

TITLE PAGE AND ABSTRACT

Use of Pressurized Ozone and Dissolved Air Flotation with Reverse Osmosis Membrane Filtration for Industrial Process Water Treatment at a Forest Products Facility

Final Project Report

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Abstract:

Pactiv Corporation, along with its key project partners NYSERDA, The Energy Office Of Michigan, Cellulose Products and Services LLC, Western Michigan University's Department of Paper and Printing Science and Engineering, have successfully completed a grant funded project, titled "The Use Of Pressurized Ozone and Compressed Air Flotation with Membrane Filtration for Industrial Process Water Treatment at a Forest Products Facility". The project was funded in part by the US Department of Energy's State Technology Advancement Collaborative (STAC) program (administered by the National Association of State Energy Officials – NASEO) and New York State Energy Research and Development Authority (NYSERDA) funding as well as Pactiv Corporate funding. The project objective was to design, install and operationally demonstrate a full scale new technology system that used pressurized ozone along with clarification and filtration to effectively remove total dissolved solids (TDS) from mill process water, compile the data and results for evaluation and dissemination to interested water intensive industries. Pactiv's Plattsburgh, New York 40 ton per day molded fiber mill, acting as the host site, has successfully completed an installation and evaluation of the new technology application system for cleaning dirty mill process water and producing both clean process water and a fresh water substitute for mill use. Each day, roughly 10% of the mill's process water is discharged and replaced, the new makeup water entering at city water supply temperature in the range of 55 degrees F and must be warmed to mill operating temperatures (representing approximately a 50 degree F increase). Control of TDS is a crucial step for reducing (or eliminating entirely) the daily purge.

The new technology system actually installed and evaluated uses pressurized ozone injection in conjunction with dissolved air flotation (DAF) clarification and reverse osmosis (RO) filtration to convert total dissolved solid (TDS) contaminants in the mill's process water into total suspended solid (TSS), allowing for easier removal of the contaminants as TSS to clean the process water. The new technology application allowed for the capability to clean and reuse the mill process water internally, and also was found to have the capability to make a fresh water substitute out of mill process water, suitable for replacing purchased fresh water. The technology capability has the potential to provide significant energy and production cost saving and environmental benefits that have wide ranging application in the Pulp and Paper Industry and other water intensive industries worldwide. This technology allows for the reduction of effluent discharged from the mill, or potentially has the ability to run the mill's process water in a closed loop fashion with zero effluent discharge. Water conservation will yield energy savings at the mill, at the city water supply plant, and at the city wastewater treatment plant.

Project results showed that TDS removal during pilot studies using the pressurized ozone and Krofta DAF component of the system was 28% to 35%. Pilot studies also showed that the used of the pressurized ozone and Krofta DAF could protect a spiral wound RO membrane filtration system so that the clean process water could be further processed and cleaned. The actual full scale system installed at the Plattsburgh mill with pressurized ozone, Krofta DAF clarification and spiral wound RO membrane filtration resulted in the full scale capability to remove almost 100% of the TSS, TDS and mineral contaminants, and produce a fresh water substitute, while allowing cost effective production efficiencies and flow rates from the water cleaning system. The system was estimated to produce process water and fresh water substitute cheaper than available process water treatment and purchased fresh water costs, resulting in savings of between \$67,000 and \$99,000 per year for the mill.

The pressurize ozone, Krofta DAF and RO membrane filtration technology application was shown to hold great promise for Pulp and Paper Industry and other water intensive industries to convert dirty mill process water TDS to TSS for more efficient removal and for the production of clean process water and fresh water substitute.

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NARRATIVE

The Pactiv Corporation mill site in Plattsburgh, New York, acting as the host site, has successfully completed an installation and evaluation of a new technology application system for cleaning dirty mill process water. The grant funded project, titled “The Use Of Pressurized Ozone And Compressed Air Flotation With Membrane Filtration For Industrial Process Water Treatment At A Forest Products Facility”, was funded in part by the US Department of Energy’s State Technology Advancement Collaborative (STAC) program (administered by the National Association of State Energy Officials – NASEO) and New York State Energy Research And Development Authority (NYSERDA) funding as well as Pactiv Corporate funding.

Pactiv Corporation’s Plattsburgh mill is a 40 ton per day molded fiber mill that like many Pulp and Paper Industry mills uses significant quantities of water in their manufacturing process. The buildup of total dissolved solids (TDS) in industrial mill process water is the main reason mills “purge” their dirty process water, this so as to not detrimentally affect mill production and quality (otherwise, build-up of salts can cause crusting and clogging of spray nozzles, etc). The Plattsburgh mill thus needs to continuously purge dirty process water as effluent and replace this with fresh water for their production and operations. Each day, roughly 10% of the mill’s process water is discharged and replaced, the new makeup water entering at city water supply temperature in the range of 55 degrees F and must be warmed to mill operating temperatures (representing approximately a 50 degree F increase). Control of TDS is a crucial step for reducing (or eliminating entirely) the daily purge.

The new technology system actually installed and evaluated uses pressurized ozone injection in conjunction with dissolved air flotation (DAF) clarification and reverse osmosis (RO) filtration to convert total dissolved solid (TDS) contaminants in the mill’s process water into total suspended solid (TSS), allowing for easier removal of the contaminants as TSS to clean the process water. The new technology application allowed for the capability to clean and reuse the mill process water internally, and also was found to have the capability to make a fresh water substitute out of mill process water, suitable for replacing purchased fresh water.

This technology allows for the reduction of effluent discharged from the mill, or potentially has the ability to run the mill’s process water in a closed loop fashion with zero effluent discharge. The technology capability has the potential to provide significant energy and production cost saving and environmental benefits that have wide ranging application in the Pulp and Paper Industry and other water intensive industries worldwide. Water conservation will yield energy savings at the mill, at the city water supply plant, and at the city wastewater treatment plant.

ORIGINAL PROJECT GOALS AND VARIANCE

The project objective was to design, install and operationally demonstrate a full scale new technology system that used pressurized ozone along with clarification and filtration to effectively remove TDS from mill process water, compile the data and results for evaluation and dissemination to interested water intensive industries. To achieve that objective, the following specific goals were identified:

- Perform necessary research and development, including pilot trial work
- Design and engineer a pressurized ozone, clarification and membrane filtration system
- Procure construct and install the system at the Pactiv Plattsburgh mill
- Start up and operate the system at the Plattsburgh mill using mill process water
- Compile and analyze operational data, document and report on the results
- Provide for technology transfer and outreach to industry and interested parties

All of the above technical and performance goals have been accomplished. The project work included significant research and development work alongside of pilot trial work at Western Michigan University's Paper Industry Pilot Plant that resulted in design and value engineering improvements to the original system. These improvements exceeded the original project concept's capabilities to just clean process water for reuse, whereby not only the system could provide clean process water but even a fresh water substitute. The improved system design was installed in the Plattsburgh mill, started up and run operationally, with data compiled and analyzed, and the initial results being positive.

The project was funded in part by the Department of Energy STAC program, which provided for \$380,750 in grant funding, with NYSERDA providing \$214,422, with Pactiv Corporation providing the remainder of the project funding total. The total project actual cost \$920,173 was slightly higher than the original \$848,132 estimated, however the grant funded portions were in line with the original budget. Table 1 provides that project's grant funding budget and use had minor internal variance but in total had no variance.

Table 1: Grant Funding Budget and Use

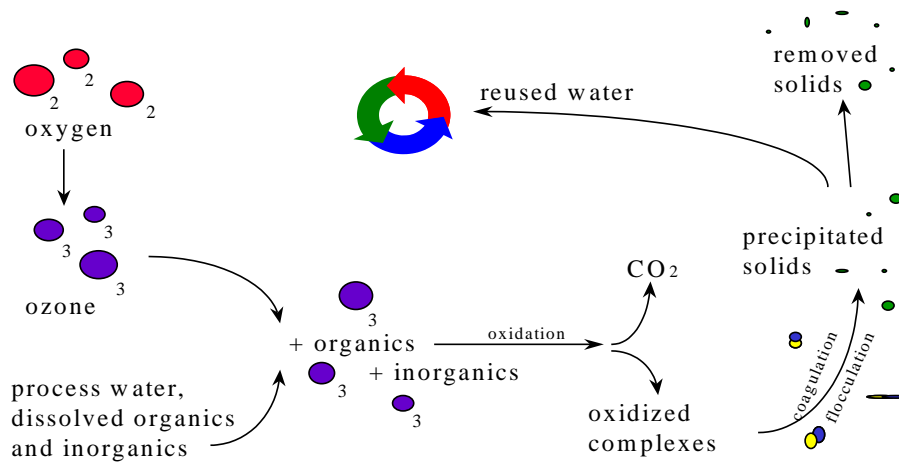
Project Cost Item	NYSERDA DOE-STAC Budget Funds	Grant Funds Used Project Start Through June 30 2007	Percent Grant Funded Budget Remaining
Pactiv Labor	\$0	\$0	0%
Pactiv Travel	\$0	\$0	0%
Contract Services	\$184,450	\$190,042	-3%
Equipment	\$340,000	\$342,069	-1%
Supp/Mat	\$70,000	\$63,060	10%
Pactiv Misc.	\$722	\$0	100%
Total	\$595,172	\$595,172	0.0%

The project partners and key vendors that participated in the project are listed and provided in Appendix A.

PROJECT BACKGROUND AND TECHNOLOGY BASIS

The use of ozone for water treatment has been available for decades. Ozone, consisting of three oxygen atoms, is a highly reactive molecule. Ozone is a powerful oxidizing agent and can react effectively to provide oxidized complexes to the TDS organics in the process water typically forming carboxyl and hydroxyl groups. These charged organics can then agglomerate and flocculate to form TSS particles. Figure 1 provides a schematic of how the ozone based technology application works.

