



How membrane technology, and the right detergent, lets a valve manufacturer recycle more than just wastewater from its washers.

# The Ultimate in Ultrafiltration

by Mark Carroll, Senior Environmental Engineer

The use of a combination of proven and cutting-edge membrane technology along with a unique detergent has allowed a gas valve manufacturing company to completely recycle the wastewater and detergent from their aqueous parts washers. Our company is a division of a Fortune 100 industrial corporation with manufacturing plants in Arkansas, Missouri, Mexico, Puerto Rico, the Dominican Republic and several joint ventures in Europe and Asia. The division focuses on products for the residential and commercial heating and air conditioning market, with our customers being the major names in the HVAC business. Our plant employs approximately 800 people on shifts that operate 24 hours per day, seven days per week, to manufacture electronic natural gas valves such as those seen on residential hot water heaters and furnaces. Production volume is roughly 15,000 castings and finished valves per day.

The valve manufacturing process begins by melting aluminum ingots. The molten aluminum is die-cast into raw valve bodies and components, which are then machined to produce

the various precision surfaces and fittings. The machining operations are lubricated and cooled by a petroleum-based oil spray, which inevitably produces a casting that is literally dripping with oil and covered in aluminum chips. In the days of old (prior to the vapor degreasing NESHAAP), the wet castings were cleaned in a chlorinated-solvent vapor degreaser. These days, like many former vapor degreasing operations, we have converted to aqueous washing to remove the oil and chips from our castings. After the castings have been washed and dried, they are sent to final assembly and shipped to the customer.

## The “Old” Aqueous Wash

The concept of aqueous washing in itself is not rocket science – dirty parts are sprayed with soapy water and rinsed – but doing it well without spending a fortune on detergent and water, or creating environmental compliance problems can be tricky. Our operation consists of five large batch washers, with each having a wash tank holding 700 gallons and two separate rinse tanks of 300 gallons each.

The three tanks supply a common spray chamber in a wash-rinse-rinse sequence, much like a residential dishwasher. In the original configuration, the rinse tanks were constantly overflowed with fresh (and cold) tap water to control carryover detergent and oil from the wash tank. All overflow water was discharged to the municipal sewer system. The wash tank had no overflow in order to avoid diluting the detergent. Finally, every four days, all tanks would be drained and cleaned. Again, this wastewater was discharged to the municipal sewer.

This method of operation had several drawbacks that needed to be improved upon. In order of urgency, those problems were:

- High oil content in the discharged wastewater, up to 0.5% (~5,000 mg/l), causing constant violations of the “oil and grease” limit in our discharge permit.
- Very high detergent usage – 98 gallons per day – which was creating annual detergent bills of \$350,000.
- High fresh water consumption and high wastewater discharge – 10,000 gallons per day.

- Short bath life – four days.
- Wasted energy from discharging hot wastewater (150°F) and refilling with cold (60°F) fresh water.

### The Investigation Phase

After reviewing the list of problems, it gradually became clear that the ideal solution was some sort of wastewater recycling system – a closed loop that would be able to recover the beneficial constituents (detergent, water, and heat) and segregate the wastes (oil and particulate). With this goal in mind, we began investigating the available wastewater technologies, including centrifugation, dissolved air flotation, coalescing plate separation, and ultra-filtration. Many of the processes we examined would have been successful at removing the oil and making the wastewater “sewerable,” thus addressing our most pressing issue, the environmental compliance problem. However, our thinking was that if we were going to spend time and money on this situation, we wanted to do it right – merely producing sewerable wastewater was not enough. Only one technology held the promise

of being able to recover the hot water and the detergent – ultrafiltration.

At this point, many readers will begin shaking their heads, having experienced or heard the horror stories of membrane systems that never lived up to their billing. In fact, we were not impressed with what we initially found available in the membrane separation market. Most membranes systems would not tolerate a high oil and particulate loading, tended to require frequent cleaning, were easily damaged in the cleaning process, and had short life spans. We began to lose hope for the membrane separation concept. Fortunately, the manufacturer of our washers, CAE Ransohoff of Cincinnati, Ohio, has a vested interest in helping its customers handle wastewater from aqueous washers. Ransohoff has an internal division, the Environmental Systems Group (ESG), that is dedicated to research and development on wastewater recycling systems to aid its washer customers. It was the ESG group that found a new type of membrane that appeared to address the usual shortcomings of traditional

polymer membranes. This particular membrane is constructed of porous stainless steel tubing with a ceramic lining, which combine to make it an extremely durable membrane tube with an average pore size of 0.1 microns (uM). We were immediately impressed with the abuse it would stand up to, especially when it came to the cleaning cycle (See Figure 1). With the exception of a few unusual acids, just about anything can be used to clean the membrane, which translates to reduced cleaning time because a very aggressive solution can be used. After some initial skepticism, we were soon sold on this particular mem-

