An Investigation in Rinse Water Sustainability

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ABSTRACT
Today's society is more focused on sustainability and recycling than ever before, which is good for our planet. Manufacturing Class III hardware has cleaning requirements, almost always involving high-purity de-ionized (DI) rinse water. It's often been said, "You're only as clean as your final rinse," as incomplete rinsing can lead to decreased device reliability, lifespan, lower yields, rework, and other manufacturing costs. Therefore, ensuring high-purity water is available in sufficient quantity is critical for ensuring reliable production.

Many small footprint, or "batch" style cleaners have closed-loop rinse water options that recycle rinse water. However, high production volume in-line washers can consume over 2,000 gallons of DI water per 8-hour operating shift. This can be a challenge as water becomes a more precious resource, especially in certain parts of the world.

This paper investigates options for improving sustainability in Class II and III manufacturing environments by studying options for reusing and/or recycling rinse water.

Key words: Cleaning, Reliability, Rinse Water, Sustainability, Recycling.

INTRODUCTION
The June 2024 edition of Chemical & Engineering News contained an article under the Climate Change heading, "Drought in Mexico shuts chemical plants". It reported authorities diverting water from industrial consumers to local communities due to Mexico's worst drought in more than a decade. Whether due to environmental conditions, such as drought, or regulations on plant effluent water, recycling rinse water and minimizing disposal is an increasingly common topic.

Modern electronic assemblies often have very complex and densely packed circuits that are needed to achieve the required functionality and footprint requirements. Aqueous cleaning agents are formulated with a low surface tension to meet that challenge. For maximum reliability, all traces of the wash solution and any dissolved contamination should be completely rinsed away with high-quality deionized (DI) water.

IPC-AC-62A, Chapter 10.6, states: "Good quality deionized water is the preferred rinse medium…Water in the 1 to 5 megohm-cm region will be satisfactory for most operations. The temperatures of the rinse water should be as high as possible, but comparative with parts and process."

Pure DI water has a surface tension of approximately 72 dynes/cm, more than double that of common engineered aqueous cleaning agents. A key impact of that is that complete rinsing often requires a greater volume of fluid (water) than washing as the higher surface tension water doesn't wet out on the surface or penetrate under low-standoff components as quickly.

The conveyorized design of in-line aqueous cleaning machines makes them ideally suited for medium and high-volume production applications. Figure 1 depicts a typical configuration with a wash → chemical isolation (CI) → rinse → final rinse (FR) → drying stages.

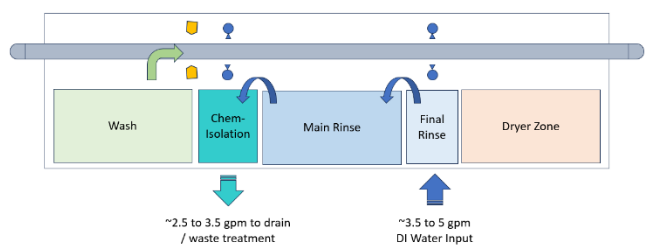

Figure 1. Typical In-Line Cleaner Stages

The highest quality DI water is fed to the final rinse stage, the last water the assemblies are exposed to before drying. Depending on the machine configuration (such as conveyor belt width, number of spray nozzles, etc.), the DI water input to the cleaner is generally in the 3.5 to 5 gallons per minute (gpm) range. Water from the final rinse cascades into the main rinse zone, which then supplies the chemical isolation zone at a rate of 2.5 to 3.5 gpm. The 1 to 1.5 gpm net difference between what's fed into the final rinse and leaving chemical isolation is lost to a combination of liquid going into the dryer and fine mist from the high-pressure sprays being drawn up the exhaust stack.

The primary function of the chemical isolation zone is to remove the bulk of the wash solution that is being carried out of the wash zone on the horizontal conveyor, the assemblies being cleaned, and any baskets or fixtures that may be used and add surface area to hold fluid. This lost cleaning solution is often called “drag-out” because the traveling work carries it to the rinse. Removing most of the drag-out wash solution in the chemical isolation zone reduces contamination in the main rinse, preserving higher water quality and reducing the chance of foaming or re-contamination.

