

APPLICATION GUIDE

Cleaning Methods for Fouled Ion Exchange Resins

This Application Guide presents step-by-step procedures for using chemicals to clean fouled or contaminated ion exchange resins to extend resin life and improve system performance. The paper focuses specifically on fouling by bacteria and algae, iron and manganese, organics and oil.

CLEANING METHODS FOR FOULED ION EXCHANGE RESINS

This Application Guide presents procedures for cleaning fouled or contaminated ion exchange resins for improved system performance. For more detailed information, visit www.purolite.com or contact the closest Purolite regional office listed on the back cover.

INTRODUCTION

Purolite is a leading manufacturer of ion exchange, catalyst, adsorbent and specialty resins. With global headquarters in the United States, Purolite is the only company that focuses 100% of its resources on the development and production of resin technology.

Responding to the needs of our customers, Purolite has built the largest technical sales force in the industry, the widest variety of products and five strategically located Research and Development groups. Our ISO 9001 certified manufacturing facilities in the U.S.A, Romania and China combined with more than 40 sales offices in 30 countries ensure complete worldwide coverage.



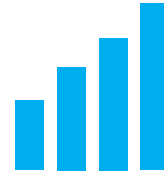
PREMIER PRODUCTS

The quality and consistency of our products is fundamental to our performance. Throughout all Purolite plants, production is carefully controlled to ensure that our products meet the most stringent criteria, regardless of where they are produced.



RELIABLE SERVICE

We are technical experts and problem solvers. Reliable and knowledgeable, we understand the urgency required to keep businesses operating smoothly. Purolite employs the largest technical sales organization in the industry.



INNOVATIVE SOLUTIONS

Our continued investment in research & development means we are always perfecting and discovering innovative uses for ion exchange resins and adsorbents. We strive to make the impossible possible.

Most types of ion exchange resins can become polluted or contaminated with suspended solids. Precipitations can occur as a result of changes in pH, and/or concentration of relatively insoluble salts. Adsorption or ion exchange of other species that are not easily removed by the normal regeneration procedures can also cause gradual accumulation and fouling of the resin. Regular cleaning treatment can prevent accumulation of fouling and extend resin life.

Increased quantities of regenerant, increased frequency of regeneration and elevated temperature of regeneration can reduce fouling by preventing contaminants from gaining a permanent hold.

It is good operating practice to ensure that all resin in the ion exchange units is regularly contacted with sufficient regenerant and is subject to a regular cleaning procedure.

Causes of deteriorating performance in an ion exchange plant

Despite precautions taken, the following conditions can cause deterioration in plant performance.

- Loss of resin from the operating units.
- Change in feed analysis. If the concentration of ions to be removed is increased, throughput will be reduced proportionately. It is good practice to check the feed analysis regularly and make adjustments to plant operation to accommodate any change.
- Faults in the operating process. Incorrect conditions of regeneration, failure to operate the plant according to instructions, faulty backwash procedures, and inconsistent rinse volumes and regenerating concentrations are relatively easy to check to ensure all functions are operating correctly.
- Malfunction of the plant engineering hardware. This is the most difficult condition to diagnose.
- Resin pollution or degradation.

If bed levels, feed analysis, operating processes and engineering hardware check out satisfactorily, a sample of resin should then be examined.

Evaluating resin through sampling

Evaluation of resin will depend upon how the sample is taken. It is preferable to ensure that the sample used for testing is representative of the bulk quantity of the bed. If this is not desirable or possible, the method used to obtain the sample should be supplied to the analyst.

To obtain a representative sample of the resin contents of a mixed bed or other unit fitted with air mixing, the resin should be mixed for five minutes and the sample should be taken after the mixing process. Where there is no mixing facility, beds are generally in the classified state as a result of regular backwash operation. Depending on the mode of regeneration and service, pollution of a surface sample can vary significantly and both the content of pollution and resin grading can be unrepresentative. Because of this, it is necessary to obtain multiple samples at different bed depths and remix, or obtain a core sample and mix before submitting the sample for analysis.