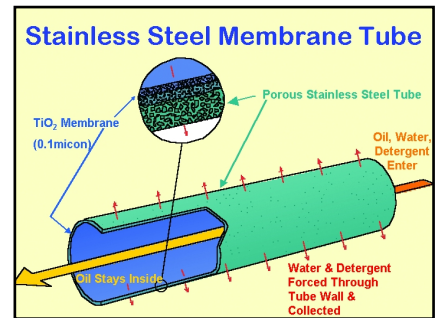


Figure 1: Membrane Filter Tube.

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brane, and decided to continue investigating ultrafiltration.

Next up was the seemingly simple task of finding a detergent that would properly clean our parts, not foam out of control in a spray washer, *and* pass through a 0.1 micron membrane intact. Simple task? Not quite. The detergent we were using, which, until then, we were quite happy with, fell on its face when it came to passing through the membrane. During a frank discussion with the manufacturer's chemists, we were told that they absolutely did not have a product that would work, and furthermore, they doubted that it could be done successfully at all. This revelation was quite discouraging. We embarked on a detergent testing odyssey, with each trial ending in some sort of failure. Once again, it looked very grim for the closed loop project. And once again, Ransohoff's Environmental Systems Group came through. Claiming to have found the silver bullet, ESG asked us to try an interesting, neutral-pH detergent they had recently come upon. With skepticism oozing out of our ears, we started yet another trial using this new detergent they called Evercycle 2000. Fully expecting another failure, we were shocked when the initial results showed that the detergent cleaned our parts well *and* passed through the laboratory UF membrane unchanged.

We proceeded to convert a single washer over to the new detergent and began learning how to use it properly. Initially, we had to figure how to measure the detergent concentration in the wash tank — we couldn't use the traditional acid-base titration to measure concentration because the Evercycle 2000 was a neutral-pH detergent. Luckily, the detergent does have a strong effect on the conductivity of a solution (and correspondingly, TDS), which can be used to measure detergent concentration assuming there is no change in the basic water quality.

Unfortunately, we were using municipal water supply with a conductivity of about 300 microsiemens. As water evaporated out of each washer, the hard water salts would begin concentrating in the tanks and made our conductivity readings completely unreliable. Furthermore, we knew that the same hard water salts would precipitate in a membrane sys-

tem and foul it. It was obvious that a full-scale system would require some form of pure water, and by the same token, the pilot system would require it to produce representative results. We decided to splice in a temporary set of deionizing cylinders into the municipal supply line that fed the washer. After that our conductivity readings were able to provide a reasonably accurate picture of detergent concentration on a day-to-day basis. As an additional benefit, the washer stopped accumulating a thick white cake of precipitated hard water salts on the tank walls and heating elements. We were now ready to begin an ultrafiltration pilot system trial on a single washer.

### The Pilot System Trial

For those not familiar with membrane separation terminology, a few definitions are in order.

- *Permeate* – the liquid that passes through the membrane (i.e. the water and detergent).
- *Concentrate* – the liquid that is unable to pass through the membrane (i.e. oil and particulate).
- *Flux Rate* – the rate of permeate flow per unit of membrane surface area, commonly specified as gallons/square foot/day.

We divided the trial into two phases. In the first phase we set up a temporary pump to pull water directly out of the wash reservoir and pump it to the ultrafilter, which would then discharge the permeate directly back to the wash reservoir on a continuous basis. There were four primary goals

for this phase:

- Determine, if indeed, that the detergent would pass through the membrane in practice (versus in a laboratory setting).
- Find the flux rate for our wastewater with this type of membrane (flux rate differs for every fluid).
- Determine the duration a minimum flux rate could be expected.
- Figure out how to clean the membranes, once the flux rate has dropped to an unacceptable level.

Once the temporary plumbing and electrical supply were set up, we flipped the switch and began collecting data. The first job, determining whether the detergent passed through the membrane properly, monitored by measuring the conductivity of the permeate and the concentrate, which provided a reasonably accurate picture of detergent passage. Throughout the trial, we consistently found less than a 15 percent loss in conductivity across the membrane, which in turn indicated less than a 15 percent loss of detergent across the membrane. One concern that cropped up was that the conductivity method might not be indicative of total detergent passage, in other words, what if a non-conductive component of the detergent (perhaps an antifoam compound) was not making it through the membrane, and therefore, not showing up as a loss on the conductivity meter? We decided that that issue could only be resolved when we sent the permeate and concentrate samples to the lab for the more extensive testing (we eventually found a laboratory testing method



Figure 2: The Ultrafilter System

that did provide better support for the assumption that the detergent was passing through intact).