While the main rinse zone has multiple spray bars and high-pressure pump(s), the chemical isolation step provides a coarse rinse, effectively removing the bulk aqueous cleaning solution with just one top and one bottom spray bar. Figure 1 depicts them as blue circles. Some in-line washers will also have air knives positioned at the exit of the wash zone to conserve cleaning solution and reduce drag-out going over into chemical isolation. Figure 1 shows them in orange. Air knives reduce the amount of wash solution (chemical & dissolved contamination) that needs to be rinsed off; however, they do not directly affect the amount of water exiting chemical isolation.

When possible, based on location and local regulations, the chemical isolation water is often sent directly to drain. Larger facilities may also have an in-house water treatment system. However, as plant emission regulations evolve, even dilute chemical streams may be challenging to send to drain, and not every facility has an in-house treatment plant.

The question of “Can I close the loop?” or have a zero discharge aqueous in-inline cleaning process is coming up more frequently now.

A CI flow rate of 2.5 gpm will fill a 55-gallon waste drum in just over 20 minutes. That’s 1,200 gallons for 8 hours of operation, which at 8.34 lbs/gal is 5 tons of water per shift. Directly capturing the CI water is not practical, nor will it be economical for sustained operation.

This paper investigates methods to recycle the chemical isolation water.

METHODOLOGY

Part 1: Determining CI Water Composition

To better understand and size potential water purification or recycling options, we began by studying the flow rate and composition of the chemical isolation stream.
Numerous factors will influence the volume and/or concentration of the CI discharge stream. This study evaluated the following operating conditions as a baseline:

Wash: 15% leading aqueous cleaning agent @ 150°F
Belt Speed: 1.0 feet per minute (fpm)
CI Rinse flow: 2.4 gpm

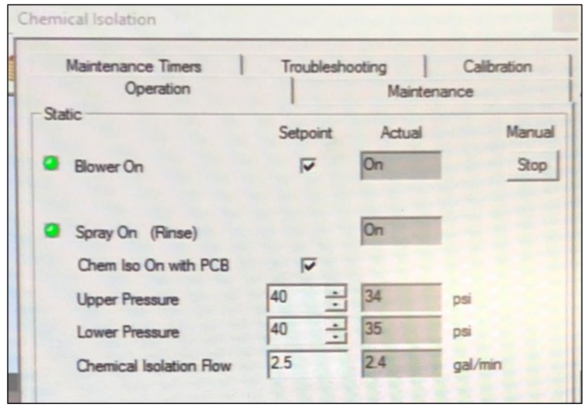

Image 1. Chemical isolation configuration

Image 1 shows the operating conditions of the chemical isolation section for Phase 1 of the study. This particular machine displays the actual water flow at 2.4 gpm. Another way to quickly estimate water flow through a spray manifold is to count the number of individual nozzles and then use a reference table from the nozzle manufacturer that provides flow rate data at given pressures.

Table 1. Spray nozzle reference example

Equiv. Orifice Diameter (in)	CAPACITY AT VARIOUS PRESSURES (USGPM)								
	5 psi	10 psi	20 psi	30 psi	40 psi	60 psi	80 psi	100 psi	200 psi
0.043	0.11	0.15	0.21	0.26	0.30	0.37	0.42	0.47	0.67

The 35 psi CI spray pressure falls halfway between the 30 and 40 psi values in the table, so the flow rate can be estimated at 0.28 gpm per nozzle.

Taking the total number of nozzles in the upper and lower spray bars, nine (9), and multiplying by the 0.28 gpm/nozzle results in a total estimated flow of 2.52 gpm.

For collecting the CI water sample(s), we didn’t want to take one reading that would represent a snapshot point in time; rather, the goal was to measure the chemical concentration of the CI rinse water collected over a period of time.