Figure 2: Ozone Injection Based Technology Schematic



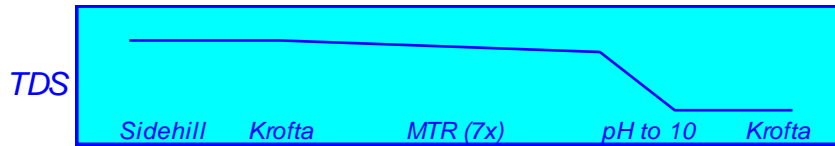
The ozone work for TDS/TSS removal described above involves four basic steps:

- 1. Removal of Large Fiber and Suspended Solids.** The initial step involves for the removal of cellulose fiber and large suspended solids in the process water. In this step, the process water can be provided from a low suspended solids operation in the mill were solids are removed by using coarse filtration or screening or fiber based flotation. The process water may come from a mill operation such as a saveall, sidehill screen, filtration device, or clarifier. At the end of clarification, the water appears less cloudy or dirty, but it still contains significant quantities of organic and inorganic dissolved solids. Removing TSS through easy mechanical screening techniques can recover wood fiber for use in making the products, and minimizes the demand for more-expensive ozone in subsequent treatment steps.
- 2. Oxidation of Dissolved Solids through Ozone Injection.** Ozone is injected into the clarified process water. Once in the water, the ozone oxidizes some of dissolved solids in the water. Some organic dissolved solids are converted into carbon dioxide. Other organic and inorganic solids are modified into complexes so that they more readily coagulate or precipitated in the following steps.
- 3. pH Swing-Induced Coagulation/Precipitation of Oxidized Solids.** The pH of the ozonated water is then changed in order to convert the oxidized complex dissolved solids into coagulated and precipitated suspended solids, or convert TDS to TSS, here through process chemistry changes including the addition of acid or base.
- 4. Dissolved Air Flotation (DAF) Clarification and Removal of Precipitated Solids.** The newly coagulated and precipitated TSS may be removed in the same manner as the suspended solids in the first step preferably by using a DAF where the dissolved air along with appropriate chemistry and polymer addition is used to float the precipitated solids to the surface where they are then skimmed from the surface. The floating scum thus harvested consists of ingredients that cannot be re-used in making the product, and therefore must be disposed of.

Past work has been done with ozone and process water based on the above sequence at Western Michigan University's Paper Industry Pilot Plant that this project's new technology application wanted to improve upon. The past work showed that ozone treated process water can result in as much as 42% removal of TDS by

conversion to and then removal as TSS, this when the TSS removal included steps such as pH induced clarification, which were performed on the TDS converted to TSS post ozone injection. Table 2 provides a graph example of the results of previous ozone based pilot work performed at WMU, using a pH induced DAF clarification step for the TDS/TSS removal operation.

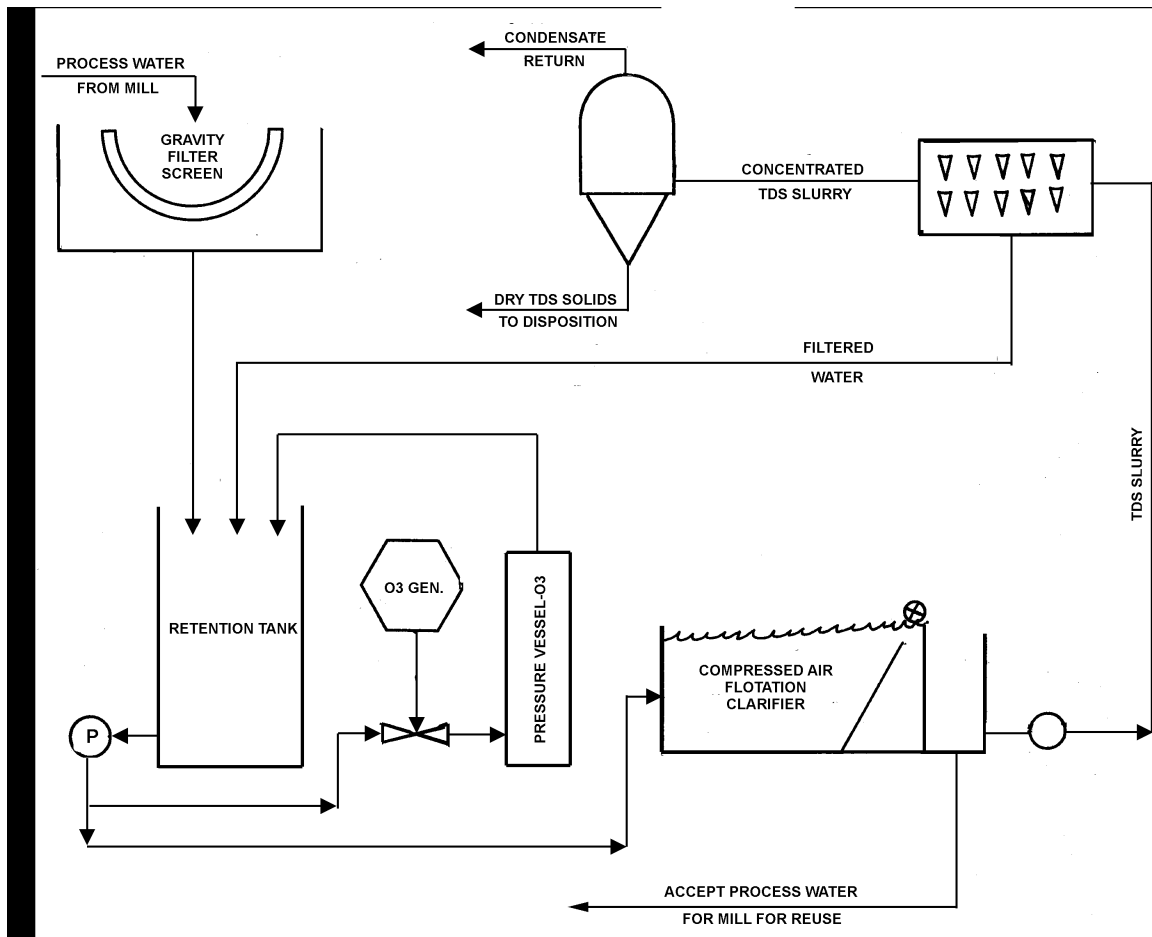
Table 2: WMU Ozone Pilot Trial Data



Source: WMU – This ozone pilot trial reduced TDS by 42%.

Many Pulp and Paper Industry mills and other water intensive industrial mills use DAF clarifiers and Compressed Air Flotation (CAF) clarifiers to remove TSS from their mill process water. Few if any mills use pressurized ozone tied to a DAF/CAF clarifier to remove TDS as TSS. One problem with the use of pressurized ozone and DAF/CAF clarification to remove TDS as TSS was that the TSS was removed in a low solids form, creating material disposition and handling problems when disposing of liquid slurry. However, steps can be taken to dewater and thicken this waste stream, which serves to improve handleability as well as reduce the volume and weight to be disposed of. The new technology application thus originally sought to include a tubular ultrafiltration membrane filtration step to take the DAF waste and recover as much liquid as was possible, feeding it back in front of the pressurized ozone step, and then allowing for the solids to be cost effectively thickened and dried. Figure 2 provides the original process flow diagram showing the pressurized ozone unit operations in front of the mill’s CAF clarifier, with the tubular Ultrafiltration membrane unit taking the CAF waste stream containing the removed TDS.

Figure 2: New Technology Application – Original Process Flow Diagram and Concept



The original process flow diagram and system concept was shown to have some significant limitations and operational problems that the eventual research and development, pilot trial work and engineering work both exposed and resolved. Some of the issues resolved were as follows:

- The placement of the pressurized ozone unit operations after the gravity screen unit operation and in front of the CAF clarifier meant a significant amount of cellulose fiber was still present in the process water, and the ozone injected into the process water had as much opportunity to react with the cellulose fiber as it did reacting with the TDS. This reduced the effectiveness of the available ozone and wasted otherwise recoverable valuable fiber.
- While the tubular ultrafiltration membrane filtration unit was very forgiving per the amount of TSS and could run without plugging its membranes, it had a low permeate or accept stream, between 10% to 30%, and a high reject stream, between 70% and 90%, which caused flow consideration problems per process water recirculation volume. This led to the investigation into spiral wound membrane technology for ultrafiltration, nanofiltration and reverse osmosis filtration, which became a preferred option flow wise. Spiral wound membrane technology has more membrane surface area and the permeate or accept stream of 70% to 90% was deemed far better flow wise for this application if the membranes could be protected from plugging.
- The mill needs to reclaim as much reusable cellulose fiber as possible from the CAF clarification for raw material needs and cost effective molded fiber production. Since the design called for the pressurized ozone step to precede the CAF unit operation, any TDS that converts to TSS in the CAF would mix with the recovered cellulose fiber and thus would simply re-dissolve in the process water when that recovered fiber was re-used in the mill.

The preliminary technical and pilot work addressed these and other issues and provided for improvements to final new technology application design, later detailed in Figure 4. Justifications for this improved design, along with the actual process flow diagram agreed to as the best design to pursue for installation purposes, are provided in the next two sections.

