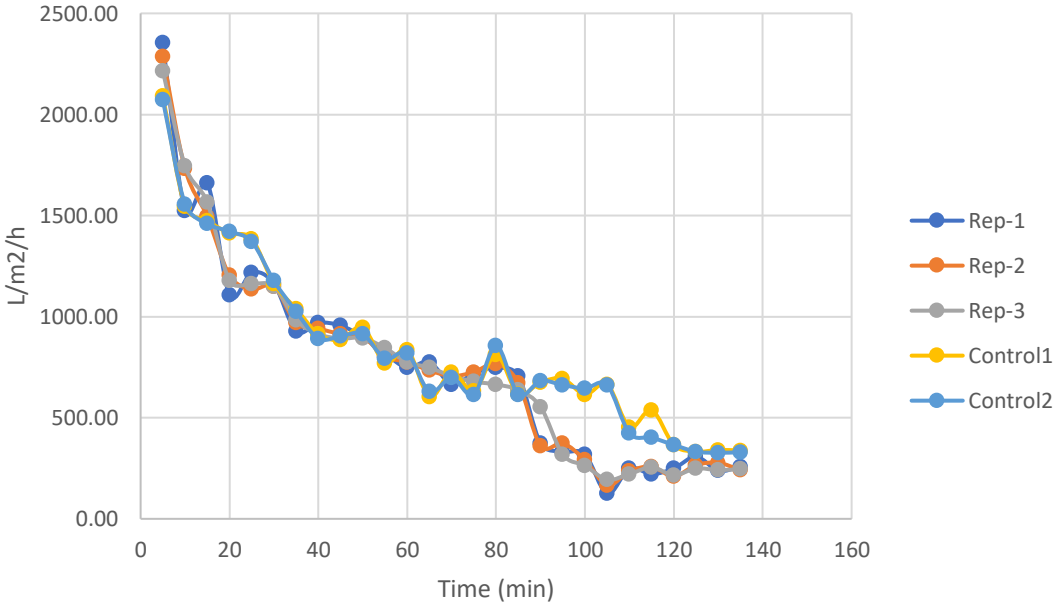
Appendix 4. Possible mechanisms by which microbubbles reduce the oil layer from the membrane.



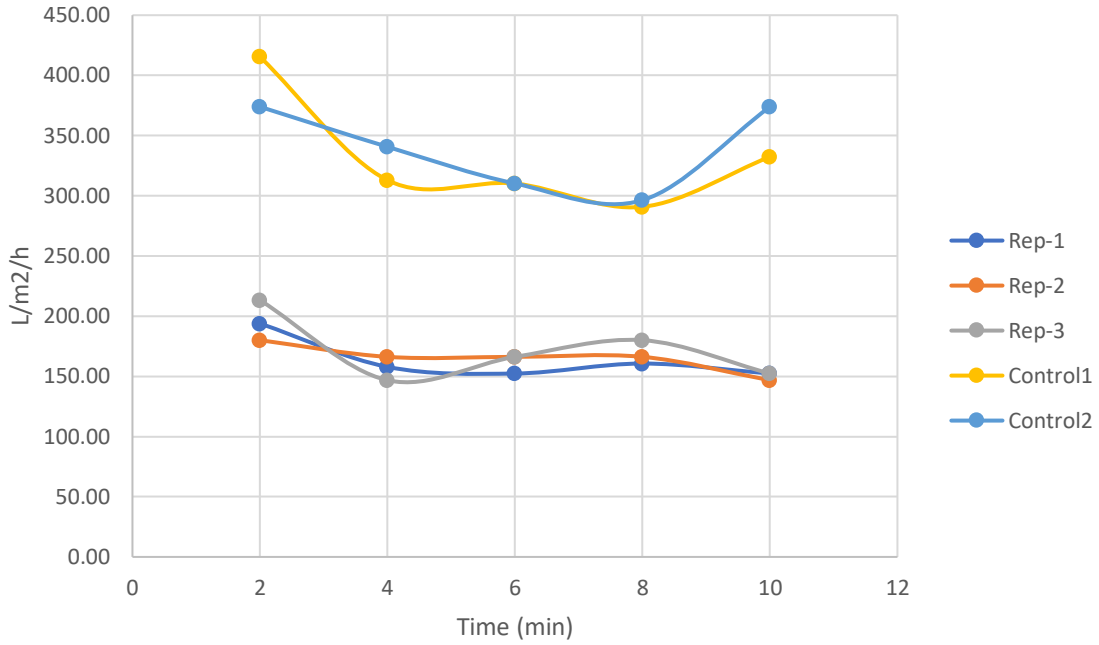
Appendix 5. Membrane flux during pre-conditioning stage for the cleaning tests at pre-rinse step.