In the meantime, we were reasonably confident of the detergent passage and moved on to the issue of sustainable flux rate. Here we made a classic membrane error. When a membrane system has just been cleaned, the flux rate is very high, however that initially high flux rate cannot be maintained. It is only after the "fouling layer" inside the membrane tube has reached an equilibrium that the flux rate reaches a more steady state (it eventually drops off, but the decrease is much slower). We made the mistake of thinking the ultrafilter should have maintained the initial flow, and were bitterly disappointed when the flux rate plummeted within hours. This occurred repeatedly. We would clean the membranes, turn the ultrafilter back on, be thrilled with the permeate flow rate, then watch dejectedly as the flow dropped hour by hour. Finally, one day as the permeate flow was dropping rapidly after a cleaning, we said "to heck with it, just let it run."

When we checked on it the next

morning, the permeate flow was about one-sixth of the flow immediately after cleaning. We weren't very happy, but we let it keep running, and the ultrafilter seemed to stay at that flow rate. Over a period of about a week, the flow rate barely budged! To us, this was a major breakthrough. While the flux rate was pretty meager, the overall concept seemed to work. Now, the viability of a larger system only depended on whether or not we could afford enough membrane surface area to produce the full scale flow we needed at the given steady state flux rate. Having discovered what seemed to be the steady state flux rate, we repeated the same pattern many times. Eventually we were satisfied that we knew what flux rate we could count on, and how long we could count on it.

That had the first three goals covered (detergent pass-through, flux rate, and duration). The fourth goal, a reliable cleaning method, was met during the period when we were expecting unrealistic flow rates. Because we were cleaning the ultrafilter virtually every day, we had the

opportunity to try multiple cleaning methodologies and chemistries, even going so far as to try a soy bean-extract solvent (which didn't work). We learned a great deal about the importance of working these issues out on a trial system when one of our cleaning processes coated the inside of the membranes with an impenetrable residue that we could not remove with anything. We ended up throwing away that membrane set and resolved to avoid those conditions on the full-scale system. We eventually settled on a two-step cleaning process that involved using an alkaline solution followed by an acidic solution.

That brought us to the second phase, which was trying to recycle the rinse water. This was actually a little trickier than the wash water because we had to remove not only oil and particulate, but also detergent that had carried over from the wash cycle (recall that our washers use a common spray chamber).

While the ultrafilter was doing a fine job of removing the oil and particulate, it did nothing about the carry-over detergent (as expected). The job of



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removing the detergent fell to a second stage nanofilter using traditional spiral-wound polymer membranes (the nanofiltration system - polymer membranes - was built by Fluid Engineering of Racine Wis.). While we did not cherish the idea of using the notoriously fickle spiral wound membranes, we felt that because the nanofilter would be using the clean permeate from the ultrafilter it would stand a better chance of being successful.

Thus, our rinse pilot system flow scheme consisted of pulling water directly out of the rinse reservoirs, passing it through the stainless steel ultrafilter membranes, sending the ultrafilter permeate over to the polymer nanofilter, and finally delivering the nanofilter permeate back to the rinse reservoir. In practice, the system worked very well, with the nanofilter doing a good job of rejecting and collecting the detergent, providing further evidence that the detergent was successfully passing through the stainless steel ultrafilter. In fact, it quickly became clear that the nanofilter concentrate tank was not a waste holding tank (as on the ultrafilter), but a tank full of valuable product – the detergent. We noted this happy situation and made plans for the full-scale system to have a provision for pumping the detergent scavenged from the rinse water back into the wash system.

### Full-Scale Installation

After six months of running the pilot systems (and overcoming numerous obstacles such as the problem of the very hot water (150° F) degrading the polymer nanofiltration membranes and the problem of preventing the cleaning solution from contaminating the wash water), we were convinced that the overall concept of using membrane separation to recover water *and* detergent from aqueous washwater and reusing it in exactly the same washing process was not only economically feasible and environmentally desirable; but would actually produce an enormous cost savings for our plant. Having solved the membrane and detergent issues, we were excited about the potential of being able to address all of our washer problems. At that point we felt we could:

- Eliminate our oily water compliance issues by eliminating the discharge entirely.