WMU PILOT TRIAL EVALUATION

Western Michigan University's Paper Industry Pilot plant was used to perform pressurized ozone, DAF clarification using their Krofta DAF, and also prepared process water for further membrane filtration pilot trial work. This pilot trial work was done on tanker truck size quantities of approximately 4,000 gallons from Plattsburgh's mill process water, shipped in to the pilot plant. The tanker truck quantities of process water was used in various trial run programs and evaluations using WMU's pilot plant pressurized ozone systems and Krofta DAF clarification capability available at the WMU facility along with additional pilot trial pressurized ozone equipment provided by ESTR. This equipment allowed for a simulation of any proposed system design the Pactiv Plattsburgh mill might consider. The WMU pilot plant trial work was done to evaluate the following basic process parameters to assist in system design improvements:

- Determine and evaluate the Plattsburgh mill process water responsiveness to the pressurized ozone injection reactions, the effect of the pressurized ozone technology per turning the mills process water's TDS into TSS, to assist in understanding the mill's process requirements for parameters such as pH, temperature, etc., assist in system design requirements, and assist in equipment sizing of the unit operations including ozone generator sizing requirements, Krofta DAF sizing requirements and spiral wound membrane filtration sizing requirements for the full scale facility.
- Evaluate standard venturi type ozone injectors against the ESTR patented hydrosparge ozone injector. The justification for this was the standard venturi ozone injector used considerable amounts of energy to make it work properly, while the patented ESTR hydrosparge ozone injector could provide the same ozone injector performance at a fraction of the energy requirements as a standard venturi. Appendix B provides a patent drawing on the ESTR hydrosparge.
- Produce treated process water for further membrane filtration work to be carried out by ESTR, this to evaluate spiral wound membrane filtration for ultrafiltration, nanofiltration and reverse osmosis membrane filtration on the post DAF process water. This work was done to understand and confirm if the DAF clarification unit operation provides adequate protection to this type of membrane filtration operation so it can be incorporated into the system, with the goal of greatly improved process water cleanliness and the potential to produce a fresh water substitute from the clean process water.

Table 3 provides results data for the pilot trial work carried out on the Plattsburgh mill's process water, using the WMU pressurized ozone injection and Krofta DAF capability, for both a standard venturi ozone injector and the ESTR hydrosparge ozone injector.

Table 3: WMU Pilot Trial Results for Plattsburgh Process Water

SAMPLE	TYPE	pH	Temp *c	Dissolved O2 ml/g	Conductivity mU	Total Solids %
	System Feed					
T1	Process Water	7.44	31.2	7.03	0.909	0.15
T2	Process Water	7.44	32.5	7.02	0.909	0.19
T3	Process Water	7.44	32.5	7.00	0.909	0.16
T4	Process Water	7.44	32.5	6.99	0.909	0.16
T5	Process Water	7.44	32.5	6.99	0.909	-
avg		7.44	32.24	7.01	0.909	0.16
	Krofta DAF Accepts					
T1	Venturi O3	7.89	32.3	7.95	0.876	0.10
T2	Venturi O3	7.89	32.3	7.95	0.876	0.12
T3	Venturi O3	7.89	32.3	7.94	0.876	0.11
T4	Venturi O3	7.89	32.3	7.93	0.876	0.10
T5	Venturi O3	7.89	32.3	7.92	0.876	-
avg		7.89	32.3	7.94	0.876	0.11
% Reduction					3.6	35.1
	Krofta DAF Accepts					
T1	Hydrosparge O3	7.84	31.1	7.60	0.699	0.11
T2	Hydrosparge O3	7.84	31.1	7.59	0.699	0.12
T3	Hydrosparge O3	7.84	31.1	7.59	0.699	0.13
T4	Hydrosparge O3	7.84	31.1	7.58	0.699	0.11
T5	Hydrosparge O3	7.84	31.1	7.58	0.699	-
avg		7.84	31.1	7.59	0.699	0.12
% Reduction					23.1	28.6

The sample test results for the trial work showed that the Plattsburgh mill process water was responsive to the pressurized ozone per conversion of the TDS to TSS and subsequent removal by the Krofta DAF. TDS removal of approximately 28-35% was consistent with past historical data and TDS removal results. Additionally, both the TDS removal and the dissolved oxygen content in the process water samples were similar for both the standard venturi injector and the ESTR hydrosparge injector. Thus the expectation was that the ESTR hydrosparge injector could be used in place of a standard venturi with similar ozone response accomplished using far less energy.

Table 4 provides the test results for a series of spiral wound membrane filtration evaluations for ultrafiltration, nanofiltration and reverse osmosis filtration, carried out by ESTR at their laboratory facility, using samples of process water from the post Krofta DAF pilot plant process water samples developed at the WMU pilot plant.

Table 4: ESTR Pilot Trial Results for Spiral Wound Membrane Filtration

Sample	pH	TSS	TDS	Turbidity	Conductivity	Salinity	COD
Infeed	7.37	562	429	739	0.787	0.4	940
Ultra-filtration	7.37	14	480	20	0.881	0.5	370
Nano-filtration	7.37	BMDL	159.9	17	0.229	0.2	63
Reverse Osmosis	7.37	BMDL	6.6	6	0.134	BMDL	2

BMDL = Below Method Detection Limits

In the above spiral wound membrane filtration trial work results of Table 4, note that care was taken to prepare infeed samples to the appropriate levels required for simulating what might occur in actual full scale operations. For instance in the reverse osmosis sample the TSS present in the infeed would have to first be reduced to

acceptable levels, typically less than 10 ppm. The results and evaluation did show the spiral wound membrane technology would have advantages in that it would have very good water quality improvements at a much higher permeate or accept rate. This fit well with the system flow requirements. The reverse osmosis membrane filtration results also provided for a fresh water substitute potential that was considered very attractive for realizing significant savings against Plattsburgh's fresh water costs when used as part of the pressurized ozone, clarification and filtration series to be called out in the system design. Figure 3 provides a photo of the actual water sample set results represented in Table 4 above.

Figure 3: ESTR Pilot Trial Membrane Filtration Water Sample Results



The visual results of Figure 3 put the test results of Table 4 into perspective per water quality that is attainable from the mill's process water. The use of pressurized ozone with DAF clarification in series with spiral wound reverse osmosis provided for the best opportunity for water and energy cost savings for the Plattsburgh mill.

SYSTEM DESIGN

Based on the research, pilot trial work and value engineering work, a new improved system design was developed that was simple yet effective, and incorporated the use of three key unit operation steps, a pressurized ozone unit operation, a Krofta DAF clarification unit operation, and reverse osmosis membrane filtration unit operation, all in series. These three unit operations are described individually as follows:

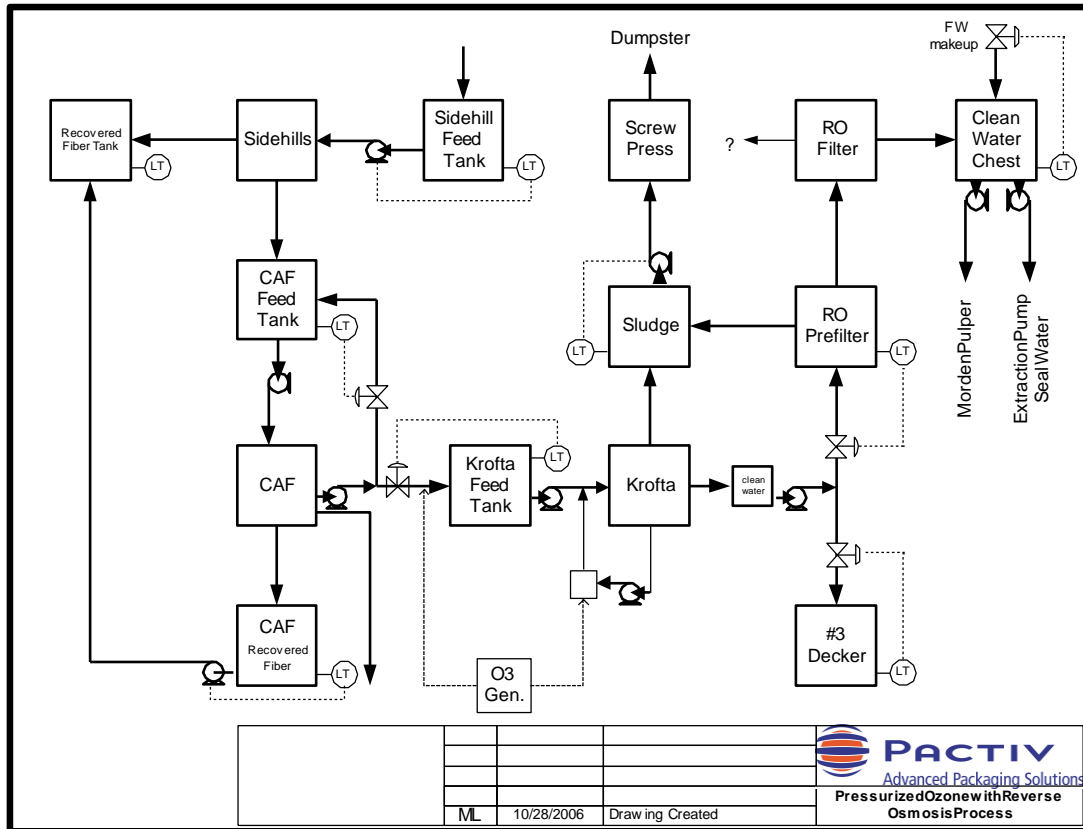
Pressurized Ozone Unit Operation: This unit operation consists of an ozone generator, which generates a continuous amount of ozone gas and feeds it to an ozone injector that then injects the ozone gas into the process water flowing in a pipeline. This ozone injection step is best provided for in conjunction with a sizeable retention tank where the process water flow can have adequate time to undergo TDS and ozone reaction and subsequent mixing to maximize agglomeration and flocculation of the TDS into TSS. The ozone injection can be done in a pipeline that feeds the tank directly or in a recirculation line that is connected to the reaction vessel tank. Appendix C provides the Ozone Water System ozone generator specifications for the project.

Krofta DAF Clarification Unit Operation: This unit operation consists of a Krofta DAF clarifier or equivalent flotation style clarifier. The goal of the Krofta DAF clarification step is to efficiently remove as much TDS as TSS as is possible to produce clean process water suitable for re-use in the mill. The Krofta DAF also has as goal to reduce the TSS to levels necessary to protect the reverse osmosis membrane filtration unit operation. Typically a TSS less than 5 ppm is required so as not to plug the spiral wound membranes. The Krofta DAF clarification will also act to reduce the TDS loading on the reverse osmosis membrane filtration unit operation and allow it to run more efficiently, with a higher permeate or accept percent, and more importantly the lowest possible membrane filtration reject stream. This will maximize the performance and life of the membrane filtration unit and reduce operational and maintenance costs. Appendix D provides the Krofta DAF vendor specifications and information.

