



Preventing Biological Growth on Ion Exchange Resins

Introduction

Ion exchange resins in demineralization applications are subjected to extreme changes in pH during regeneration, which hinders biological growth. Most biological growth problems are caused by inactivity of the resin during extended storage in the vessel or under pH neutral conditions, such as water softeners, dealkalizers or non-regenerated mixed beds in ultrapure water production. In order to minimize the potential for bio-fouling, inactive systems should be stored in a biostatic solution such as concentrated NaCl. Note that this complete exhaustion is acceptable for most demineralizer applications, but undesirable for ultrapure water applications.

The procedure to apply before plant shut-down is:

- Convert the resin to the completely exhausted form, using dilute solutions of acid for the anion resin, caustic soda for strongly acidic resins.
- Carry out a thorough backwash to remove suspended impurities.
- Apply a 15% – 25% NaCl solution to the bed, and fill the vessel so that no air is present. The concentrated salt solution will minimize biological growth.
- Perform a double or triple regeneration of the resin upon reactivation of the vessel.

Cleaning Biological Growth from Ion Exchange Resins

The detailed cleaning steps have to be adapted to each regeneration process (for example, co-flow or different types of reverse flow regeneration). Old or mechanically weak resins may suffer from osmotic (fast swelling and shrinking) and mechanical (air scrubbing) strains. It is advisable to use gradual changes in solution concentrations and to minimize the mechanical stress.

General Methods

These methods apply to systems where resins are regenerated. In cases where biological growth has occurred, an extended air scour followed by a double regeneration may be able to restore the resins to a usable condition. If this procedure is not successful, disinfection procedures can be used, but they can cause oxidative damage to the resins, so these procedures should be considered as a last resort. The next pages will cover;

1. Disinfection with peracetic acid
2. Disinfection with chlorine
3. Treatment with hot water
4. Disinfection with hydrogen peroxide (ultrapure water systems)

The present procedures apply to *clean* resins. Concentration, volume and contact time are applicable only for disinfection of the resin. If the resin is loaded with organics, for example from sugar juices, extreme care must be applied because peracetic acid will oxidize the adsorbed organics in an exothermic, possibly explosive reaction.

1. Disinfection with peracetic acid

Peracetic acid has a wide-band action for removing micro-organisms and is an effective treatment for the disinfection of both cation and anion exchange resins. To allow good disinfection without damage to the resins, it is important to control the concentration, temperature and contact time of the chemical. The resin must be converted to exhausted form prior to treatment

CAUTION

Concentrated peracetic acid is a hazardous chemical and should be handled with extreme care. Consult supplier for precautions. The ion exchange column should be vented, as gases may escape.

METHOD OF TREATMENT FOR CATION RESINS

- Clean the resin by backwashing and air scour to loosen accumulation of biofilm and remove contaminants (silt and debris).
- If the resin has iron or other metal contamination, pretreat with ~ 2 bed volumes of a 10% HCl solution.
- Put resin into exhausted form by treating with a 2 – 5% brine solution for strong acid cation resins, or with a 0.5% caustic solution in upflow direction for weak acid cation resins (take care to allow for resin swelling).
- Drain column.
- Introduce one to two bed volumes of peracetic acid at 0.2 percent concentration from the bottom of the column over 30 to 60 minutes.
- Leave in contact for one hour.
- Rinse out with 4 bed volumes DI water over a period of approximately 1 hour, until no peracetic acid is detectable in effluent.
- Apply a thorough air scour and then backwash the resin to remove dead bacteria bodies.
- Complete a double regeneration of the resin.
- Rinse thoroughly.

METHOD OF TREATMENT FOR ANION RESINS

- Clean resin by backwashing and air scour to loosen accumulation of biofilm and remove contaminants (silt and debris). If strong organic fouling is suspected, the resin should be submitted to an alkaline brine treatment (see the corresponding Tech Fact) before disinfection.
- If the resin has iron or other metal contamination, pretreat with ~1 bed volume of a 2% HCl solution followed by ~1 bed volume of a 10% HCl solution.
- Put strong base resins into exhausted form by treating with a 2 – 5% brine solution (skip if it has been pretreated with HCl), and weak base resins into regenerated form with a 2–4% caustic solution.
- Prepare peracetic acid solution of 0.2% concentration
- Introduce 1 bed volume of the solution at ambient temperature through the resin bed during a 30 – 60 minute contact time. Measure the peracetic acid in the effluent and stop when it reaches a level of around 10% of the inlet concentration.
- Rinse out with 4 bed volumes DI water over a period of approximately 1 hour, until no peracetic acid is detectable in effluent.
- Apply a thorough air scour and then backwash the resin to remove dead bacteria bodies.
- Complete a double regeneration of the resin.