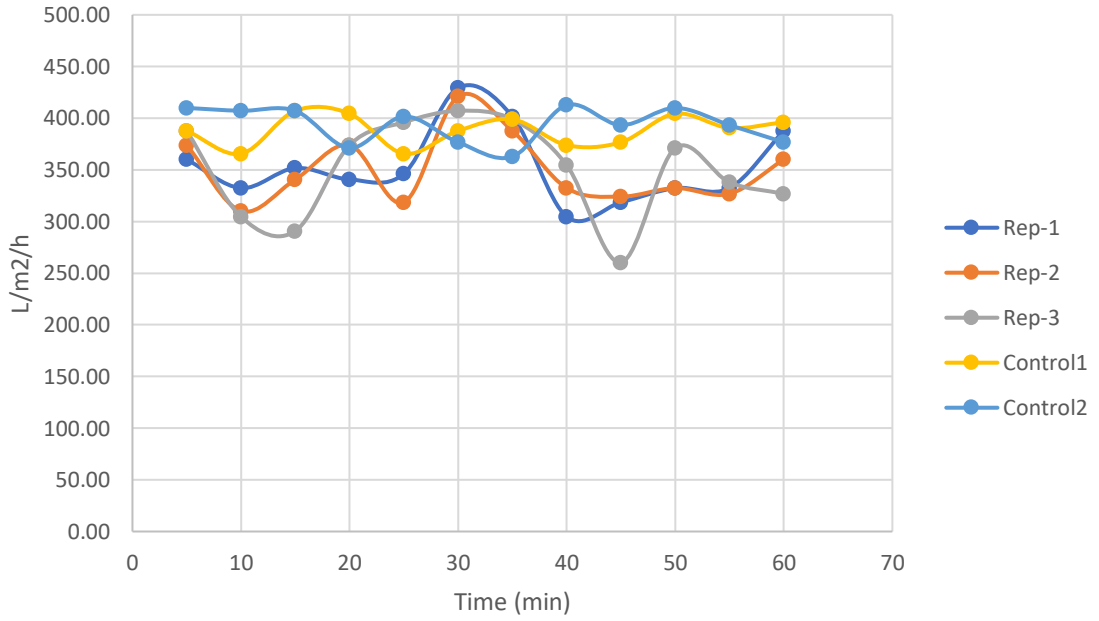
Appendix 6. Membrane flux during fouling stage for the cleaning tests at pre-rinse step.



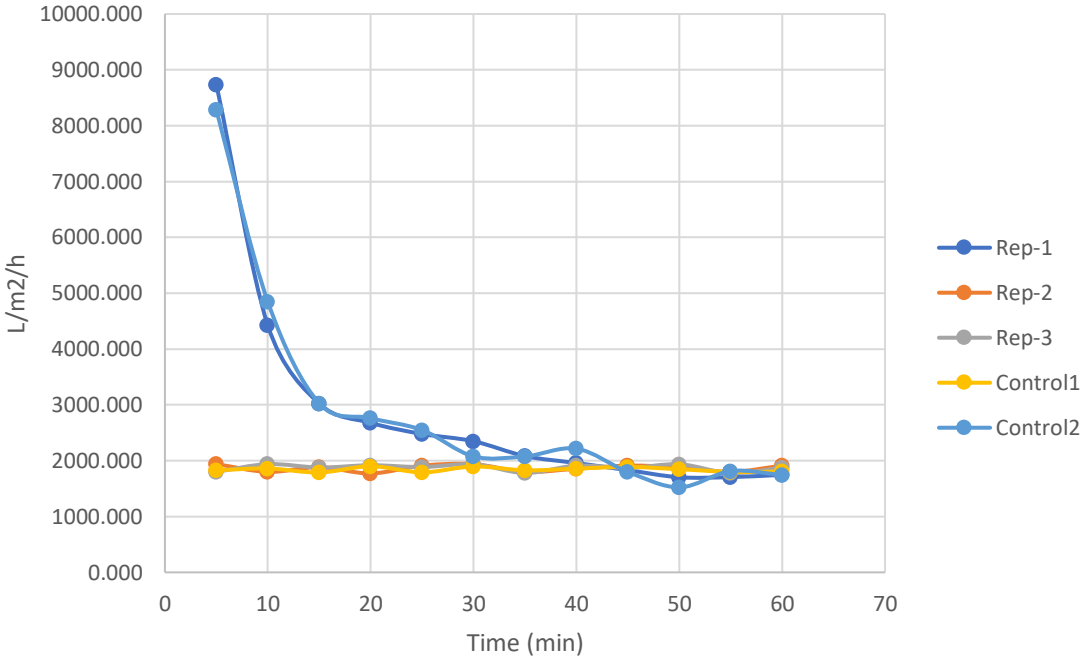
Appendix 7. Membrane flux during the cleaning treatment at the pre-rinse step.



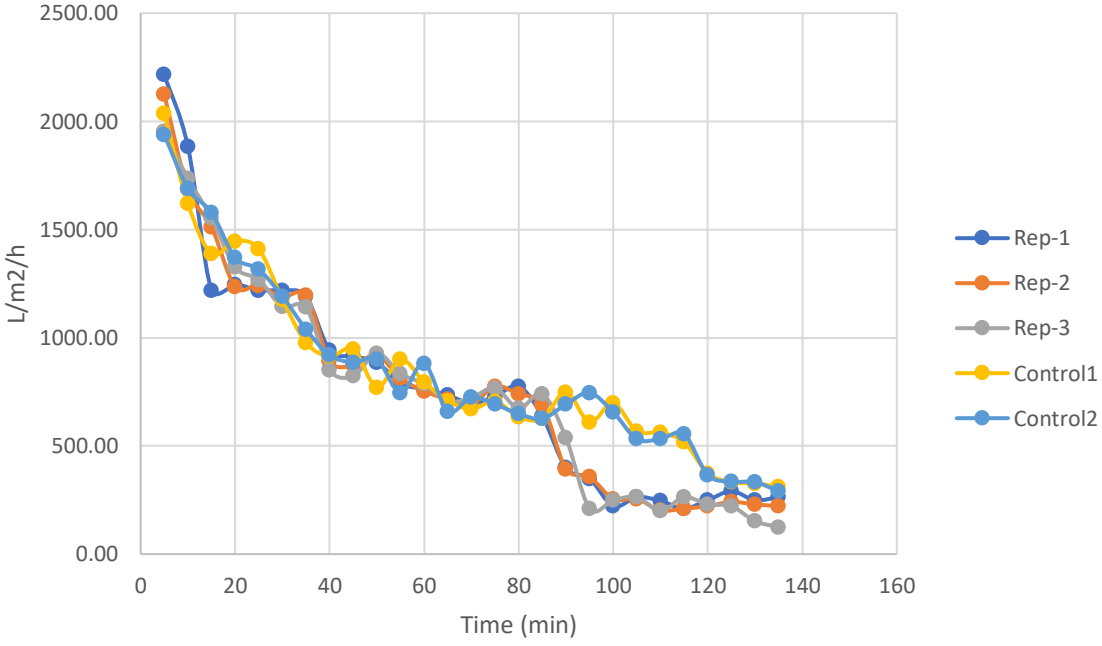
Appendix 8. Membrane flux after cleaning treatment with the cleaning protocols at the pre-rinse step.