Reverse Osmosis Membrane Filtration Unit Operation: This unit operation takes the clarified DAF process water and uses spiral wound membrane filtration to produce a fresh water substitute. The reverse osmosis unit operation typically consists of two stations, a prefilter skid station and the reverse osmosis skid station. The prefilter skid station uses 300 mesh to 500 mesh sweco filters and/or a series of bag filters from 5 microns down to 1 micron in size, this to protect the expensive reverse osmosis membranes from TSS. The reverse osmosis skid station can run at a high or low pressure, with a higher efficiency that increases permeate or accept water and decreases concentrate or reject water accomplished running at a high pressure. Permeate or accept rates above 90% are possible. However there are situations where the preference may be to run at a lower pressure, to save on energy costs for instance, particularly if recirculation of the concentrate stream flow is possible.

Based on the above three key unit operations, a system design was developed for the Plattsburgh mill using existing and new equipment. Figure 4 provides the process flow diagram of the new technology application system integrated into some of the mills existing unit operations required for system support.

Figure 4: ESTR Pilot Trial Membrane Filtration Water Sample Results



As shown in the above process flow diagram, the system consists of the following unit operations:

Sidehill Screens (existing) – the sidehills screens are fiber removal screens that remove large cellulose fiber from the process water cost effectively, using a simple gravity filtration based mechanical action.

CAF Clarifier (existing) – the CAF clarifier removes most of the TSS as cellulose fiber and ash from the process water, down to approximately 20 – 50 ppm TSS, and provides for a process water stream that has mostly TDS containing. This helps to maximize the use of the pressurized ozone at the injection point so that the ozone is reacting on TDS as opposed to TSS.

Krofta Feed Tank – This tank acts as both the feed to the Krofta DAF and the reaction vessel for the pressurized ozone treated mill process water to have some retention time for agglomeration and flocculation of TDS into TSS, this prior to moving forward to the Krofta DAF for clarification removal efforts.

Ozone Generator and Pressurized Ozone Injection – The ozone generator produces ozone gas and sends it to the ozone injector point, here an ESTR hydrosparge injector. The ozone is injected into the process water in the recirculation pipe and is looped back into the Krofta Feed tank where it can realize some retention time.

Krofta DAF Clarification – The Krofta DAF clarifier takes the pressurized ozone treated process water whereby as much TDS has been converted to TSS as is possible, and uses bubble flotation clarification methodology to float TSS to the surface forming a matt, whereby it is removed by a clarifier’s reject scoop.

Accept water is returned to the mill as clean process water or sent forward to the reverse osmosis membrane pre filtration step.

RO Prefilter Skid – The prefilter skid has a sweco vibrating screen with a small mesh size of between 300 and 400 and a series of 5 micron to 1 micron bag filters to protect against failure of the Krofta DAF removing TSS and potentially plugging up the reverse osmosis spiral wound membranes. Process water from the prefilter skid can be taken from the sweco vibrating screen accept flow or the bag filter accept flow to provide a very clean process water that is suitable for mill shower water, etc..

RO Membrane Filter Skid – The RO membrane filter skid processes the water from the prefilter skid using the spiral wound membranes. The permeate or accept flow from this unit operation is a fresh water substitute quality, suitable for replacing fresh water applications in areas like chemical additive makedown, pump seal water, etc.

The above pressurized ozone, DAF clarification and reverse osmosis system was installed and integrated into the Plattsburgh mill, with the new technology application's startup and operation. Appendix F provides for a photo log of the installed system's unit operations for review and correlation to the process flow diagram.

PROJECT RESULTS

The full system was run operationally using mill process and sample sets where taken to provide for analytical test results. For the purposes of sampling, five sample points where used as indicator points, with the tracking labels as follows:

- **MST** – This sample point was the Main Storage Tank, which is also the CAF Feed Tank, which represents the used mill process water and starting point for the treatment system.
- **CAF** – This sample point was the CAF clarifier accept flow, which represents the process water for the mill's current configuration. Typically the majority of TSS is removed during this stage, and residual TSS is in the range of 20 ppm, however there is little to no TDS removal.
- **Krofta** – This sample point was the Krofta DAF clarifier accept flow, which represents the process water after it has been treated with pressurized ozone and clarified using the Krofta's dissolved air flotation. The residual TSS in the Krofta DAF accept stream is at or below 5 ppm, and typically you will see a 25% to 35% reduction in TDS from the upstream pressurized ozone step converting TDS to TSS.
- **Sweco** – This sample point is the 300 – 400 mesh sweco vibrating screen accept stream, which helps to protect the reverse osmosis membrane. Typically this stream is similar to the Krofta DAF accept, however it may differ if there are upsets to the Krofta DAF that allows TSS to pass through.
- **Bag** – This sample point is the bag filter accept stream, which is after the 5 micron and 1 micron bag filters. The bag filter also works to protect the reverse osmosis membrane filtration unit. Typically the bag filter will remove any residual TSS, even small particle TSS but allows TDS to pass through.
- **Membrane** – This sample point is the permeate or accept stream of the reverse osmosis membrane filtration unit. This stream typically has test results equivalent or even superior to fresh water.
- **Concentrate** – This sample points is the concentrated reject stream from the reverse osmosis membrane filtration. This stream should be the most contaminated in the system since the reverse osmosis unit removes contaminants from as much as 90% of the flow and concentrates it in the remaining 10%.

Figure5 shows a photograph of the typical sample set take as per the list of sample points above, and thus moving left to right shows the general improvement in water quality as you move forward through the system, with the obvious exception of the concentrate sample on the far right.

Figure 5: Sample Set Example Photo



The system generally performed well and exceeded expectations per output. The Krofta DAF is specified to have an output of about 350 gallon per minute and readily exceeded that flow rate, producing as much as 450 gallons per minute in output flows of quality process water. The Reverse Osmosis Membrane Filter's output is specified at 85 gallons per minute but it was producing as much as 200 gallons per minute at times. What this signifies is the system is working well together to reduce the loading and thus improve the overall outputs and efficiencies, and increasing the capacity of the system to produce clean process water and fresh water substitute.

The analytical test results generally showed a very good quality improvement in the process water quality. However it should be mentioned that at the time of these samples the mill had a sizable excess of chemical additives and dispersant in their mill process water, and this seemed to interfere with the pressurized ozone unit operations capability to convert the TDS to TSS and thus the typical reduction in TDS at the Krofta was not realized. However the upset showed the system was robust enough to keep producing quality clean process water and fresh water substitute while a mill upset was occurring.

Table 5 shows the results for the TSS analytical testing. A large amount of TSS as cellulose fiber is contained in the mill process water at the MST sample point. This is removed at the CAF clarifier and returned to the mill for raw material use. Note that the CAF TSS average amount of 18.3 mg/L would be detrimental to the reverse osmosis unit if allowed to pass though, however the Krofta DAF clarifier removes about 75% of the remaining TSS and the result is a very low average TSS value of 4.8 mg/L in the Krofta DAF accepts. By the time the

process water passes through the bag filter there is no TSS remaining, thus the reverse osmosis unit operation is fully protected.

Table 5: TSS Analytical Test Results (mg/L)

Sample	MST	CAF	KROFTA	SWECO	Bag	Membrane	Concentrate
1	924	10	5	6	<4	<4	<4
2	1230	18	4	<4	<4	<4	6
3	1360	27	4	<4	<4	<4	<4
4	1470	18	6	6	<4	<4	9
Average	1246.0	18.3	4.8	3.0	0.0	0.0	3.8

Table 6 shows the TDS analytical test results for the process water. There are only minor TDS drops through the system until you get to the reverse osmosis unit operation, where the TDS drops to only 11 mg/L. Note how high the TDS level rises in the concentrate. While a dramatic drop in TDS in direct response to ozone injection was not observed (comparison of the CAF and Krofta samples does not show direct conversion of TDS to TSS), it is possible that key reactions of ozone do occur to prepare the TDS for removal in later treatment stages. Future experiments would be warranted to vary the dosing of ozone (including experiments where complete elimination of ozone dosing is attempted) in order to isolate its specific contribution to the overall treatment train.

Table 6: TDS Analytical Test Results (mg/L)

Sample	MST	CAF	KROFTA	SWECO	Bag	Membrane	Concentrate
1	522	526	504	466	474	<20	2040
2	506	524	504	460	474	<20	5060
3	524	484	486	456	423	22	1230
4	454	426	412	366	422	20	4840
Average	502	490	477	437	448	11	3293

The analytical test results for turbidity obviously see the biggest drop in the CAF clarifier, due to the large amount of cellulose fiber removed. However turbidity does drop by significant amounts on a percentage basis through the rest of the system's unit operations, until it reaches zero at the reverse osmosis membrane permeate or accept flow. This matches up well with the visual effects in the sample photo of Figure 5.

Table 7: Turbidity Analytical Test Results (NTU)

Sample	MST	CAF	KROFTA	SWECO	Bag	Membrane	Concentrate
1	866	9.11	5.85	4.38	1.98	<1	6.32
2	890	8.41	3.8	4.73	2.1	<1	26.6
3	907	5.43	4.18	3.32	1.73	<1	3.89
4	971	10.2	6.1	7.58	1.5	<1	12.8
Average	908.5	8.3	5.0	5.0	1.8	0.0	12.4

Table 8 provides the analytical test results for conductivity. Conductivity can indicate excesses in both organic and inorganic ionic material, and can be very detrimental to chemical additive use, interfering with sizing

chemical or retention aid polymer use for example. While a small improvement is realized at each unit operation, as expected the largest gain is seen at the reverse osmosis membrane unit operation.