- Slash our detergent costs by recovering our detergent and reusing it.
- Drastically reduce our fresh water consumption by reusing our wastewater.
- Improve our bath life by being able to overflow all tanks, including the wash tank, without dilution.
- Save energy by reusing our hot wastewater instead of cold fresh water.

With those goals in mind, construction of a new 4,500 square foot recycling facility began in March 2000. Over two miles of insulated piping was installed to connect all the washers to the central recycling system. The overall cost of the installed system was about \$800,000. After a month of debugging, which included eliminating unexpected siphoning problems, adjusting the coalescing plate oil/water separators to maximize oil recovery and minimize water removal, experimenting with filter bag pore size to optimize large particulate removal and minimize premature blinding, and finding optimal operating pressures on the ultrafilters and nanofilters, the system came on line in December 2000.

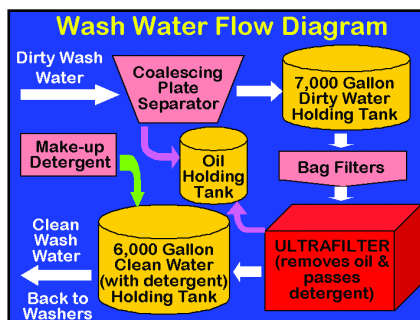


Figure 3: Flow Diagram.

### Recycling System Operation

Before discussing the operation, it is useful to note that essentially everything in our system (pipes, tanks, ultrafilters, meters, etc.) is in duplicate – one set for the wash water and one set for the rinse water. The sole exception is that the rinse water system has the previously mentioned nanofilter as a second stage to remove the carry-over detergent.

The washers would be considered the beginning of the closed loop. Total washer cycle time (including drying) is approximately 20 minutes per load. During every dry cycle, the washer's Programmable Logic Controller (PLC) opens a solenoid on the incoming dedicated "clean wash" and "clean rinse"

pipes (two separate pipes) and a set volume of hot, clean, water is pumped into the wash and rinse reservoirs. The incoming clean wash water has the detergent already premixed to the proper concentration (more on that later). This incoming fresh water displaces an equal volume of "dirty" water from each tank, which is discharged through overflow weirs to collection sumps (separate wash and rinse sumps). The collection sumps then pump the hot dirty water out of the main plant and over to the new recycling facility where it passes through coarse bag filters, flow meters, followed by coalescing plate oil/water separator. The plate separators drain to 7,000 gallon dirty water holding tanks (again, dual separators and dual tanks). (See Figure 3).

The dirty water hold tanks directly feed the ultrafilters. The permeate from the wash ultrafilters (*with the majority of the detergent still in it*) is sent on to a clean wash water holding tank. The rinse ultrafilter permeate goes to the nanofilter, and the nanofilter permeate is then sent to the clean rinse water holding tank. At this point it is important to note that while the Evercycle 2000 detergent is eminently recyclable, a small percentage is lost in the ultrafilter concentrate. That small loss is made up in the clean wash water holding tank by an automatic metering system that maintains the detergent concentration at the desired level. The clean, hot water (both wash and rinse) is then ready to be returned to the washers as the PLC calls for. This loop operates twenty-four hours per day, seven days per week.

In addition to the normal overflow wastewater flow, the recycling system also processes the wastewater from the routine bi-weekly washer cleanouts when each tank is completely drained and cleaned out. The flow pattern is exactly the same as that of the overflow water.

As mentioned in the "Investigation Phase" section of this article, the membranes will not tolerate hard water salts. To alleviate that problem, the recycling facility also houses a stand-alone reverse osmosis system that takes the municipal supply water and generates high-purity water. This purified water was used to initially charge the entire loop, and is currently used to makeup water in order to account for evaporation losses, and is

also used as the spray-down water during the washer cleanouts.

Now those familiar with membrane separation will point out that this is not a free ride – ultrafiltration is waste concentration, not waste elimination. After some period of time (which is specific to each system) the ultrafilter concentrate liquid will have collected enough oil and particulate that the fluid is no longer practical to process by the ultrafilter. This end-concentration point is usually indicated by a fairly rapid dropoff in permeate flow rate, but not always. Our system usually begins to drop off after we reach about a 100x concentration (400 gallon concentrate tank and 40,000 gallons of permeate), although occasionally the ultrafilter continues right on past 100x concentration point. In those cases, we act preemptively and discharge the concentrate tank anyway and refill it with a “fresh” batch of dirty water. The difference being that because the permeate flow has not dropped off, we don’t need to clean the membranes.