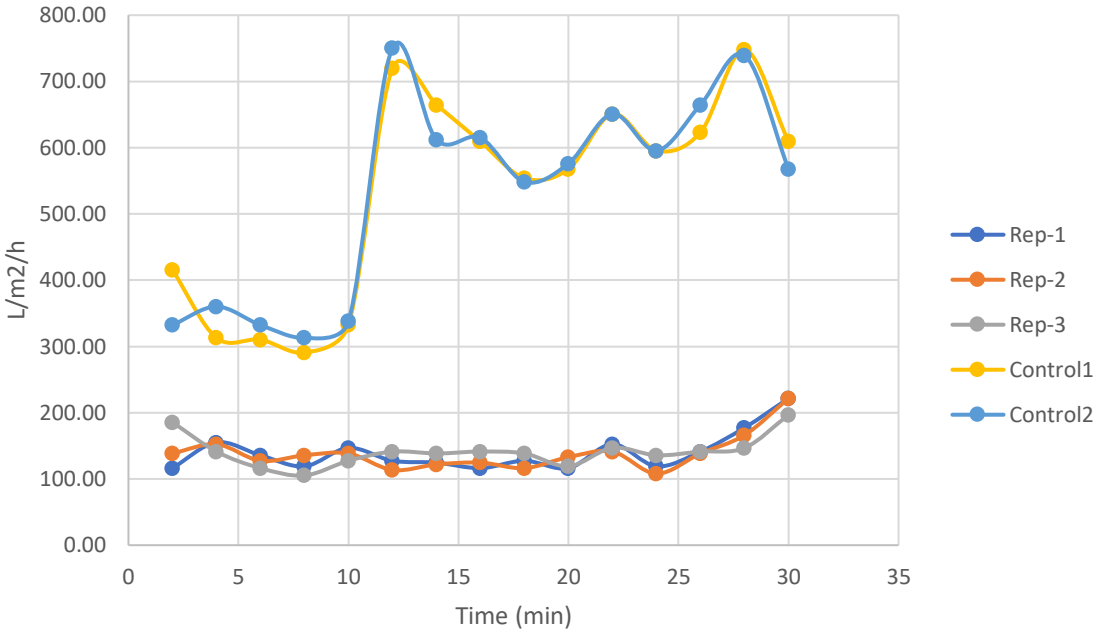
Appendix 9. Membrane flux during pre-conditioning stage for cleaning test at pre-rinse & alkali steps.



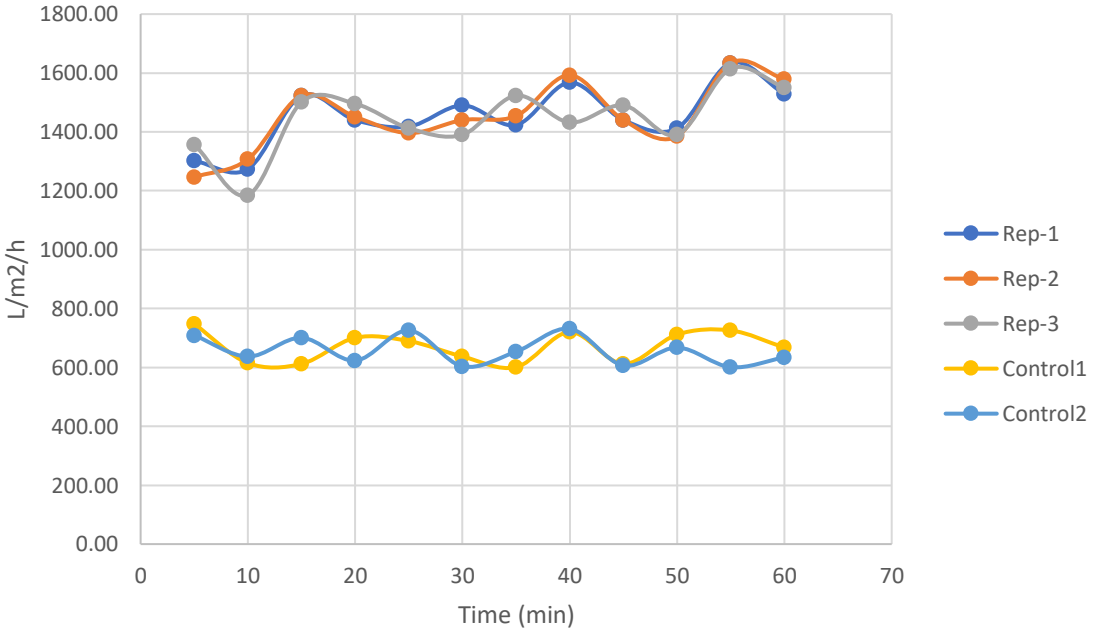
Appendix 10. Membrane flux during fouling stage for the cleaning tests at pre-rinse&alkali steps.



Appendix 11. Membrane flux during cleaning treatment at pre-rinse&alkali steps.



Appendix 12. Membrane flux after cleaning treatments with the cleaning protocols at pre-rinse&alkali steps.