Various procedures can be used to clean fouled resins depending upon the nature of the foulant. This document covers treatment procedures for the following:

- Fouling by bacteria and algae
- Fouling by iron and manganese
- Fouling by organic species
- Fouling by oil

Disinfection treatment procedures for bacteria and algae

Under certain conditions, resin can become fouled either by bacteria or algae when contaminated water sources are fed to ion exchange systems. When contamination of resin beds is observed, consider the following treatments.

Peracetic acid

Peracetic acid, a derivative of hydrogen peroxide, is a good treatment against a wide variety of microbes. Research has shown that peracetic acid will be used to an ever-increasing degree in the field of medicine due to its anti-bacterial, fungicidal, sporicidal and anti-viral action.

Work done by the Degussa Technical Applications Department in conjunction with Chemiewerk Homburg AG determined that peracetic acid is suitable as a disinfectant for deionizers because of its wide spectrum of attack. Using a 0.1% peracetic

acid solution in water with a reaction time of one hour, a slime and mold concentration of $10^4 - 10^5$ /mL was reduced to almost zero. The short rinsing time after using peracetic acid is of importance (typically about 45 minutes or 10 – 15 BV).

Experiments have shown that in addition to excellent disinfection action, peracetic acid has minimal effect on the ion-exchange properties of cation or anion resins.

If peracetic acid is used as a disinfectant, the following procedure should be used for both cation and anion resin.

- Ensure anion resins are fully exhausted as peracetic acid performs best at a pH < 8.
- Make up 1 bed volume (BV) * of peracetic acid solution containing 0.1% peracetic acid.
- Inject 1 BV of disinfectant at a flow rate of 5 BV/h, with displacement discharged to a drain approved for chemical waste disposal.
- When all the peracetic acid has been injected, close all valves and retain the disinfectant for at least one hour to soak the resin and pipe work.
- Carry out a displacement rinse using raw water for at least 60 minutes at 5 BV/h, followed by a fast flush for 30 minutes.
- Regenerate the resin once and return the unit to service.

Sodium hypochlorite

Sodium hypochlorite is widely available in the form of small carboys and containers. For resin sterilization, a 0.1% available chlorine solution should be used. This is obtained by diluting the commercially available hypochlorite.

To prepare the treatment, adhere to the following procedures.

- Regenerate the column with brine before treatment to convert all resin to the exhausted form (a double or triple regeneration is often required.) Note that cation resin will produce chlorine gas if not properly exhausted before treatment.
- The minimum volume of solution required to treat the bed is 3 BV (i.e. 3 times the resin volume installed in the unit).

- Pass the first bed volume through the bed at normal regeneration flow rate, or approximately 4 BV/h.
- Retain a portion of the second bed volume in the bed for no more than 2 hours.
- Pass the third bed volume through the bed at approximately 4 BV/h.
- Displace the sodium hypochlorite at a rate of approximately 4 BV/h with softened water, then rinse thoroughly to remove any trace of sodium hypochlorite. At least 8 – 10 bed volumes are required.
- Triple regenerate the resin before returning to service.

Note that this form of treatment may slightly break down the cross-linked matrix of the resin. As such, frequent treatments are not advised.

The procedure is also not recommended for phenolic, polycondensation and chelate resins.

For anion resins, the oxidizing effect of the sodium hypochlorite is on the amine groups, and therefore disinfection in sodium hypochlorite should only be considered in extreme cases on a once-off basis.

Suitable safety precautions should always be taken when using sodium hypochlorite. Additionally, all environmental laws and regulations should be followed when discharging waste into drains. All discharge areas should be free from acids or other chemicals that may react adversely with the dilute hypochlorite discharge.