The concentrated waste liquid dis-

charged at the end of a processing cycle is why ultrafiltration is not a free ride – there are disposal costs associated with this liquid. In our case, the waste liquid, which we pump to a bulk holding tank, is an extremely oily water emulsion, with a large quantity of suspended fine particulate. Also included in this waste liquid is the spent membrane cleaning solution, which for us is a neutralized acid/base salt mixture. This liquid must be properly disposed of in some manner. We generate approximately 4,000 gallons of waste liquid per month, which we then send by tanker truck to an oil recycling facility (our used oil recycler is Safety-Kleen of Elgin, Ill.) where it is either re-refined into lower grade lubricants or into fuel.

### The Results

Overall, we were able to achieve and in some cases, vastly exceed our goals. First and foremost, since the system came online we have been able to maintain compliance with our wastewater discharge permit because the

heavy oil loading from the washers is no longer being discharged to the municipal sewer. Along with eliminating the oily discharge, we have also drastically reduced our overall wastewater discharge volume for the same reason. We were previously discharging approximately 10,000 gallons of wastewater per day. We now discharge less than 1,000 gallons per day. This reduction may take on more significance if the proposed Metal Products and Machinery pretreatment regulation becomes law, as it will allow us to be exempted from the regulation by keeping our discharge volume under the one million gallon annual threshold. The regulation is currently scheduled to be promulgated toward the end of 2002, with compliance mandatory by 2005.

Second, we have not merely “slashed” our detergent cost, we have decimated it! The recovery rate of the Evercycle 2000 detergent is beyond our best expectations. Prior to the recycling system coming online, our peak detergent usage was approximately 98 gallons per day. Our auto-

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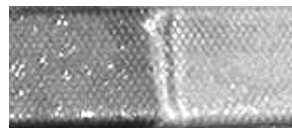
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matic detergent makeup system now dispenses less than three gallons per day to maintain exactly the same detergent concentration. Needless to say, the cost savings produced by the system have skyrocketed. Based on the aforementioned \$800,000 capital cost of the system, we expect a pay-back period of less than three years based on detergent savings alone. Thereafter, the system will be saving us over \$300,000 annually.

Third, we have dramatically reduced our fresh water consumption for the same reason we have reduced wastewater discharge, we are reusing our wastewater. While water is relatively inexpensive in our area, we still feel that it makes sense to conserve this resource.

Fourth, we wanted to extend the bath life on our washers by regularly overflowing the tanks with clean water. Without the recycling system, we were unable to do this with the wash tank because, obviously, the city water supply contained no detergent and would rapidly dilute our wash tank. The recycling system allows us

to overflow all the tanks and has resulted in a 100 percent increase in the bath life (from a one week cycle, to a two week cycle). We had actually hoped to get a much longer bath life than two weeks, but that benefit did not materialize so we just have to be happy with the extra seven days that we did get.

Finally, we are saving energy costs by reusing our hot wastewater instead of constantly bringing in cold fresh water. Our washers operate at 150°F using electric heating elements. By insulating every part of the recycling system that we could think of (including the holding tanks), we receive the dirty water at the recycling plant at about 120°F. The water is then brought up to 140°F by the ultrafilter heaters, and is received back in the main plant by the washers at about 130°F.

Our system works better than we ever thought it could. There are several keys to this success:

- Very consistent wastewater mixture. Because we take great pains to ensure that no unapproved chemicals get into the washers, we can be

reasonably sure that the ultrafilters will be processing the same liquid, day after day.

- Plenty of time spent on pilot testing. The importance of finding out how your particular waste stream performs in an ultrafilter in real life (versus bench tests) cannot be stressed enough.
- A detergent that is truly designed to be recyclable. Had we not come upon the Evercycle 2000 detergent, it is possible that this project would have withered on the vine.

Although the term "closed loop" has become somewhat cliché, we believe that our system demonstrates the technological and economic feasibility of just such a concept. ■

#### About the Author

Mark Carroll is the Senior Environmental Engineer at his facility with responsibility for all environmental compliance issues and select facility engineering matters. He holds a Master of Science degree from Purdue University and a Class IV wastewater treatment license. Mr. Carroll may be reached at (870) 793-1892.

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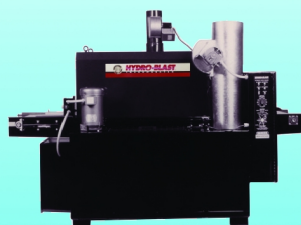
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