To accomplish this, a set of new 275-gallon totes were used to collect all of the rinse water exiting the chemical isolation section for the specific test condition. With an estimated flow rate of 2.5 gpm, each tote should collect about 110 minutes of CI water flow.

During that time, the in-line washer was operated at a continuous steady-state like condition. As noted in Table 2, the conveyor belt was loaded with an array of cards and baskets to simulate real-world drag-out of the wash solution into the chemical isolation stage.

Table 2. CI tote collection table

Chem-ISO Sample Collection				
Sample	*Air Knives*	*Fill Time (min)*	*Approx Flow (gpm)*	*Simulated Production*
Tote 1	On	62	~4.3	Baskets + Long Cards
Tote 2	On	105	~2.6	Baskets & Typical Cards
Tote 3	OFF	96	~2.8	Baskets & Typical Cards

The first tote filled in just 62 minutes, much faster than anticipated. (~270 gallons / 62 min > 4.3 gpm.) The in-line was still reporting a CI feed rate of 2.4 to 2.5 gpm, which, as calculated above, is similar to the theoretical flow rate at the known operating pressure.

Further investigation revealed that some of the cards used to simulate the drag-out were sufficiently long to bridge between the main rinse and the chemical isolation zone.

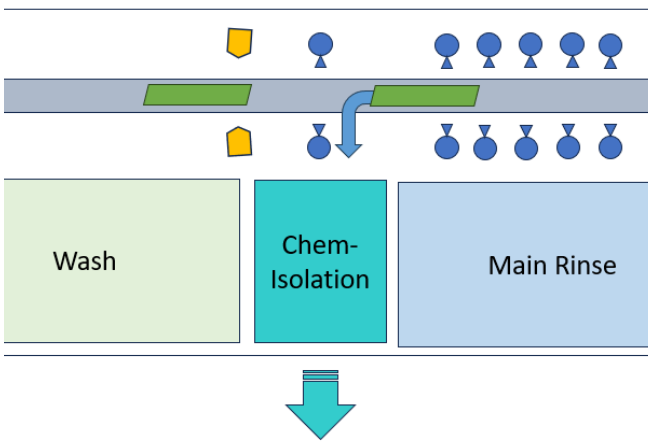

Figure 2. Example of rinse water bridging

This solved the mystery of extra water and was an excellent reminder of the importance of matching the machine configuration to the assemblies being cleaned. This also drew more attention to the air knives at the exit end of the wash zone and their effectiveness in preventing excess drag-out from the wash into the chemical isolation zone.

Tote #2 was filled with the in-line washer operating under the same conditions but without processing the long assemblies. Drag-out was simulated by loading the belt with typical-sized assemblies and some baskets. There was at least 4" of spacing between cards to eliminate unwanted bridging.

As noted in Table 2, the 2nd tote filled in 105 minutes of cleaner operation, which aligns with the expected time.

To study the effectiveness of the air knives at the exit of the wash section, a 3rd tote of CI water was collected. The only operational change from the Tote 2 test was that the air knives were turned off. All other parameters, including the cards and baskets used to simulate production drag-out, were kept the same.

Tote #3 filled in 96 minutes for an average fill rate of 2.8 gallons per minute. Compared with filling Tote 2, using the air knives at the exit of the wash solution reduced the CI flow rate by approximately 0.2 gpm.

The composition of each of the three (3) totes of collected chemical isolation water was then analyzed by:

- Refractive Index
- Conductivity
- COD (Chemical Oxygen Demand)
- TOC (Total Organic Carbon)

COD is commonly used to estimate the potential impact of wastewater or effluent on the environment, particularly for regulatory purposes since it reflects the oxygen-depleting potential of organic and inorganic materials.

TOC is used when a more specific measure of organic carbon is required, such as in water quality assessments, drinking water treatment, and environmental monitoring.

Those values were then used to calculate an approximate concentration of wash chemistry in the CI water stream.