Treatments for iron and manganese fouling

Iron is present in several different forms within water. For example, in the case of un-aerated borehole water, iron can be present in the ferrous form (Fe^{++}), but on oxidation, it is converted into the ferric form (Fe^{+++}).

Iron can also be complexed with organic matter, in which case it is present as an anionic complex.

Normally, iron present in the ferric state is removed by cation resin operated in either the sodium or hydrogen forms.

In the case of hydrogen form cation resin representing the first stage of a demineralization system, the iron is removed from the water and eluted on regeneration with mineral acid. The situation is different with softening resin. Here, the ion exchange resin removes the iron from the water, but the regeneration procedure—which uses brine—does not elute the accumulated iron from the resin

* 1 BV = 1 liter per liter of resin.

during the regeneration cycle. Consequently, the iron accumulates on the resin from cycle to cycle and steadily causes progressive iron fouling.

In the case of iron being present as organic/iron complexes, the complex is present as an anion, and is removed from the solution by the anion resin.

Because anion resin is regenerated with caustic soda, the iron is retained on the resin even though organic material is substantially removed with each regeneration cycle. This accumulation of iron on the resin causes the anion resin to become iron fouled.

When iron content of water is > 0.5 ppm, it is recommended that some form of pre-treatment is used to reduce the iron level to < 0.1 ppm.

The use of sulfuric acid while iron is present in feed water can result in an accumulation of iron on the resin and cause reduction in performance. In these cases, treatment with hydrochloric acid should be considered providing the internal construction of the units and attendant pipe work are compatible with hydrochloric acid.

In cases where iron accumulates on base exchange softening resin, either hydrochloric acid or sodium dithionite treatment can be considered for treatment.

Sodium dithionite

Sodium dithionite is a powerful reducing agent. When applied to an iron fouled resin bed, it will reduce any ferric iron present to the soluble ferrous form and free the bed from iron during a normal aqueous cycle.

We recommend the following procedure for applying sodium dithionite to a resin bed.

- Add sodium dithionite to water (not the reverse) to form a 4% solution. Use caution when mixing the sodium dithionite because strong noxious fumes will be generated. Sufficient solution should be mixed so that when applied to the resin bed, there is enough to fully immerse all of the resin.
- Add the mixture to the resin bed.
- Agitate the resin so that the sodium dithionite solution is evenly distributed through the bed. Air should not be used in the agitation process as this will oxidize the sodium dithionite.
- Keep the solution in contact with the resin bed for a minimum of 3 hours. Solution can be left in contact for up to 6 hours, if possible.

- Drain and rinse thoroughly. After pressurizing the unit, rinse in a down-flow mode and then backwash for a full 30 minutes to remove any extraneous matter.
- Next, regenerate the unit in its usual manner prior to returning to service.

Because of the relative instability of sodium dithionite solution, a treatment method using a combination of sodium dithionite and sodium tripolyphosphate has been found to be even more effective than using sodium dithionite alone.

Sodium dithionite and sodium tripolyphosphate

- Mix a solution of 2% sodium dithionite and 2% sodium tripolyphosphate. The resulting solution is more stable, and retains its iron removal power for up to 16 hours.
- In instances where a preventive procedure may be considered, we recommend the addition of 1 gram of sodium dithionite to every 100 grams of sodium chloride used during the regeneration sequence.
- Take precaution to prevent the oxidation of sodium dithionite by addition immediately prior to brine injection, as well as adding to the brine solution.

Sodium dithionite will decompose under the influence of heat or moisture. For this reason, it should be kept in sealed, watertight containers and stored in a cool, dry place. Under such conditions, this material can be stored over a prolonged period with negligible loss in activity. Care should be exercised in handling sodium dithionite. On contact with water, this product decomposes rapidly forming highly flammable gases.

Sodium dithionite is classified as a flammable solid and is shipped under the appropriate caution label. Any material that is spilled should be promptly cleaned up, and the site washed with copious amounts of water. Partially used containers present a fire hazard.