Appendix 13. Filtration System User Manual.

FILTRATION SYSTEM

USER MANUAL

By
Juan Velásquez

APRIL 1, 2019

FOOD SCIENCE DEPARTMENT, PURDUE UNIVERSITY
Food Process Sustainability Laboratory

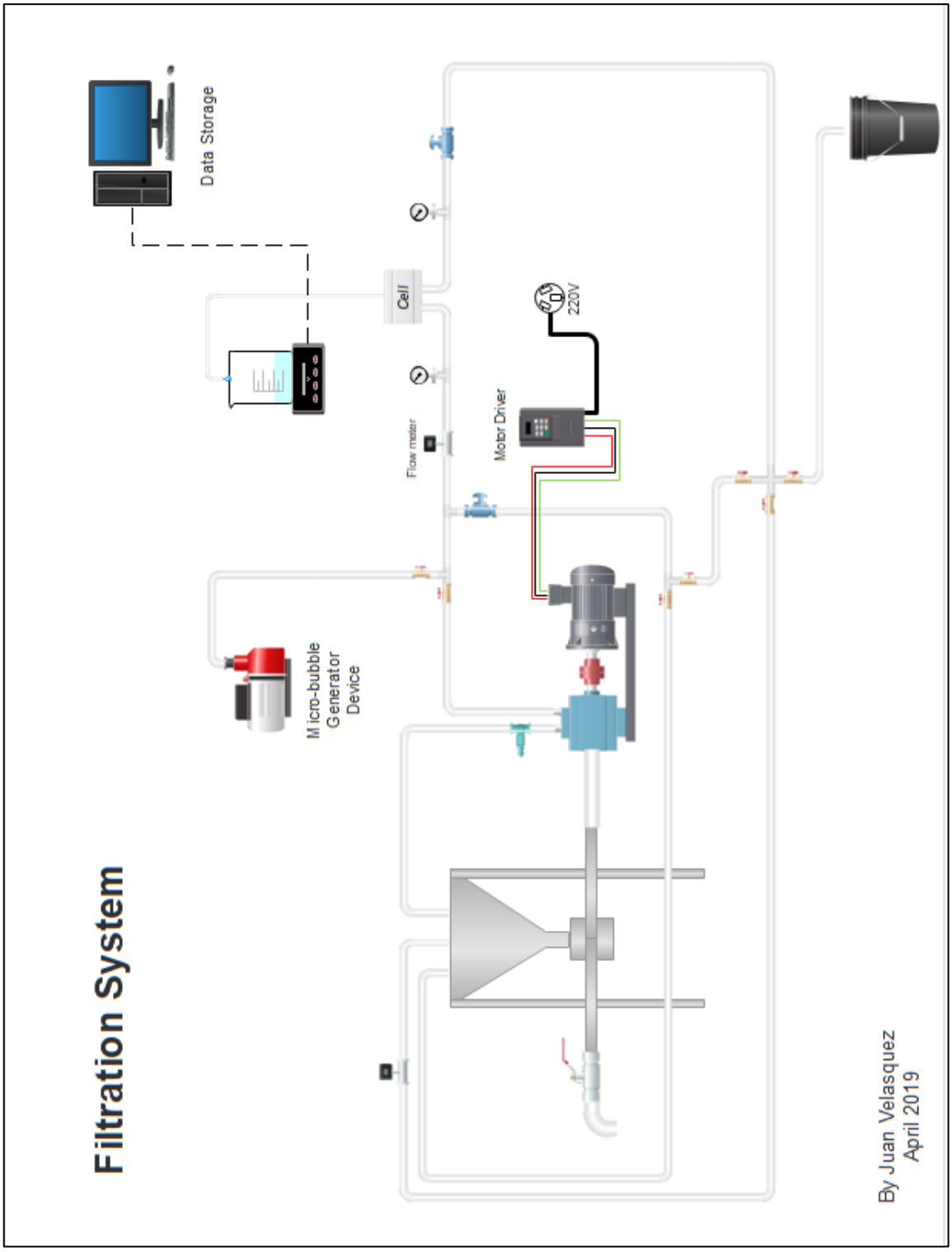
Continuation Appendix 13.

CONTENT

FIGURES

TABLES

Continuation Appendix 13.



Continuation Appendix 13.

1. INTRODUCTION

The Filtration System is a bench-scale crossflow filtration system designed for wastewater treatment under controlled conditions and adapted to simulate the Clean-In-Place (CIP) process for membrane cleaning. Also is adapted to introduce micro-bubble solutions during the CIP process in order to reduce the amount of water and chemicals used in membrane cleaning protocols. The system can be adjusted to a different test conditions depending on the type of membrane that could be microfiltration, ultrafiltration nanofiltration, and reverse osmosis.