Table 3. CI analytical results

	Tote #1	Tote #2	Tote #3
Refractive Index:	0.0°Bx	0.0°Bx	0.2°Bx
Concentration by RI:	0.00%	0.00%	0.30%
Conductivity:	17.3 µS/cm	26.9 µS/cm	84.3 µS/cm
COD:	390 mg/L	800 mg/L	6730 mg/L
Concentration by COD:	0.02%	0.04%	0.32%
TOC:	195.1 mg/L	364.4 mg/L	1002.8 mg/L
Concentration by TOC:	0.05%	0.09%	0.24%

Refractive index doesn't provide the resolution to measure very dilute aqueous solutions accurately. Both Tote #1 and Tote #2 measured 0.0 Brix, so more accurate means of analysis were necessary. Tote #3, with the air knives turned off, measured 0.2 Brix or about 0.3% wash chemistry.

The conductivity, COD, and TOC values for Tote #1 all further confirmed the excessive dilution due to rinse water bridging over into the CI. Tote #1 filled almost twice as fast as Tote #2, and the values were each about half.

Tote #2, which filled at the correct expected rate and represented normal in-line washer operation (air knives on), and typical simulated production was therefore used as the baseline for Phase 2 of the study.

Part 2: Carbon & DI Resin Adsorption

Activated carbon and DI resin are frequently used to purify water streams by stripping out organic and ionic contaminants. For example, most new refrigerators today have an activated carbon filter to remove trace organic content from drinking water. Small filters containing DI resin that attach to the end of a garden hose are available to provide a spot-free rinse when washing your car.

Common details and uses of activated carbon and DI resins (anion resin and cation resin) are well-published in literature (CRN, 2023). A few key highlights:

- *Activated carbon* effectively adsorbs organic contaminants in a water stream until saturated. However, once saturated, the carbon needs to be disposed of and replaced. It is a consumable filtration item. Note: there is no simple in-line meter or gauge that indicates when the carbon is saturated.

- *DI Resin* media uses a combination of anion and cation resins to adsorb the corresponding ions from a water stream. The media health is easily monitored by measuring the output water's resistivity (or conductivity). DI resins are also sensitive to organic fouling, so the activated carbon bottle is normally placed ahead of the DI Resin for best performance. While the DI resin can be regenerated, the bottle supplier normally does it off-site. Therefore, for the end user, it is also a consumable item that needs to be regularly monitored and replaced.

High-quality final rinse water (~18 MΩ-cm) is often made by sending tap water through a series of carbon and DI resin bottles. The incoming tap water is generally low enough in both organic and ionic contamination that the consumable media lasts for months.

In batch-style cleaning systems, the rinse water volume is small, allowing the systems to be effectively closed-looped by recirculating through on-board carbon and DI resin bottles. The output water is monitored by conductivity, and when it declines below the user's threshold, both the carbon and resin media are replaced.

The challenge with close looping most in-line aqueous cleaning machines is simply the high volume of water that must be treated. Using our example of ~2.5 gpm, that works out to be 10,000 lb of water in an 8-hour period. Even at a 0.05% dilution, that equates to 5 lb of chemistry that needs to be adsorbed. Potentially per shift. Using the more conservative 0.1% (TOC) based value, the amount of contamination to adsorb doubles to approximately 10 lb.

In an attempt to quantify how much wash chemistry would be adsorbed by each the activated carbon and DI resin, the following experiments were performed.

Using the same aqueous cleaning agent as in Part 1, a set of 400-gram solutions were carefully prepared at a 15% concentration (60 g agent + 340 g DI water).

High-quality activated carbon and "mixed" DI resin (50% anion, 50% cation) used for water purification were obtained. Additionally, two small 304 stainless steel tea strainers were purchased, one filled with activated carbon (22.1g) and the other with the DI resin media (27.6 g).

The objective was to use a quantity of each carbon and DI resin such that they would be saturated by immersing in the known concentration of wash solution, which could then be analyzed afterward. A sonic velocity (SV) instrument was used to monitor the adsorption rate and determine the saturation point.

Image 2. Solution on a stir plate, tea strainer

The cleaning agent used in this study is a typical multi-phase material that will form a measurable solvent layer at 15% concentration. Therefore, a heated stir plate was used to ensure the solution was fully mixed and circulating through the tea strainer when immersed.