Table 8: Conductivity Analytical Test Results (microhos/cm)

Sample	MST	CAF	KROFTA	SWECO	Bag	Membrane	Concentrate
1	645	626	612	598	590	<20	2190
2	622	625	604	562	578	27.8	4540
3	625	602	607	551	540	26.5	1390
4	577	536	509	484	524	25.9	4400
Average	617	597	583	549	558	20	3130

Tables 9, 10, 11, and 12 provide the analytical test results for hardness, total calcium, dissolved calcium and dissolved magnesium. Once again only minor improvements are realized through the system until the reverse osmosis unit operation is reached. The hardness, calcium and magnesium that are in the process water can be a severe detriment to mill production by fouling fiber molds or equipment, leading to significant downtime.

Table 9: Hardness Analytical Test Results (mg/L)

Sample	MST	CAF	KROFTA	SWECO	Bag	Membrane	Concentrate
1	200	199	209	210	200	<5	853
2	198	200	207	183	203	<5	2080
3	209	189	206	193	176	<5	545
4	191	191	184	161	185	<5	2030
Average	200	195	202	187	191	0	1377

Table 10: Total Calcium Analytical Test Results (mg/L)

Sample	MST	CAF	KROFTA	SWECO	Bag	Membrane	Concentrate
1	117	70	62.8	67.4	64.5	<1	272
2	106	68.1	62.9	63.1	61.9	<1	709
3	108	67.7	64	59.3	55.4	<1	171
4	92	62.8	56.5	52.2	56.5	<1	632
Average	106	67	62	61	60	0	446

Table 11: Dissolved Calcium (mg/L)

Sample	MST	CAF	KROFTA	SWECO	Bag	Membrane	Concentrate
1	65.1	62.4	67.2	66.6	63.8	<1	268
2	63.6	64.6	66.2	57.5	64	<1	678
3	66.8	60.3	65	59.8	54.5	<1	172
4	60.2	58.9	56	48.9	56.6	<1	634
Average	64	62	64	58	60	0	438

Table 12: Dissolved Magnesium (mg/L)

Sample	MST	CAF	KROFTA	SWECO	Bag	Membrane	Concentrate
1	9.2	9.4	10.1	10.5	10	<0.5	44.3
2	9.5	9.4	10.2	9.5	10.6	<0.5	95.2
3	10.3	9.3	10.5	10.5	9.6	<0.5	28.1
4	9.9	10.7	10.7	9.4	10.7	<0.5	108
Average	10	10	10	10	10	0	69

Appendix G provides a preliminary analysis of cost saving estimates based on the Pactiv Plattsburgh installation. These cost savings estimates are summarized in Table 12. This estimate is based only on the savings realized from producing clean process water and fresh water substitute. The clean process water would mean that a similar amount of dirty process water does not have to be discharged as effluent, and thus a saving on effluent treatment charges, and the fresh water substitute means a savings on a similar amount of fresh water purchases. The analysis uses reverse osmosis membrane life expectancies of one year and two year durations. This results in yearly savings of \$67,000 to \$99,000 depending on how long the membranes last.

Table 12: Systems Savings Estimate per Year

Period	Fresh gal/yr	Process gal/yr	Total gal/yr	Treatment \$/yr	System \$/yr	Savings \$/yr
System (1yr)	75,600,000	100,800,000	176,400,000	\$ 311,119	\$ 244,101	\$ 67,018
System (2yr)	75,600,000	100,800,000	176,400,000	\$ 311,119	\$ 212,101	\$ 99,018

The saving analysis in Table 12 does not include energy savings that could be realized. For instance the biggest process water cost in the mill is to heat fresh water to process water operational standards. Typically this represents a temperature rise of 50 degrees, which based on current energy costs for producing or buying steam could provide for \$100,000 to \$400,000 in additional savings depending on where the system's produced process water and fresh water substitute is used. For instance if Pactiv Plattsburgh was able to use all the fresh water substitute as fresh water and eliminate the need to heat fresh water up to process water operational standards, an additional \$150,000+ in savings could be realized. Also, the savings due to improved chemical additive costs, reduced maintenance downtime and production rejects could also add tens of thousands of dollars to the mill's bottom line.

CONCLUSIONS AND FUTURE WORK

The technology as implemented exceeded the expectations of the original grant proposal per its technical capability. More work needs to be done to optimize the technology and fully understand the technology capabilities and industrial applications. The original grant proposal's goal was to produce cleaner process water. The actual technology implemented here produced a far superior series of cleaner process waters suitable for production requirements and even suitable for higher quality shower waters, etc., and it also allowed for the

production of a fresh water substitute from the dirty process water. Future work with the technology installation should consist of the following tasks:

- Perform minor equipment and control modifications and upgrades to the system to allow the system to operate in production at optimum levels fully integrated into the mill environment.
- Develop a better understanding of the process chemistry associated with the system per ozone reactions for optimizing TDS to TSS conversion, pH interactions, DAF charge vs. removal efficiency, etc. In particular, while a dramatic drop in TDS in direct response to ozone injection was not observed (comparison of the CAF and Krofta samples does not show direct conversion of TDS to TSS), it is possible that key reactions of ozone do occur to prepare the TDS for removal in later treatment stages. Future experiments would be warranted to vary the dosing of ozone (including experiments where complete elimination of ozone dosing is attempted) in order to isolate its specific contribution to the overall treatment train.

The evaluation of results for the new technology using pressurized ozone, DAF clarification and reverse osmosis membrane filtration allow for the following conclusions:

- The use of the pressurized ozone and DAF unit operations can minimize solids-loading to a reverse osmosis membrane filtration unit and allow for the more efficient production of a fresh water substitute from dirty process water, helping to reduce the amount of fresh water purchases.
- A sequence of the pressurized ozone, DAF clarifier and reverse osmosis membrane filtration can be used cost-effectively and provide overall savings to a mill while improving production related to water quality.
- The technology potentially has a broad application in the Pulp and Paper Industry and other water intensive industries to allow process water to be re-used in the mill to reduce or eliminate effluent discharge or to produce fresh water substitute as an energy and cost saving measure.

Appendix A

Project Partners and Key Vendors List

New York State Energy Research and Development Authority

17 Columbia Circle, Albany, New York

Contact: Dr Dana Levy, Senior Project Manager, email - dll@nyserdera.org

NYSERDA provided matching funds and senior project management as well as technical expertise to the project. NYSERDA acted as interface between the Pactiv project team and the Department of Energy STAC program. NYSERDA also assisted Pactiv in the cost effective procurement of the Reverse Osmosis membrane filtration unit.

The Energy Office, Michigan Department of Consumer and Industry Service

Contact: John Trieloff, email – erdinfo@michigan.gov

The Energy Office worked with the Michigan based project team members and will provide technology transfer and outreach to interested industrial parties and entities.

Pactiv Corporation, 74 Weed Street, Plattsburgh, New York

Contact: Mark Lowther, Plant Engineer, email – MLowther@Pactiv.com

The Pactiv Corporation's Plattsburgh, New York mill is a 40 ton per day molded fiber products mill. Pactiv Corporation is the Host Site where the new technology application system was installed and operated. Pactiv provided the required in kind matching cost share. Pactiv provide management, technical engineering, purchasing, maintenance and operational staff to support the project activities.

Cellulose Products and Services LLC, 304 Kings Hwy, Wyandotte, MI

Contact: Peter Rudy, Senior Consultant, email: pjrudy@wyan.org

Cellulose Products and Services LLC, with representation by Peter Rudy, who created/developed the intellectual property and is a leader in the field of dissolved solids removal technologies. Mr. Rudy served as Principal Investigator for the project, and is the primary technology contact. Mr. Rudy has many affiliations with industry representatives who work with or possess new technologies applicable to dissolved solids problems.

Western Michigan University, Paper Industry Pilot Plant, Kalamazoo, MI

Jan Walter, General Manager, email – jan.walter@wmich.edu

Western Michigan University's Department of Paper and Printing Science and Engineering, represented by Jan Walter, provided the Paper Industry Pilot Plant's water recycling facility to perform pilot plant work. The pilot plant allowed for ozone study and evaluation and analysis for determining process and system. Western's prestigious Department of Paper Science and Engineering is the premier Pulp and Paper research university in North America, and was awarded a \$1.3 million EPA grant to study just this kind of industrial recycled process water usage and treatment technologies. The Paper Industry Pilot Plant located at Western has a full scale pressurized ozone system available for pilot trial work on process waters.

Key Vendors:

GEA Filtration, Niro Inc, 1600 O'Keefe Road, Hudson, WI

Contact: Bob Keefe, Market Manager, email - rjk@niroinc.com

Niro Inc. supplies membrane filtration equipment to the industry and supplied retrofit and maintenance services, startup and operational assistance for the RO filtration unit, and Reverse Osmosis filtration expertise to the project.

Ozone Water Systems Inc, 5401 South 39th Street, Suite 1, Phoenix, AZ

Contact: John Overby, email - overby@ozonewatersystems.com

Ozone Water Systems Inc provided the ozone generator system along with expertise in ozone chemistry. This system along with all peripheral ozone equipment requirements such as ozone destructor units was provided. The ozone generator unit included the capability to produce oxygen from mill air to use as the ozone raw material.

Krofta Technologies LLC, PO Box 7, 401 South Street, Dalton MA,

Contact: Rick Ziomek, Sales Manager, email - rziomek@kroftatech.com

Krofta Technologies provided the Krofta DAF along with expertise on DAF clarifier and flotation technology and assisted with equipment upgrades and recommendations.

Environmental Systems Technology & Research Inc., 1824 Brussels Road, Brussels WI

Contact: Gaylen LaCross, President, email - estrinc@itol.com

Environmental Systems Technology & Research Inc provided the hydrosparge which was used for ozone injection, as well as provided pilot plant and on site sampling, laboratory and analytical testing capability to assist in the evaluation of the new technology.