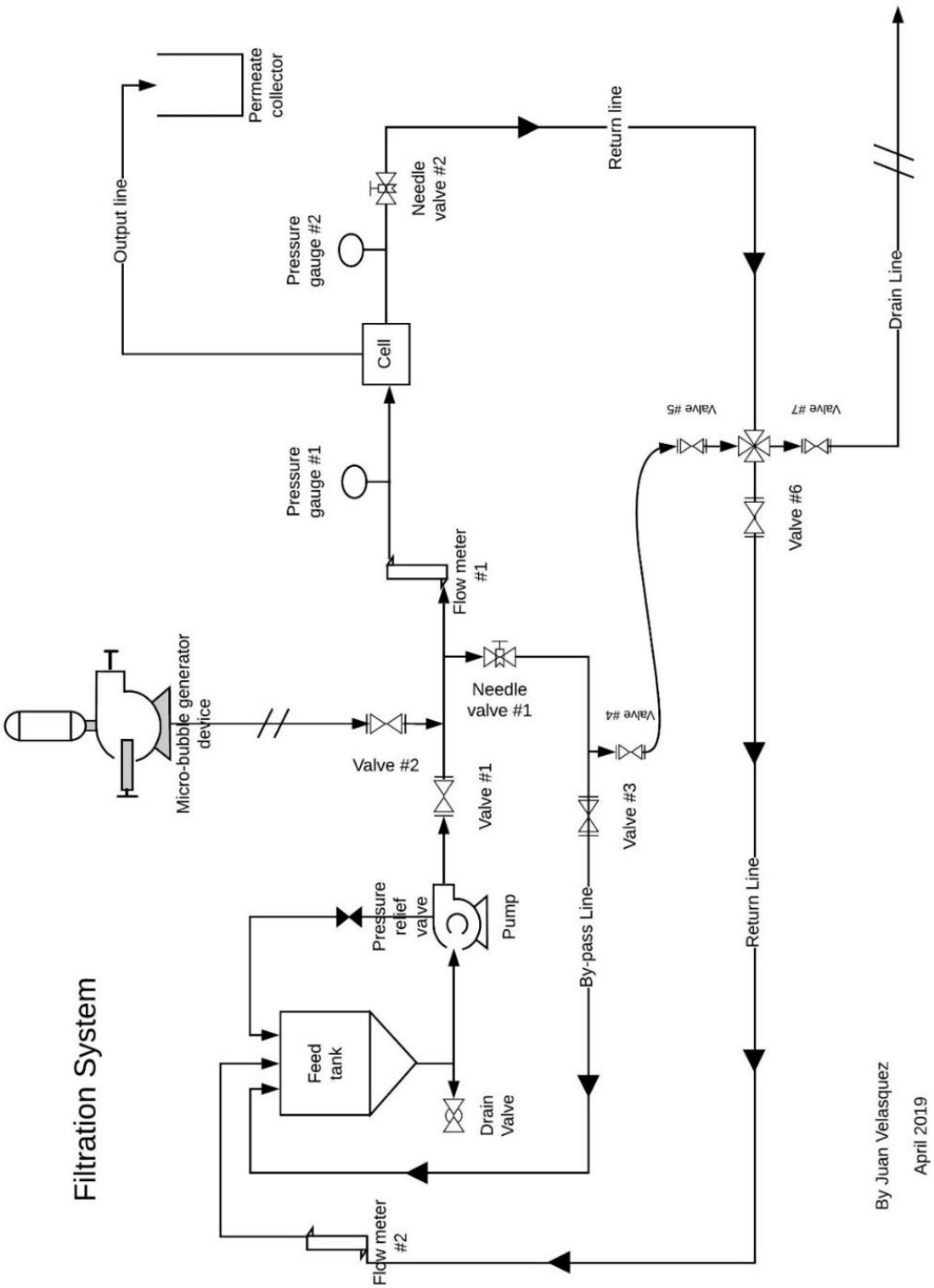
2. PARTS AND SPECIFICATIONS

Table 13. Filtration System parts and specifications.

No.	Part	Description
1	Conical feed tank	10 L max capacity, stainless steel
2	Feed pump	Poly-Diaphragm pump model DP-43-P, 1Hp, max 650 rpm, max 218 PSI, max 5 GPM @ max PSI. ¾´ inlet& ½´outlet.
3	Motor	1 Hp, 1760 RPM, 3PH, 60 Hz. Model BALD-CEM3546
4	Motor driver	AC 1PH, 220V, 50/60Hz. ATO model GK3000-2S0015G
5	Flow meter	Blue-White F-550, max 2GPM
6	Drain valve (tank)	1´ OD, stainless steel
7	Bypass control valve	3/8´ Needle valve, stainless steel
8	Concentrate pressure gauge	Max 400 PSI
9	CF042 Cell	Acrylic cell model CF042, max 400 PSI
10	Concentrate control valve & gauge assembly	3/8´ Stainless steel needle valve & pressure gauge
11	Scale for measuring permeate mass	Analytical balance for mass measure, max 10 Kg
12	permeate collector	Beaker, 3000mL max capacity
13	Pressure relief valve	3/8´, 200 PSI max pressure
14	Concentrate feed line	3/8´ high pressure rigid tubing
15	Bypass line	3/8´ low pressure flex tubing
16	Concentrate return line	3/8´ low pressure flex tubing
17	Permeate output line	1/8´ low pressure rigid tubing
18	Drain line	3/8´ low pressure flex tubing
19	Pressure relief line	3/8´ low pressure flex tubing
20	Control valves	3/8´ OD, in-line plastic valves
21	Micro-bubble generator device	Nikuni pump A3:C22 17LPM water flow rate, 1.3 NL/min air flow rate, model KTM20ND

Continuation Appendix 13.

Figure 9. Filtration System general schematic flow diagram.



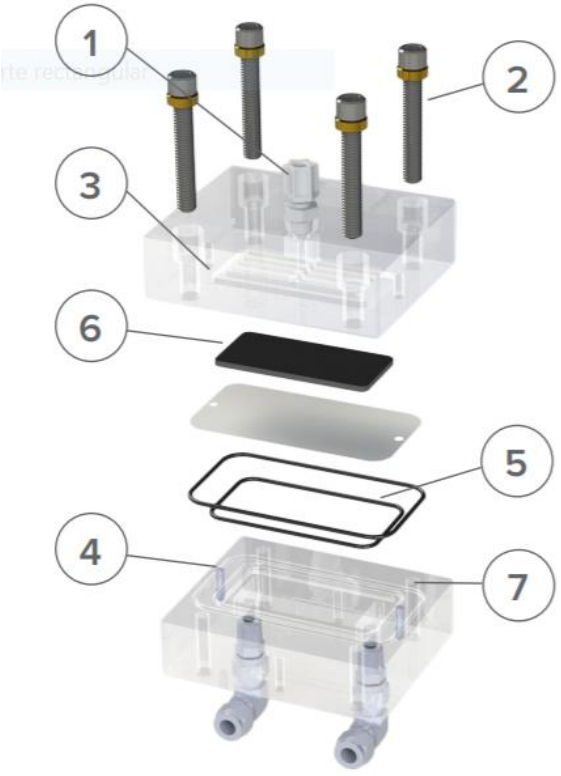
Continuation Appendix 13.