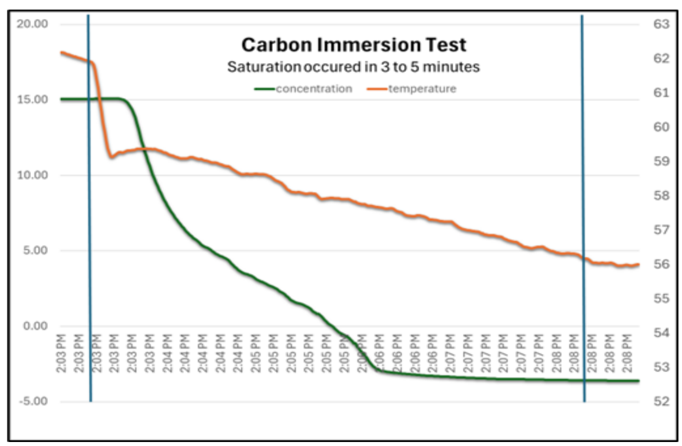

Figure 3. Rate of carbon adsorption

The tea strainer filled with carbon was added to the stirred beaker at 2:03pm. The temperature dropped rapidly when the SV sensor was briefly removed, the strainer was added, and then the SV sensor returned to monitor the solution. Over the next 3 minutes, the displayed concentration declined steadily as the activated carbon adsorbed material from the agitated solution. The rate of change stopped almost as quickly as it started, and then the displayed concentration remained constant. Indicating saturation occurred within only 3 to 5 minutes of exposure.

Note: Modern cleaning agents are highly engineered formulas with a blend of ingredients – some organic, some ionic in nature. The displayed final concentration is not an accurate value because the activated carbon only adsorbs some elements of the cleaning media. However, the rate of change is a good gauge of the activity or rate of adsorption.

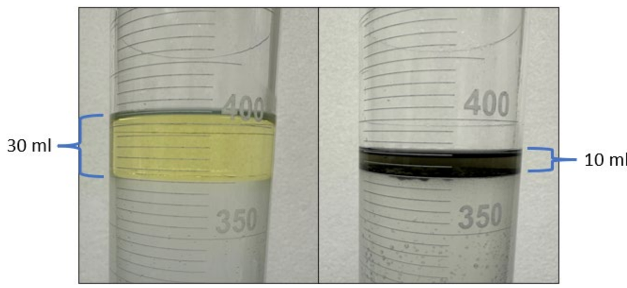

Image 3. Solvent layer before & after carbon adsorption

Although the carbon was pre-rinsed with water until clear, the solvent layer was black in color after exposure to the low surface tension cleaning fluid.

The total fluid level declined by about 20 ml, starting at 405 ml before to 385 ml after the test. While some liquid may have remained trapped in the tea strainer, not easily draining back to the beaker. Notably, the solvent layer was also reduced by approximately 20 ml. The aqueous phase/solvent phase interface is very near the 375 ml line in the before and after images.

The activated carbon effectively adsorbed the organic material up to its saturation point in just a few minutes.

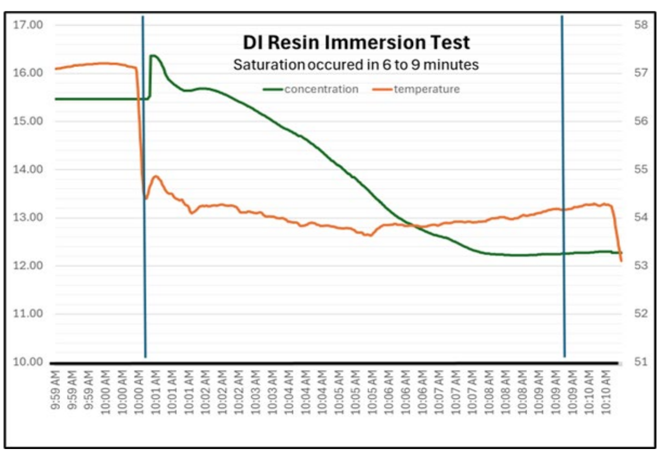

Figure 4. Rate of DI Resin Adsorption

Repeating the experiment with the stainless steel tea strainer filled with fresh DI resin yielded similarly interesting results. The adsorption rate was perhaps slightly slower, taking 6 to 9 minutes.