Appendix B

ESTR Injector Patent Drawing

US006682057B2

(12) United States Patent
La Crosse

(10) Patent No.: US 6,682,057 B2
(45) Date of Patent: Jan. 27, 2004

(54) AERATOR AND WASTEWATER TREATMENT SYSTEM
(75) Inventor: Gaylen R. La Crosse, Brussels, WI (US)
(73) Assignee: ESTR, Inc., Brussels, WI (US)
Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

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Primary Examiner—C. Scott Bushey
(74) Attorney, Agent, or Firm—Stiennon & Stiennon

(57) ABSTRACT

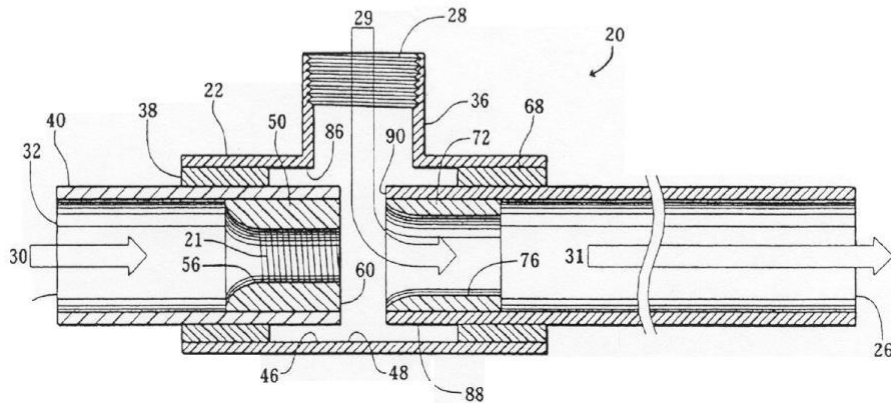
An aerator has a housing which contains a fluid inlet nozzle and a fluid discharge nozzle positioned on either side of an air inlet formed in a T-pipe. The fluid inlet nozzle has a bore with a flared inlet, and a cylindrical outlet, in which a spiral groove or rifling is formed which extends to the end of the inlet nozzle, allowing the infed contaminated water to pass through, being swirled by the spiral groove, and then exit into an expansion chamber in communication with the air inlet, where air is entrained within the swirling water. Banks of the aerators are used in a wastewater treatment system, having a rectangular tank with a serpentine flow path. Dissolved oxygen meters provide data to a Programmable Logic Controller to control the pumps recirculating liquid within the tank. Pumps are turned on and off to achieve target minimum levels of dissolved oxygen.

14 Claims, 4 Drawing Sheets

(21) Appl. No.: 09/847,064
(22) Filed: May 1, 2001
(65) Prior Publication Data
US 2002/0163089 A1 Nov. 7, 2002
(51) Int. Cl.⁷ B01F 3/04
(52) U.S. Cl. 261/76; 261/79.2; 261/DIG. 75
(58) Field of Search 261/76, 77, 79.2, 261/DIG. 75; 210/221.2, 760, 765

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C 1 BackFlow Prevention Device

Dimensions: 21" wide x 23" tall x 8" deep

MATERIAL: PVC (WHITE- UV RATED)

Material: Stainless Steel (type 316), PVDF/PFA
Tubing is PFA w/ Flared Fittings

Ozone Gas Connections: 1/2" FNPT

Air/Gas Inlet: 1/2" FNPT (80 psig required MINIMUM)

Voltage: 120 Volt/1phase/60 Hz

D 1 Refrigerated Dryer

Installed upstream of oxygen generator and downstream of customer air supply

E 1 Interface Panel

Ambient Ozone Monitor *SOFT*
Ozone Cooling Water Flow Switch *SOFT*
Ozone Gas Flow Switch *SOFT*
Backflow Prevention Device Alarm *HARD*
Emergency Stop *HARD*

All Interlock Devices Shall be Non-Latching
Except on Hard Shutdown.

Panel shall have status lights to indicate these conditions.

Panel shall have 24 VDC Power Supply

F Oxygen Concentrator OC-250

General:

Oxygen Output: **250 SCFH @ 0-45 psig**
6.6 Nm³/hr @ 0-310 kPa

Dew point: -150° F [-100° C]

Oxygen Purity: 90% +/- 5%

Feed Air Requirements:

47 SCFM @ 90-150 psig

73.6 Nm³/hr @ 620-1035 kPa

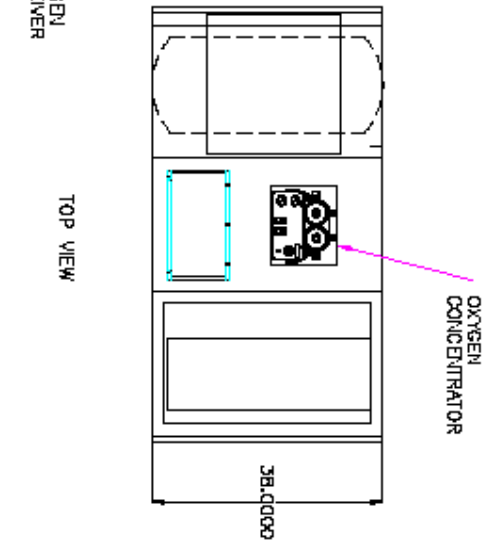
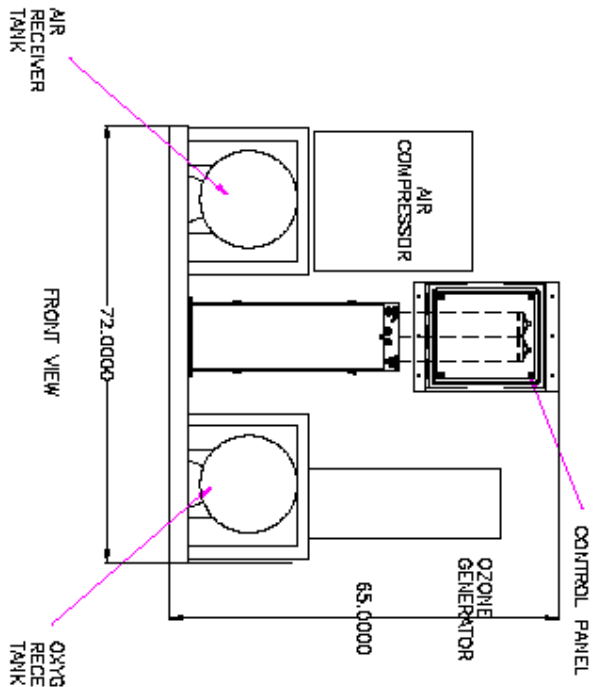
Dimensions: 30" W x 28" D x 78" H
76 cm W x 71 cm D x 198 cm H

Weight: 1000 lb. (454 kg)

Power Req: 120 VAC, 60 Hz, 1 Phase (1.5 ampere)

- G** **1** **Ambient Ozone Analyzer**
 Ozone Detection: 0 – 2 ppm
 Units of Measurement parts per million (ppm) (4 Digit LED)
 Enclosure: NEMA 4X
 Dimensions: 7.17” Wide x 7.09” high x 4.33” deep
 Input Power: 85 – 265 VAC; 50/60 Hz
 Complete with Self Checking Ozone Sensor
 Audible and Visual Alarms
- H** **1** **Power Distribution Panel**
 Customer to supply 575V/3ph/60Hz to skid and OWS will distribute the necessary power to each piece of equipment on the skid.
- I** **1** **Skid Assembly of water components A-G**
 Skid shall be painted carbon steel. All equipment to be skid mounted with one common electrical connection.
- J** **2** **Days for equipment check out and start-up**

NOTE: ALL DIMENSIONS UNLESS OTHERWISE SPECIFIED ARE IN METERS. DIMENSIONS IN PARENTHESIS ARE IN FEET AND INCHES. DIMENSIONS IN PARENTHESIS ARE UNCONTROLLED.



REVISION 1
OZONE GENERATOR AND AIR COMPRESSOR MOVED

THIS DRAWING HAS BEEN GENERATED AS A 2D REPRESENTATION OF A 3D MODEL. DIMENSIONS MAY VARY SLIGHTLY FROM THE 3D MODEL. DIMENSIONS IN PARENTHESIS ARE UNCONTROLLED.

TYPICAL SKID LAYOUT	
NO. OF SKIDS	1
SKID LAYOUT	AS SHOWN
APPROVED BY	J. [Signature]
DATE	11/25/08

Pact IV Ozone System Procedures

Start-Up

1. Plug in control power cord.
2. Turn AirSep switch to on. Wait until middle gauge is above 45 PSI.
3. Turn on ozone generator main power. (red switch on left hand side of cabinet)
4. Push purge on button. (ozone generator keypad)
5. Turn on injector pump and open injector gas valve.(If down for 1-3 days purge for one hour, 4-10 days purge for 6 hours, over 10 days purge for 24 hours)
6. Adjust gas flow to desired flow and set pressure at 1.2 Bar.
7. Open water flow to 2.5 GPM.
8. Set ozone monitor gas flow to 1 liter per minute.
9. Push PSU on. (ozone generator keypad)
10. Push purge gas off. (ozone generator keypad)
11. Push remote control settings. (this engages safety interlocks)
12. Push set point up or down to get ozone production to go up and down.(keep the set point control in local)

Pact IV Ozone System Procedures

Shut Down

1. Put control in local.
2. Turn set point all the way down to zero.
3. Push PSU off button.
4. Push Purge on button. Wait 20 minutes.
5. Close gas ball valve at injector pump.
6. Turn off injector pump.
7. Push purge off.
8. Close cooling water flow valve.

9. Turn off ozone generator (red switch on left side of ozone generator)
 10. Turn AirSep switch to off.
- Unplug control power.

Safety Notice

The ATI ozone analyzer visible through the doors glass window displays the ambient ozone levels inside of the room. If the ozone in the room gets above .1 PPM the equipment will turn off.

The 8 hour exposure level is .1 PPM.
The 15 minute exposure level is .3 PPM.

Consult the MSDS before entering.