3. CF042A CROSSFLOW CELL SET UP.

Figure 10. CF042A features and technical specification.

Parameter	Description
Membrane Active Area	42 cm^2 (6.5-$inch^2$)
CF042A	Clear Cast Acrylic
Maximum Pressure	27 bar (400 psig)
Maximum Temperature	88 °C (190 °F)
Maximum Bolts Torque Setting	45 in-lbs
Maximum Fittings Torque Setting	25 in-lbs

Figure 11. CF042A Cell body parts.



No.	Part
1	Permeate outlet
2	Top screws and washers
3	Cell top
4	Alignment pins
5	O-rings
6	Sintered metal support
7	Cell bottom

Continuation Appendix 13.

For assembling the cell before a test, follow the next steps:

4. Wet the O-rings with a small amount of water or the fluid to be processed and install them into the grooves on the cell bottom.

5. Place a piece of pre-cut membrane on to the cell bottom using the two alignment pins to hold the membrane in position. The membrane should be installed with the shiny or active side down toward the cavity in the cell bottom.

   Note: The membranes do have a sided-ness. One side of the membrane should appear shinier/smoother than the other side. This side is the active layer and should be oriented towards the feed solution.

6. Place the cell top and the integrated sintered steel support onto the cell bottom. The alignment holes in the top should fit over the alignment pins in the cell bottom.

7. Insert one of the brass washers into each of the four holes at the corners of the cell top.

8. Secure the cell top by inserting and tightening one of the provided screws into each of the corner holes.

The CF042A crossflow cell is now assembled and ready to be connected to a feed pump for operation.

For more info review the CF042A Crossflow Cell manual at:
https://www.sterlitech.com/media/wysiwyg/Manual2018/Manual_CF042A_CF.pdf

4. BASIC OPERATION

For the fouling test it is necessary to set up the system as follows:

1. Close valves 2, 4, 5, and 7 & drain valve completely closed.
2. Make sure that the valves 1, 3, and 6 & needle valves #1 and #2 are completely open.
3. Fill the tank with the wastewater solution.
4. With all the valves ready, turn on the pump and check that the RPM are correct (review the VFD operation parameters in the next chapter).
5. Adjust the inlet flow rate opening/closing the needle valve #1 in the bypass line.
6. Adjust the Trans-Membrane Pressure (TMP) with opening/closing the needle valve #2 in the return line.

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7. Turn on the mass balance and make a lecture every 5 minutes since the TMP and inlet flow rate were adjusted to test conditions.
8. Once the experiment is done turn off the pump (when the permeate flux is stable and not changing, it means that the membrane is completely fouled and open all the valves to relieve any pressure build up.
9. Drain all the retentate from the feed tank opening the drain valve.
10. Turn on the pump again and let it run for 5-10 min without any solution.

Important: refer to the Filtration System general schematic diagram to review the position and the number of the valves in the system.

To replace a membrane filter:

1. Turn the feed flow pump OFF.
2. Open all the valves in the system to relieve any pressure build up.
3. Loosen the screws in the cell top.
4. Separate the cell body top from the cell bottom.
5. Remove the membrane.
6. Install the new membrane.
7. Reassemble the cell top and bottom.
8. Secure the cell top to the cell bottom using the screws.
9. Turn the feed flow pump on.

For cleaning test (CIP process), set up the system as follows.

Pre-rinse and alkali steps (using micro-bubbles).

1. Close valves 1, 3, and 6,
2. Make sure that the valves 2, 4, 5, and 7 & needle valves #1 and #2 are completely open.
3. With all the valves ready, turn on the Micro-bubble Generator Device (MBGD).
4. Wait 20-30 min (to create the MB solution) before open the outlet valve of the MBGD).
5. Connect the outlet of the MBGD to the filtration system and then open completely the outlet valve.
6. Adjust the inlet flow rate opening/closing the needle valve #1 in the bypass line.
7. Adjust the trans-membrane pressure (TMP) with opening/closing the needle valve #2 in the return line.
8. Turn on the mass balance and make a lecture every 5 minutes since the TMP and inlet flow rate were adjusted to the test conditions.
9. Once the time is done, turn off the MBGD.

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Intermediate rinse, acid, final rinse steps.

1. Close valves 2, 4, 5, and 7 & drain valve completely closed.
2. Make sure that the valves 1, 3, and 6 & needle valves #1 and #2 are completely open.
3. Fill the feed tank with the cleaning solution.
4. With all the valves ready, turn on the pump and check that the RPM are correct
5. Adjust the inlet flow rate opening/closing the needle valve #1 in the bypass line.
6. Adjust the trans-membrane pressure (TMP) with opening/closing the needle valve #2 in the return line.
7. Turn on the mass balance and make a lecture every 5 minutes since the TMP and inlet flow rate were adjusted to test conditions.
8. Recirculate the cleaning solution.
9. Once the time of each cleaning step is done turn off the pump and open completely the needle valves.
10. Drain cleaning solution from the feed tank opening the drain valve.
11. Repeat steps 3-10 in each step of the CIP process.