When fighting a sodium dithionite fire, the burning material should be deluged with water since too little water may cause additional problems. Carbon dioxide and dry fire extinguishers are not effective as the product provides its own oxygen for combustion.

Full details of the recommended procedure for storing and handling sodium dithionite should be obtained from the chemical supplier and strictly adhered to in order to ensure full compliance with local health and safety regulations.

Hydrochloric acid

In many instances, it is not possible to treat softening resins with hydrochloric acid in situ because it is not compatible with the materials of construction of the softening unit.

However, where possible, a 6% hydrochloric acid should be used, and three bed volumes applied, retaining the middle bed volume in contact with the resin for over two hours. Warming the acid to 104°F (40°C) is also beneficial.

The resin should then be regenerated twice with 10% brine solution before it is returned to service.

Treatments for organic fouling

Anion resins are susceptible to fouling by humic and fulvic acids sometimes found in surface waters.

These organic species become trapped within the resin matrix due to their large molecular weights. Symptoms of organic fouling include long rinse requirements, poor capacity and in the case of strong base resins, higher silica leakage.

The following treatment helps return the original ion exchange properties of the resin.

- Treat the resin at the end of the normal exhaustion cycle.
- Prepare three bed volumes of 10% w/v brine solution containing 2% w/v caustic soda. Temperature of the solution should be between 95°F (35°C) and 140°F (60°C) to ensure optimal organic elution effect.
- Introduce one bed volume into the ion exchange unit at a flow rate not exceeding 2 BV/h followed by a second bed volume.
- Retain the second volume in the unit for as long as possible (minimum 4 hours). Periodically agitate the bed throughout the retention period.
- At the end of the retention period, the last bed volume of brine should be passed through the resin at a rate of 1 BV/h and the resin rinsed thoroughly with clean water until free from brine.
- Subject the resin to at least two complete regeneration cycles before returning online.

Hydrochloric acid

Occasionally, the presence of iron is detected on the anion resin. This can arise from iron/organic complexes being present in the raw feed water.

In these cases, it is advisable to treat the anion resin with 6% hydrochloric acid immediately after the

brine treatment. The procedure that should be followed is similar to that given for brining.

It is very important that all traces of hydrochloric acid are removed from the unit before introducing caustic soda regenerant, and that all parts of the unit that come into contact with the hydrochloric acid are compatible with and resistant to the acid.

Treatments for oil fouling

Oil in feed water or regeneration solutions will lead to fouling of ion exchange resins. Oil coats the surface of resin, making it difficult for ions to penetrate through the oil layer into the beads, where the majority of exchange sites are. Oil-based resin fouling results in deterioration of resin kinetics and treated water quality, as well as reduced operating capacity.

For best performance, there should be zero oil in feed solutions to the ion exchange resin bed.

Cleaning resins fouled by oil is extremely difficult. If resin is heavily fouled, it may be impossible to clean them sufficiently to make them suitable for ongoing use. The following procedure uses a low foaming, non-ionic surfactant and is recommended for lightly fouled ion exchange resins and inert polymers.

- Thoroughly backwash the fouled resin.
- Drain the unit and fill with a solution containing no more than 0.1% of surfactant. The treatment is most effective if the solution is administered at approximately 104°F (40°C). Using lower temperatures may produce considerable foaming. Note that it is important to use surfactant that does not foul resin.
- Cleaning will be more efficient if air is introduced to the resin bed as this causes agitation while the resin soaks in the surfactant solution. Agitation should continue for approximately a half an hour.
- Following this, thoroughly backwash the unit and rinse in down-flow mode until the foaming completely dissipates. Water temperature for the first part of the rinse is optimal at 104°F (40°C).
- Finally, thoroughly regenerate and rinse the resin before proceeding to the next service cycle.

Inspecting, sampling and cleaning the resin regularly can prevent the accumulation of contaminants and severity of resin fouling. This, in turn, will help optimize performance and prevent permanent degradation of the resin.

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