2. Disinfection with chlorine

Sodium hypochlorite or bleach cleaning is a very intense treatment for sterilizing and removing organic contaminants on cation exchange resins.

CAUTION

As a result, this treatment should be carefully controlled in order to prevent possible resin damage (de-crosslinkage or de-functionalization). Note that chlorine can be explosive under certain conditions.

METHOD OF TREATMENT FOR CATION RESINS

- Apply a thorough air scour and then backwash the resin.
- If the resin has iron or other metal contamination, pretreat with ~ 2 bed volumes of a 10% HCl solution.
- Ensure that the resin is completely exhausted by treating with a 2-5% brine solution for strong acid cation resins, or with a 0.5% caustic solution in upflow direction for weak acid cation resins (take care to allow for resin swelling.), as any residual H⁺ on the resin can lead to the generation of free chlorine gas.
- Use a sodium hypochlorite solution of 0.10% concentration (1 g/L).
- Apply 5 g free Cl₂ per liter resin by passing 5 bed volumes of the NaOCl solution at ambient temperature down through the resin bed with a 30 – 45 minute contact time.
- Allow the resin to soak in the solution for 1 – 2 hours.
- Rinse out with 1 to 2 bed volumes DI water.
- For the most effective treatment, apply more solution if free Cl₂ did not reach the effluent.
- Perform a final rinse with 3 to 4 bed volumes DI water (until no Cl₂ is detectable in effluent).

METHOD OF TREATMENT FOR ANION RESINS

- Apply a thorough air scour and then backwash the resin.
- If the resin has iron or other metal contamination, pretreat with ~ 1 bed volume of a 2% HCl solution followed by ~ 1 bed volume of a 10% HCl solution. Rinse out thoroughly.
- Put strong base resins into exhausted form by treating with a 2–5% brine solution (skip if it has been pretreated with HCl), and weak base resins into regenerated form with a 2– 4% caustic solution.
- Use a sodium hypochlorite solution of 0.05% concentration (0.5 g/L).
- Apply 2g free Cl₂ per liter resin by passing 4 bed volumes of the NaOCl solution at ambient temperature through the resin bed with a 30-45 minute contact time. Measure the effluent and stop if free Cl₂ reaches a level of around 10% of the inlet concentration.
- Rinse out with 3 – 4 bed volumes DI water (until no Cl₂ is detectable in effluent).

3. Disinfection with hot water

In ultrapure resin systems, it is not desirable to exhaust the resin or to introduce ionic species, such as peracetate, Na or hypochlorite, so the procedures 1 and 2 should not be used. Methods that can be applied are to expose the resin to hot water at 80 – 90°C (175–195°F) for 2 hours or ozone treatment at a concentration < 10 µg/L (10 ppb) for up to 1 hour at 20°C (70°F).

4. Disinfection with hydrogen peroxide

An alternative is to use hydrogen peroxide. This method can also be used for regenerated resin systems.

Hydrogen peroxide is an effective treatment for sterilizing both cation and anion exchange resins. To allow good disinfection without damage to the resins, it is important to control the concentration, temperature and contact time of the chemical.

The recommended procedure is as follows:

- Prepare a hydrogen peroxide solution of 2% concentration (20 g/L).
- For cation resins, pass 1 bed volume of the solution at ambient temperature down through the resin bed (20 to 30 minutes contact time).
- For anion resins, use 0.5 bed volume of the 2% solution over 20 to 30 minutes.
- Rinse out with DI water until essentially no hydrogen peroxide is detected in the effluent (minimum 1 hour).
- If the resin is heavily contaminated, it may be necessary to repeat the treatment.

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WARNING: Oxidizing agents such as nitric acid attack organic ion exchange resins under certain conditions. This could lead to anything from slight resin degradation to a violent exothermic reaction (explosion). Before using strong oxidizing agents, consult sources knowledgeable in handling such materials.

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