5. VARIABLE FREQUENCY DRIVER (VFD)

1 hp variable frequency driver, 0.75 kW, 1 phase 220V - 240V for 1/3 phase AC motor.

Table 14. VFD specifications.

Model	GK3000-1S0007
Capacity	1 hp (0.75 KW)
Rated Current	7 Amperes
Input Voltage	Single phase AC 220V±15%
Input frequency	47-63Hz
output voltage	0-rated input voltage
output frequency	V/f control: 0-3000Hz
Starting torque	1Hz/150%

Continuation Appendix 13.

Figure 12. VFD keyboard.

Figure 13. Keyboard indicator.

Item			Function description	
Display function	Digital display		Display VFD's running state parameters and setting parameters	
	State indicator	A、Hz、V	The physical units correspond to presently digital display parameters (current is ampere A, voltage is volt V, frequency is Hertz Hz) .	
		MOD	In the non-monitoring state, the indicator is on. If there is no press for one minute, the indicator is off and returns to the monitoring state.	
		ALM	The alarm indicator, indicates that the VFD is currently in an overcurrent or overvoltage state or a fault alarm state.	
		FWD	Forward indicator, indicates that the VFD outputs positive phase sequence. When the motor is connected, the motor rotates forward.	If the FWD and REV indicators are on at the same time, it indicates that the VFD is working in DC braking state.
		REV	Reverse indicator, indicates that the VFD output reverse phase sequence, when the motor is connected, the motor is reversed.	

Continuation Appendix 13.

Figure 14. Keyboard function.

Key	Item	Function description
MENU ESC	MENU/ESC key	Enter or exit programming state
▶▶	Shift/monitor	In the editing state, the modification bit of the setting data can be selected; in other states, the display state monitoring parameter can be switched
ENTER DATA	Shift/monitor	Enter the submenu or data confirmation
JOG REV	Reserve/Jog key	In the operation keyboard mode, press this key to reverse or jog according to the one-bit setting of parameter P3.46.
FWD	Forward key	In the operation keyboard mode, press this key to run the VFD in forward
STOP RESET	Stop/reset key	When the VFD is in normal running state, if the running command channel of the VFD is set to the keypad stop effective mode, press this button, the VFD will stop according to the set mode. When the VFD is in the fault state, press this button to reset the VFD and return to the normal shutdown state
●	Analog potentiometer	For frequency given; when P0.01=0, the analog potentiometer is set to frequency given
▲	UP key	Increment of data or function code (increasing the incremental speed when pressed continuously)
▼	DOWN key	Decrement of data or function code (increasing decrement speed when pressed continuously)

VFD basic parameters:

There are three basic parameters that is necessary to set up before operating the motor with the pump. P0 group (Basic Running Function Parameter), P5 group (Protection Function Parameter), P9 group (Swing Frequency Function Parameter) and PA group (Vector Control Parameter).

P0 Group - Basic Running Function Parameter

Press MENU key to P group selection interface and press ENTER key to the current parameter group P0.

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P0.00 is control mode selection, 0 means V/F control, and 1 means sensorless vector control.

Select 0 and press ENTER to confirm the selection.

P0.01 is channel selection of frequency given. There are 8 options. We choose 0 (analog potentiometer given on control panel) here. For the rest of code, please refer to the manual.

P0.02 is running frequency setting, currently 50Hz, we can press › › key to select digit position and press ▲▼ key to modify.

P0.03 is running command mode selection, 0 means use control panel key RUN, STOP/RESET, JOG to operate VFD, 1 means terminal control mode, 2 means serial port control mode. Select the default 0.

P0.04 is running direction setting. 0: Running forward allowed, 1: Running reverse allowed

P0.05 is FWD/REV dead time set, unit is s.

P0.06 is max output freq. Can be set according to own needs.

P0.07 is basic running freq. The basic running freq. is the corresponding min freq. when VFD outputs highest voltage. Generally, it is rated freq. of motor.

P0.08 is max output voltage, generally is rated voltage of motor.

P0.09 is torque boost. It can improve the low-frequency torque characteristics of the inverter and compensate for the output voltage. We choose 2.0%.

P0.10 is torque boost cut-off freq. It is related to the P0.09 we just set.

P0.11 is torque boost mode, 1 means auto boost, 0 means manual boost, select 1.

P0.12 is carrier freq., mainly affects the noise of motor and heat loss. Set P0.12 = 5.

P0.13 is Acc/Dec mode selection, 0 means linear Acc/Dec, 1 means S curve Acc/Dec.

Since the linear acceleration has just been selected, the S curve related settings of P0.14 and P0.15 are not required.

P0.16 is Acc/Dec time unit, 0 means second, 1 means minute.

P0.17 is acceleration time setting. P0.18 is deceleration time setting.

P0.19 is upper limit freq., related to P0.06 (max output freq.).

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P0.20 is lower limit freq. setting.

P0.21 is lower limit freq. running mode, 0 means run at lower limit freq., 1 means stop.

P0.22 is V/F curve setting, here choose the default 0 (constant torque curve)

The rest of the parameters of P0 Group are set according to the system default, user can modify it as needed.

P5 Group - Protection Function Parameter

P5.00 is motor overload protection mode selection, 1 means inaction (No protection feature), 0 means stop outputting. Choose 0.

P5.01 is motor overload protection coefficient, select 100%.

P5.02 is motor overvoltage stall selection, select 0.

P5.03 is overvoltage stall point, choose the default 120%.

P5.04 is auto current limit level, 120%.

P5.05 is freq. drop rate during current limit, can be set by default.

P5.06 is auto current limit mode selection, select overcurrent stop outputting 2.

P5.07 is restart setting after power failure, this we need to pay attention, choose 0 (inaction).