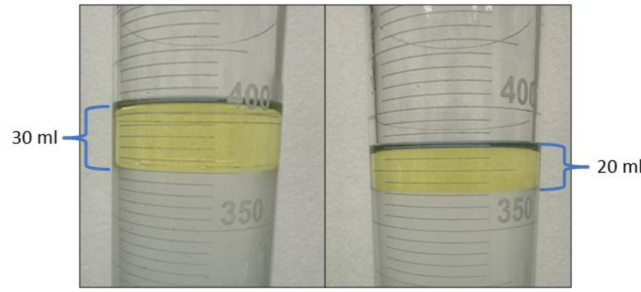

Image 4. Solvent layer before & after resin adsorption

The total fluid volume decreased more in this test, dropping approximately 20 mils to a final volume of 385 ml. The tea strainer filled with activated carbon test was pre-rinsed to remove as many black carbon particles as possible, whereas the strainer filled with DI resin did not need pre-rinsing. Thus, leaving air pockets between the resin beads rather than filling them with water. Additionally, some wash liquid may have been trapped in the tea strainer after immersion, not easily draining back into the beaker.

The mass of the wet tea strainer and resin immediately after immersion and drip drying was 59.2 g. After several days of sitting at ambient room conditions, the mass was 38.0 g, indicating that 21.2 grams of liquid had dried out from the resin. A density of near 1 helps explain the ~20 ml fluid loss shown in Image 4.

The volume of the solvent layer was reduced by approximately 10 ml, indicating some of the organic phase was pulled out by the immersed strainer test. That was more than expected as the resin media is not designed to strip out the organic phase but rather remove ionic contaminants.

The remaining solutions were further analyzed by conductivity and COD; the results are below in Table 4.

Table 4. Carbon & resin adsorption analysis

	15% Cleaning Agent	Carbon Test	Resin Test
Conductivity:	3610 µS/cm	4010 µS/cm	2481 µS/cm
		16.7%	10.3%
COD:	340,500 mg/L	247,000 mg/L	266,500 mg/L
Concentration by COD:		10.9%	11.7%

The results are interesting and demonstrate why carbon and DI resin should always be used in combination with each other, as they have different functions.

By COD, the activated carbon reduced the chemical concentration by approximately 4%. The adsorption capacity of the carbon then works out to be ~75% on a mass-to-mass basis. The reduction of 4.1% / 15.0% * 60 g initial chemistry equals 16.4 g removed by 22.1 g of carbon or 74.2% capacity. This value is significantly higher than expected based on published organic absorption rates (Gabelman, 2017). More work can be done to verify the results.

Activated carbon does not remove ionic species, which was confirmed by the conductivity value, which increased slightly compared to the virgin 15% solution. A quick test showed that adding activated carbon to DI water will increase the water's conductivity. There is a potential interaction between the carbon adsorption and the wash solution. The key takeaway is that activated carbon removes organic contaminants, and any ionic contaminants must be addressed separately.

DI resin is designed to remove ions from water, it is not intended to remove organic compounds. It works by exchanging ions (such as calcium, magnesium, sodium, and chloride) with hydrogen and hydroxyl ions, effectively removing charged particles from the water and thus purifying it.

The resin media is prone to organic fouling, which is another reason why carbon beds should be placed in front of the resin. The change in conductivity will be used to determine its adsorption efficiency rather than chemical oxygen demand (COD), which is more applicable to organic materials.

The Conductivity decreased by a value equal to a 4.7% lower chemical concentration. Applying a similar calculation, 4.7% / 15.0%* 60 g of initial chemistry equals 18.8 g being removed by 27.6 g of DI resin or a 68% effective loading capacity.