Appendix D

Krofta DAF Specifications

Ref: **CANAM # 23661**

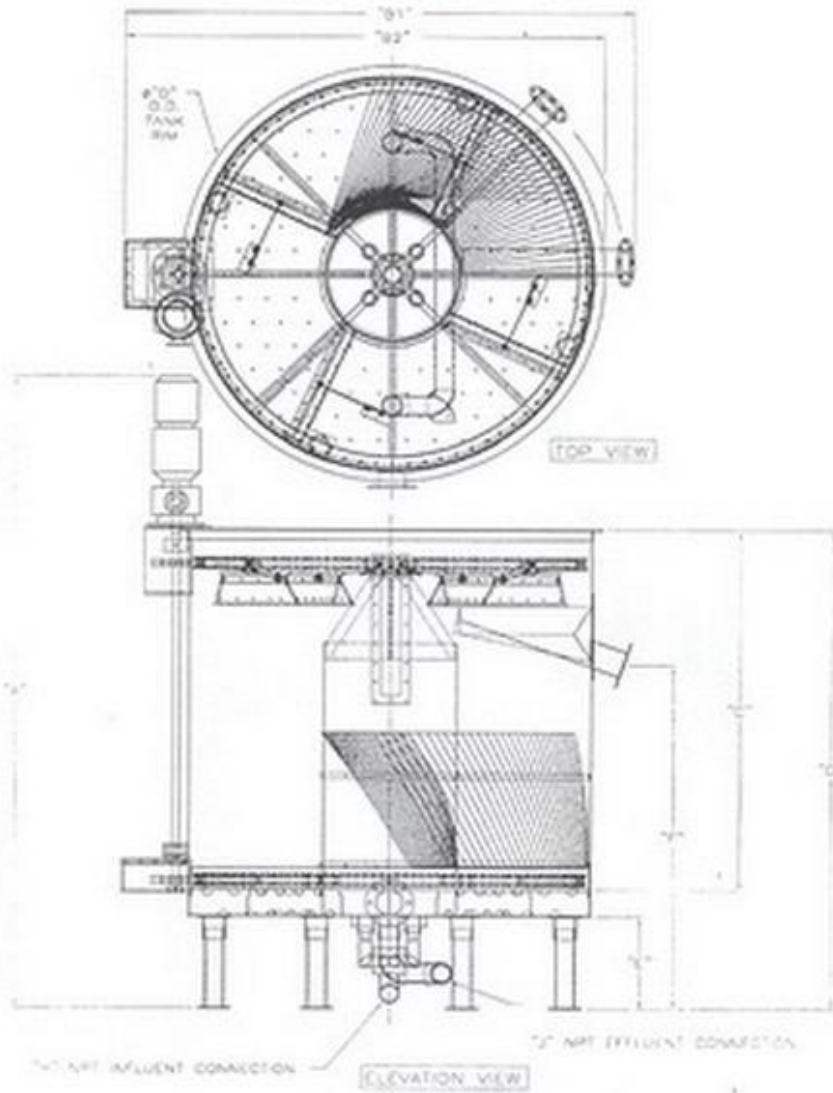
This is to follow up on your recent request for equipment from **Can-Am Machinery**.
At present we have the following available:

Class	Qty	Part No	Unit Price	Terms
<hr/> WASTE WATER/CLARIFIERS				
	1	17318	\$39,000	
FLOTATION CLARIFIER, 12 FT., STAINLESS STEEL , Mfg: KROFTA , Model: SUPERFLOAT12				

If this is of any interest or you would like additional information, please let us know.

Sincerely,
CAN-AM MACHINERY, INC.

KROFTA SUPERFLOAT-III GENERAL ARRANGMENT
6, 8, 10 & 12 FT DIAMETER



SFR SUPERFLOAT-III TANK DIA.	NOMINAL CAPACITY GPM	A"	DIMENSION TABLE VALUES - INCHES								
			B1"	B2"	C"	D"	E"	F"	G"	H"	
6 FT	150	115	92	84	65	76	17	63	87	1	3
8 FT	250	115	104	96	65	76	17	63	87	4	4
10 FT	400	120	142	134	65	78	22	68	92	5	5
12 FT	600	120	166	158	65	78	22	68	92	6	6

Description of typical Krofta DAF clarifier technology operation

Supracell DAF

Features and Advantages:

- There is generally only 18"-22" of water in the clarifier, affording the unit a very low floor loading.
- Units typically weigh 160 pounds/sf or less.
- The shallow tank design provides for easy tank inspection and cleaning.
- A viewing window mounted on the side of the tank facilitates observation of the flotation process and assists in optimizing chemical dosing (if necessary).
- The shallow, open tank design makes the unit ideal for installation on a wide variety of applications.
- Because all inlet and outlet connections are on the bottom of the unit, it can be installed in an elevated position. This is beneficial when draining the clarified water or floated sludge by gravity, eliminating the need for an additional pumping stage.
- The spiral scoop mechanism affords precise sludge removal by 'biting' into only the sludge layer, avoiding the clarified water. This increases floated sludge consistency, benefiting downstream sludge handling equipment by reducing flow and chemical consumption on the press.
- An automatic level control system monitors flow flocculations and can keep the level in the tank accurate within 1/4" to 1/2" to ensure consistent and precise sludge removal.
- Stainless steel construction.

May be stacked for smaller footprint.

Appendix E

Niro Reverse Osmosis Membrane Filtration

MEMBRANE FILTRATION BACKGROUND AND THEORY

INTRODUCTION

The intention of the following discussion is to familiarize the reader to membrane filtration / separation at an introductory level.

Membrane filtration, as we discuss, has been used and known of for many years, but only more recently has been used on a commercial scale successfully. This is due to recent introductions of membrane materials and construction methods.

Before the 1970's, the membrane material was sensitive to the most commonly used cleaning chemicals and procedures and thus difficult to clean. The materials during the 1970's and 1980's have progressed to the point where membrane cleaning is typically not a problem if certain guidelines are followed.

Systems are thus found in many different types of industries, such as dairy and food, pharmaceutical, and biotech.

Another advance that is equally, if not more important, is the method of construction. Many systems today are equipped with spiral wound elements.

This construction method results in a much greater membrane area with equal energy consumption, less cleaning chemical use and requires less floor space than the designs which it replaced, such as plate and frame.

Ceramic membranes have also evolved during the 1980's and have become a major player in the micro-filtration area.

DEFINITIONS

Before the discussion goes too far, it would be helpful to define a few terms, which are often times used in the membrane filtration field. By no means do we wish to include all related terms, but just a few which are pertinent to the discussion and to the successful operation of the NIRO system.

A.T.D.

Anti-telescoping device. This device is located on the downstream end of the spiral element to prevent the leaves from telescoping, or being pushed downstream. Often times the A.T.D and interconnector are built as one.

B.O.D.

Generally expressed as BOD₅. An abbreviation of Biological Oxygen Demand.

This is the quantity of oxygen required to decompose organic material, expressed a parts per million (PPM).

BACK PRESSURE

The system operating pressure which is measured at the concentrate end of the element.

BACK PRESSURE VALVE

The valve which the concentrate is throttled through. Often times referred to as the concentrate flow control valve.

BOOSTER PUMP

See recirculation pump.

CENTER TUBE

See permeate tube

CONCENTRATE

See retentate

CONDUCTIVITY

Used as an approximate measurement of mineral content. Units commonly used are micro mhos/cm.

ELEMENT

Often times referred to as membrane. Can be used interchangeably. This refers to the filtration material that fits inside the pressure vessel.

FLUX

Permeate flow through a membrane. Generally calculated as liters/m²/hour (lmh).

FOULING

Refers to the flux decline of an element.

INTERCONNECTOR

This device connects two spiral element together or an element to the permeate end cap

MEMBRANE

Term used to refer to the semi-permeable material by which the separation process takes place.

MEMBRANE CUT-OFF

Term use to describe the molecular weight size which becomes too large to pass through a specific membrane. The molecules which are larger than the cutoff will be retained or rejected, whereas the smaller ones will pass through (permeate). Could be an absolute or nominal cutoff.

MODULE

A device which contains the element, such as a ceramic module, or plate and frame module.

PERMEATE

The material which passes through the membrane.

PERMEATE END CAP

This device is generally, but not always, located on the downstream end of the vessel and the permeate exits through it.

PERMEATE TUBE

A tube at the center of a spiral wound element through which the permeate flows into and thus exits the element. The interconnector also connects into this. Sometime referred to as the center tube or product water tube.

RECIRCULATING PUMP

Sometimes called a booster pump. In a recirculation loop, the pump is necessary for creating the optimum cross flow across the membrane surface.

RECOVERY

Generally described as a percentage. Used with reverse osmosis systems.

Calculated as the permeate flow rate divided by the feed flow rate and multiplied by 100.

REJECTION

Generally described as a percentage of salt rejection in a reverse osmosis membrane. Calculated as the percentage of salt which is held back by the membrane. $R=1-(C_p/C_b)$, where

- R= Rejection
- C_p = Concentration in permeate
- C_b = Concentration in retentate

RETENTATE

Often times called the concentrate. This material is held back by the membrane and is thus concentrated.

VESSEL

Often times called the pressure vessel. This tube contains the spiral element(s) inside it.

PROCESS

Generally, the process of separation takes place across a semi-permeable membrane. A pressure gradient is required to perform the separation. The type of filtration we are discussing involves a pump(s) which creates this gradient.

Thus, the pressure difference will cause a separation of different size particles or molecules to take place. The determination of what molecules pass is dependent on the membrane itself.

There are generally four different types of filtration which Niro deals with. These are; microfiltration, ultra-filtration, nano-filtration, and reverse osmosis. Often times, microfiltration and ultra-filtration are discussed together as are nano-filtration and reverse osmosis.

MICRO-FILTRATION AND ULTRA-FILTRATION

Micro-filtration and ultra-filtration (MF & UF) are separation processes which use a membrane that is more open than those used for reverse osmosis. Molecules such as salts and sugars will pass through (permeate) whereas the larger molecules such as proteins will be retained. Refer to the diagram included with this section for a schematic view of the various filtration processes.