P5.08 is restart waiting time after power failure.

P5.09 is restart setting after power failure, select 0 (no auto reset function)

P5.10 is self-recovery interval time.

P5.11 is input missing phase protection. 0: Inaction, 1: Action

P9 Group - Swing Frequency Function Parameter

Here we pick P9.13 to introduce.

P9.13 is motor type selection.

In single-display VFD, 0 means constant torque load, 1 is suitable for fan pump type load, and motor phase type does not need to be selected.

In dual-display VFD, 0 means constant torque load, 1 is suitable for fan pump type load. And thousand's digit: 0 means ordinary 3-phase asynchronous motor (220V), 1 means 1-phase asynchronous motor/Two hot line (removing capacitor), 2 means 1-phase asynchronous motor (keeping capacitor).

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PA Group - Vector Control Parameter (motor parameter setting)

PA.00 is motor parameter auto tuning function, 0 means inaction, 1 means static auto-tuning.

PA.01 is motor rated voltage setting.

PA.02 is motor rated current setting.

PA.03 is motor rated frequency setting.

PA.04 is motor rated rotating speed setting.

PA.05 is motor poles number.

Then we look back and see how to operate the motor auto tuning function.

Set PA.00 = 1, press ENTER, and interface displays FUN0.

Then press FWD key to run VFD, VFD starts motor parameter auto tuning, and VFD keypad displays FUN1. After the auto tuning, VFD auto stops and auto tuning is completed.

Start-up Motor

Press FWD to start the motor.

Press ›› to switch to B Group to view monitoring function parameters.

b-00 is output frequency.

b-01 is set frequency.

b-02 is output voltage.

b-03 is output current.

b-04 is bus bar voltage.

B-05 is module temperature.

B-06 is motor speed.

B-07 is running time.

B-08 is Input/output terminal state.

b-09 is analog input VI.

b-10 is analog input CI.

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b-11 is external pulse width input value.

b-12 is VFD rated current.

b-13 is VFD rated voltage.

b-14 is water supply control when the set pressure of the pipeline.

b-15 is water supply control feedback pipeline pressure.

Here we should pay attention to check b-03 (the output current), can't be greater than motor rated current.

When used in 1-phase motors, selected VFD should be one level higher than motor power, such as a 0.75 kW VFD with a 400 W motor, a 1.5 kW VFD with a 0.75 kW motor.

Basic speed regulation function demo

1. Speed regulation through panel analog potentiometer.

Set frequency given channel P0.01 = 0.

After pressing FWD, the freq. adjustment (speed regulation) can be performed by the knob on the panel. The upper limit freq. is determined by P0.19.

2. Speed regulation through digital given

Set P0.01 = 2.

Set P0.02 running frequency.

After pressing FWD, VFD running at the normal operating freq. set by P0.02.

For more info check the GK3000 SINGLE PHASE VFD User Manual

For a better understanding of programming the VFD check this video:

https://www.youtube.com/watch?v=D8zavX-9vVw

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6. FOULING PROTOCOL

For the membrane fouling protocol, it is necessary to follow the next steps as shown:

Membrane Preconditioning

When the membrane is fresh, it is necessary to preconditioning it using RO water (deionized water) as follows:

1. Install the membrane in the cell. (review the CF042A crossflow cell set up).
2. Set up the system and open the valves the same as the fouling test (review the fouling test basic operation of the system)
3. Fill the tank with max 10 L of RO water.
4. Run the pump and set the conditions of the fouling test (TMP and inlet flow rate).
5. Let it run until the permeate flux become stable (review the membrane features in table 3).
6. Finally, stop the pump and drain the water.

Oil-in-water emulsion (model oily wastewater) preparation

1. Mixing 10 g of vegetable oil, 1,000 g of distillate water and 1 g of Tween 20® (surfactant) at 10,000 rpm using a laboratory homogenizer for 50-60 min.

2. Dilute the mixture by adding 9000 g of distillated water for the final oil/water concentration of 0.1 wt%.

3. Mix @10,000 rpm for 5 more minutes.

7. CLEANING PROTOCOL

For membrane cleaning, a CIP process is necessary to be conducted as follows:

Membrane cleaning protocol after pre-rinsing

1. Cleaning chemicals:
   a. 0.5−1.0 wt% **NaOH** (pH 7−11.5)
   b. 0.1−0.2 wt% **HCl** (pH 2−7)

2. Operating flow pressure: 15−75 psi

3. Temperature: 35−50 ºC

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4. Cleaning time:
   a) 10−20 min of pre-rinse
   b) 10−30 min of alkali
   c) 10−20 min of intermediate rinse
   d) 10−30 min of acid
   e) 10−20 min of final rinse

5. Circulation rate
   a) 1 to 7.5 LPM

Note: every clean solution for each CIP step must be calculated in base of 10L (max capacity of the tank).

Important: to adjust the water for the clean solution to a specific temperature a water-heater must be used and a thermometer to set a specific temperature.

8. MEMBRANE FEATURES

The system is designed to conduct experiments using any types of membranes. microfiltration, ultrafiltration, nanofiltration and reverse osmosis membranes could be used perfectly. An example of a microfiltration membrane is provided in the chat below.

Type of membrane	Model	Feed	Applications	pH range @ 25 ºC	Flux (GFD)/psi	Pore size/ MWCO	Polymer
MF	V0.1	Industrial /Dairy	Fat/microbial Removal, Protein Fractionation	1−11	237−254/20	0.1 µm	PVDF

Table 15. Microfiltration membrane features & specifications.

9. MAINTENANCE

For the maintenance of the parts of the filtration system, refer to the specific manual of each part of the system. Pay especial attention to the pump, especially in the oil change.