Even using these high efficiency loading factors, stripping out 5 lb of wash solution per shift, may require:

- 6.7 lb of activated carbon (75% efficiency)
- 7.4 lb of DI resin (68% efficiency)

A recent user trying to close-loop their rinse water process with carbon and DI resin alone quickly discovered they needed to change out the media bottles every 2 to 3 days. It was both impractical and extremely costly from both production and financial perspectives.

Part 3: Reverse Osmosis Study

A reverse osmosis (RO) system is a water filtration process that removes contaminants by pushing water through a special membrane that only lets water molecules pass through. The semipermeable membrane(s) have tiny pores that are intended to allow only water molecules to pass through and block larger molecules like salts, minerals, and other contaminants.

Figure 5 shows the basic concept of a feed stream of water being sent to the RO system; the semipermeable membranes then separate it into the permeate (clean water) and the reject (dirty water) streams based on molecule size.

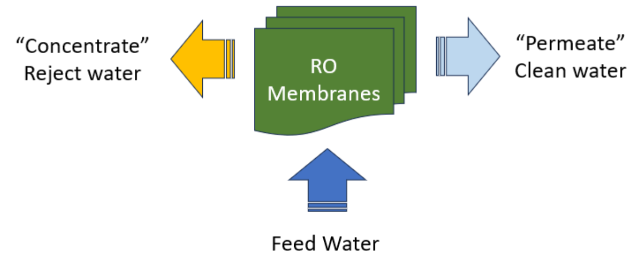

Figure 5. Basic RO concept

RO systems are used in a wide array of water purification applications. One common use is to pre-treat tap water as a lower-cost method for making high volumes of DI water. Tap water is the feed supply to the RO system, where the membranes remove most organic and ionic contaminants. The clean permeate stream is then "polished" by traditional activated carbon and DI resin beds to achieve the desired 18 MΩ-cm purity level.

The RO membranes are not adsorption-based consumable items like carbon and DI resin. However, they do need periodic cleaning, depending on use, which may be once a year or every 6 months.

RO systems can also be applied to recycle chemical isolation rinse water. Figure 6 depicts a typical configuration where the RO water is sent to a concentrate holding tank. This tank provides a buffer volume so that the RO can run at any time and does not need to be matched to the in-line cleaner's operation or the chemical isolation flow rate. Water from the concentrate holding tank is then fed to the RO system, where the membranes separate it into the clean permeate stream, which goes to a permeate holding tank. The concentrate reject side of the RO membrane is fed back to the concentrate holding tank.

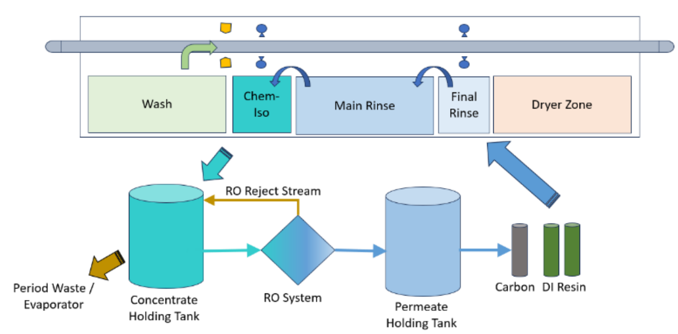

Figure 6. RO for chemical isolation

The water in the permeate holding tank is not quite DI water quality. It needs to go through traditional carbon and DI resin media, which polish it for use as final rinse water.

A periodic bleed from the concentrate holding tank will be necessary to remove concentrated contamination from the system. Some locations may send water to an evaporator for further wastewater volume reduction.

The 3rd part of this study into rinse water sustainability was to pass the nearly 275 gallons of Tote 2 chemical isolation rinse water through an RO system and measure the output water quality. Those results can then be compared with using carbon and DI resin alone.

Three (3) new Filmtec SW30-4040 RO membranes were installed in a small standard RO system. BW series or brackish water membranes are often used for purifying tap water; however, for this application, SW series or sea water membranes were chosen because they are designed for higher-concentration feed streams.