In the processes of MF and UF, the macromolecules are being separated. Since this is the case, there is negligible osmotic pressure between the permeate and concentrate (osmotic pressure will be discussed further with reverse osmosis).

Since osmotic pressure does not play a part in the process, the separation will take place at pressures less than 10 bar (145 psi).

Typical MF processes might be chemical reclaim, salt brine clarification, dextrose clarification, etc... An example of using the UF process would be production of whey protein concentrate.

NANOFILTRATION AND REVERSE OSMOSIS

Nano-filtration and reverse osmosis (NF & RO) are separation processes which use a membrane where the pore size is so fine that only water and similar small molecules can pass through. With NF, monovalent salts and some simple sugars will pass, but with RO, even these will not pass, or will pass only very minimally. The permeate from and RO will contain only pure water and a very small amount of pure salts.

A typical NF process is one where all the proteins and sugars are retained but where the water and dissolved salts will pass through the membrane. An example would be desalting of whey. A typical RO process would be used in concentrating whey where only water is removed. Another could be "polishing" water, where the impurities are concentrated and the permeate becomes a more pure water than the feed.

The amount of salt which passes through a membrane depends on the membrane type as well as temperature and pressure. The "tightest" membranes are called "sea water" and have a salt rejection of not less than 98%. A "brackish water" membrane will have a salt rejection of not less than 96%. Most of the RO processes in which Niro is involved use the brackish water element.

For NF and RO to operate, the pressure difference must overcome the osmotic pressure. The osmotic pressure varies depending on the solutions being separated. For whey concentration the designed operation pressure is typically 30 bar (435 psi).

Osmosis occurs when two salt solutions of different concentration are separated by a membrane which only allows water to pass through. Because of the difference in concentrations, an equalization will take place between the two solutions. Water will pass through the membrane from the least concentrated to the most concentrated solution.

As can be seen in figure 4, the volumes on each side of the membrane are equal at the start. However, after equalization, the water level will be lower due to the transfer of water to dilute the more concentrated side. The level difference will make a hydrostatic pressure which will counteract the transfer of the water and thus stop the transfer. This difference is called the “osmotic pressure”. See figure 5.

If an external pressure larger than the osmotic pressure is applied to the more concentrate side, water will pass the opposite way through the membrane and concentrate the salt solution further. (Fig. 6). This, in effect, is what reverse osmosis does. It overcomes the osmotic pressure of the concentrated material and thus pushes water through the membrane. If this external pressure was to end, then the water again would pass back and dilute the salt solution.

CLEANING INSTRUCTIONS

Declaration

Cleaning and disinfecting of your membrane filtration plant is a very critical operation.

Please read all cleaning instructions and information fully. Be sure to fully understand them. Failure to follow all instructions may result in irreversible damage to the membranes.

For warranty purposes, only the cleaning procedure that Niro approves, in writing, may be used. It is the customer’s responsibility to inform Niro of any cleaning procedure changes before implementing them.

Niro must approve any proposed changes before they are implemented. Niro will not assume any responsibility for damaged membranes if the above is not followed.

Because of the experimental nature of pilot testing, it is impossible to specify in advance any particular cleaning regimen. This will be determined at the time of pilot testing once the nature of the product and type of membranes has been determined. The type of cleaning agents available during the pilot testing will also help determine a specific cleaning procedure. Niro Filtration personnel can assist you in developing the correct cleaning procedure.

Because of their experience with the membrane industry, Klenzade is one of the major chemical suppliers recognized by membrane manufacturers and Niro. Klenzade also has the expertise in the food and dairy industry to assist our customers with specific cleaning problems. Trial sizes of membrane cleaning supplies are available from Ecolab Customer Service by calling 1-800-224-5797. The trial sizes may only be obtained through use of special ordering codes. These may be obtained from a GEA Filtration service representative. Because of their unique formulation, it is not recommended that substitutions be made for

these products. It shall be the responsibility of the renter to insert the appropriate MSDS sheets in this manual for the chemicals used with this system.

CIP WATER QUALITY

Water used for flushing, cleaning and disinfection of reverse osmosis and Ultra-filtration plants must conform to the following standards to obtain best possible service. It is a prerequisite that the following standards be adhered to for the membrane guarantee to be valid. It is also recommended that the water be analyzed at least every three months to ensure proper quality. If the water quality does not meet these standards, consult a Niro representative.

Every six months, the analysis must be sent to Niro for review.

- Iron Less than 0.05 ppm
- Manganese (MN) Less than 0.02 ppm
- Silicate (SiO₂) Less than 10 ppm
- Aluminum (Al) Less than 1 ppm
- Hardness as (CaCO₂) Less than 10 gr./gal (170 ppm)
- Particles Less than 25 microns
- Turbidity Less than 1 NTU
- Total plate count Less than 1000/ml
- Coli count 0/100 ml
- Chlorine (RO/NF only) 0 ppm

CHEMISTRY

For membrane cleaning there are basically three types:

- Alkaline cleaners with various types of buffer systems classifying them according to the pH range they generate
- Neutral cleaners with or without enzymes
- Acid cleaners.

Most cleaning can be done with a good alkaline product. Most types of soil that we encounter require - or are best removed with alkalinity. Protein, most any kind of protein, is more easily removed at high pH values. At these pH values the protein also is slowly hydrolyzed, making it more soluble. As neutral values are approached, the solubility decreases, and at pH 4-5 many proteins can even be precipitated, they become quite insoluble and difficult to remove.

FAT

Under normal conditions of cleaning (time, temperature and alkalinity usually encountered), not much hydrolysis (called saponification, the making of soap) occurs. The higher the alkalinity, the higher the

temperature, the more fat will be hydrolyzed, leading to salts of fatty acids. If Calcium ions are around and not enough chelating power is in the cleaner (case of commodity caustic), then Calcium soaps, insoluble deposits will form which can clog up filters and also membranes. This should be avoided. Alkalinity based on caustic alone is not sufficient. It is difficult to control the pH; no buffer system is there; no detergency. There simply is no soil carrying capacity present. Builders are needed! Sodium or potassium silicate, a favorite builder substance is not suitable on membranes. Silicates do not rinse very well, and precipitate easily, especially when pH drops into the acid range. Sodium carbonate, Soda ash or phosphates are good builders and buffer system; however care must be taken to use well soluble material. Do not use the granular type that might dissolve too slowly and get into the membrane system where it might physically damage the surface by scratching it.

The curve of protein solubility suggests that the higher the pH the better the cleaning. We also know that in most cases we simply cannot use pH values of 13 or more, seldom more than 12.5 due to the restriction of the membrane. Built products have an enormous advantage over straight caustic. With the inclusion of various sequestrants, chelating or complexing agents into the formula increasing detergency, a built product can allow for the reduction of pH while making retention of cleaning efficiency possible. Sequestrants also, or primarily, react with the calcium and magnesium ions present in either the soil or the hard water employed or both. They aid greatly in soil removal.

NEUTRAL CLEANERS

Neutral cleaners are usually “enzyme cleaners.” Certain membranes, such as the CA and some sensitive composite membranes, do not support pH values higher than 7.5 or 9.5 respectively. As the protein solubility curve indicates this is a bad region for efficient soil removal. In order to make a product that is buffered to give a pH value of 7.5 or 8 in solution do a good cleaning job on protein, an enzyme (similar to protease of the stomach) is added. The enzyme slowly digests the protein molecules and makes them into water-soluble fragments. To speed up the action of the enzyme, one should work at the optimum pH of the enzyme activity, which is about pH 9 (already too high to CA) and at temperature around 120° F which may also be too high for some membranes. One could increase the amount of enzyme in the cleaner, as more enzyme molecules will be able to do more work. This is however costly. Enzyme cleaners are not only just composed of an enzyme preparation, they have builders and buffers, surfactants for emulsifying and dispersing soil.

Depending on the type of soil, it is possible to make a neutral cleaner of quality without enzymes (a lot less expensive) if no protein is present in the soil or without surfactants when no fat is present. Enzymes other than proteases have not been tried on a large scale, mostly because of the cost factor involved.

ACIDS

An acid cleaning step can often be skipped when a powerful quality cleaner has been used. It is recommended to use acids when high amounts of mineral deposits, calcium, magnesium, iron are present in the soil or in conditioning the membrane surface in the case of inorganic membranes. A blend of acids (nitric/phosphoric/citric) correctly chosen is usually better than straight commodities.

Among other things the blend is easier to handle, has the purity not always found in raw materials (an important factor in membrane cleaning) and the benefit of the various advantages of the pure acids.

SOLVENTS

Solvents such as chlorinated hydrocarbons or petroleum derivatives are not recommended in membrane cleaning. Compatibility with the membranes or the support material is often not assured, cleaning is restricted to only a particular type of soil (oil and grease, emulsions), and toxic and environmental hazards make these cleaners more and more obsolete.

OXIDIZERS

Sodium (or potassium) hypochlorite, chlorine bleach, is often used for cleaning and sanitizing. It helps in protein removal, but is corrosive, not only on stainless steel, but on certain membranes such as PA or TC. There are other disadvantages. Even on PS there are limitations as to concentration and temperature. Again, certain effects seem to work together, but in different ways than on stainless steel. Whereas on steel the recommendation with respect to chlorinated cleaners is to remain at high pH in order to decrease the chance of corrosion, the polymeric material suffers more from the combination of high alkalinity plus chlorine than from mild alkaline chlorine bleach alone. If membrane specs state temperature range from 60° F to 140° F, pH range 2 to 12 and chlorine level tolerated up to 300 ppm, that does NOT mean that it is safe to operate at 140°F, pH 12 and 300 ppm simultaneously.

The temperature tolerance is given at similar conditions. What one can do is a matter of negotiation between the user, the manufacturer and the supplier (KLENZADE).

HYDROGEN PEROXIDE

Hydrogen peroxide is used as a cleaning booster in some applications, not as effective as chlorine, but also not as destructive. In membrane systems it is sometimes used for cleaning and once again sanitizing, but the user is cautioned to review the membrane specifications before use.