The membrane pressure operated at 180 – 190 psi, with a ~4.5 gpm feed rate. The concentrate (reject) had a flow rate of ~ 3.0 gpm, and the permeate (clean) side flow was ~1.5 gpm.

Table 5. RO Test 1 output analysis

	Tote #2 (Feed)	Test 1, Permeate	Test 1, Reject
Refractive Index:	0.0°Bx	0.0°Bx	0.1°Bx
Conductivity:	26.95 µS/cm	2.48 µS/cm	37.3 µS/cm
COD:	800 mg/L	63.5 mg/L	465 mg/L
TOC:	364.4 mg/L	<30 mg/L	627.3 mg/L

The clean permeate stream showed significantly reduced contamination levels:

- Conductivity by >10x
- COD by >12x
- TOC by >12x (below detection level)

The rejected concentrate stream values all increased. The refractive index began to measure at 0.1 Brix.

Figure 6 shows the RO reject stream returning to the concentrate tank, which would eventually be sent back through the RO system for a 2nd pass. Therefore Test 2, used the Test 1 concentrate as the feed supply, sending it back through the RO system for another pass.

Table 6. RO Test 2 output analysis

	Test 1, Reject	Test 2, Permeate	Test 2, Reject
Refractive Index:	0.1°Bx	0.0°Bx	0.1°Bx
Conductivity:	37.3 µS/cm	3.37 µS/cm	50.3 µS/cm
COD:	465 mg/L	44 mg/L	885 mg/L
TOC:	627.3 mg/L	<30 mg/L	642.8 mg/L

Again, contamination in the permeate stream was significantly reduced, and the concentrate stream increased.

For a benchmark comparison, a tote of tap water was also run through the RO system to compare how the permeate stream from recycling chemical isolation water compares to using an RO pre-treating tap water for making DI.

Table 7. RO for tap water analysis

	Tap Water (Feed)	Tap Water (Permeate)	Test 1, Permeate
Refractive Index:	0.0°Bx	0.0°Bx	0.0°Bx
Conductivity:	248.4 µS/cm	1.12 µS/cm	2.48 µS/cm
COD:	37 mg/L	40.5 mg/L	63.5 mg/L
TOC:	<30 mg/L	<30 mg/L	<30 mg/L

The RO was very effective in reducing the conductivity of tap water. Both the COD and TOC values in the fresh tap water were low; the analysis didn't show much change near the edge of the detection limits.

The permeate water quality from the CI water compares favorably with the RO output of tap water. It should be easily polished by activated carbon and DI resin to complete the recycling process.

CONCLUSIONS

This paper is intended to be a guide to understanding key factors when evaluating methods to recycle the chemical isolation water from an in-line aqueous cleaning process. Every installation or process will be unique based on the equipment, the cleaning agent, the size and quantity of assemblies being washed, etc.

Measuring the concentration and flow of the wastewater stream is an important first step. The volume may be displayed on the machine or easily estimated based on nozzle type and spray pressure. Your cleaning agent supplier or 3rd party labs can help measure the contamination levels.

Directly disposing of water that may be 99.9 to 99.95% pure will be costly due to the volume of water generated. As water becomes more precious and more restrictive disposal or emission regulations come into play, recycling the rinse water will become a more common need. Having an air knife at the exit end of the wash section can make a 2-3x reduction in the volume of wash solution carried over into the chemical isolation. This significantly affects the overall amount of contamination that must be removed if the rinse water is recycled, and it reduces total wash chemical consumption.

Activated carbon and DI resin are both highly effective at adsorbing their respective organic and ionic contaminants. That occurs very quickly right up to the point of saturation, beyond which the consumable media is ineffective and needs to be replaced/regenerated.

Incorporating an RO system can be a very effective option when recycling rinse water and greatly reduce the amount of carbon and DI resin required, possibly by a factor of 10 or more.

Future work will include further understanding the adsorption potential of carbon and DI resin media across different cleaning agent types.

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