

PUROLITE SERVICE BULLETIN

DISINFECTION TREATMENT PROCEDURES

IRON (AND MANGANESE) FOULING

ORGANIC FOULING

OIL FOULING

THE FOULING OF ION
EXCHANGE RESINS AND
METHODS OF CLEANING

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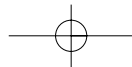
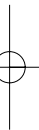
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INTRODUCTION

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. Also adsorption or ion exchange of other species which are not easily removed by the normal regeneration procedures can cause gradual accumulation and fouling of the resin.

It should be emphasised that regular cleaning treatments can prevent accumulation of fouling and therefore extend resin life.

Increased quantities of regenerant and increased frequency (and elevated temperatures) 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 regular cleaning procedure.

If, in spite of these precautions, deterioration in plant performance is experienced, the following can be the major causes:-

- 1 Loss of resin from the operating units.
- 2 Change in feed analysis. If the concentration of ions to be removed is increased, it follows that throughput will be reduced proportionally.
- 3 Faults in the operating process; incorrect conditions of regeneration; failure to operate to plant instructions.
- 4 Malfunction of the plant engineering hardware
- 5 Resin pollution or degradation.

Of the above, (4) is the hardest to diagnose. Under (2) it is good practice to check the feed analysis regularly and adjustments may then be made to plant operation to accommodate any changes. It is also relatively easy to check (3) on backwash procedures, rinse volumes and regenerant concentration to ensure all functions are being operated correctly. If (1), (2), (3) and (4) check out satisfactorily then a sample of resin should be examined.

Resin Sampling

Evaluation of resin and the conclusions will depend on how the sample is taken. It is preferable to ensure that the sample used for testing is representative of the bulk from which it was taken, or if this is not thought to be 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 5 minutes and the sample taken after mixing. 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 un-representative. Hence it is necessary to obtain multiple samples at different bed depths and remix, or obtain a core sample and mix before submission of a sample for analysis.

Procedures for Cleaning

Various procedures can be employed to clean fouled resins depending upon the nature of the foulant. These procedures are detailed in this publication.

DISINFECTION TREATMENT PROCEDURES

INTRODUCTION

Under certain conditions when contaminated water sources in particular are being fed to ion exchange systems the resins may become fouled either with bacteria or algae.

Where contamination of resin beds is observed one of the following procedures can be considered:

PERACETIC ACID

Peracetic acid, a derivative of hydrogen peroxide, displays a very wide bandwidth of attack against microbes. Research has shown that peracetic acid will be used to an ever increasing degree in the field of human medicine due to its bacterial, fungicidal, sporicidal and anti-virus action.

Because of the wide spectrum of attack peracetic acid has been shown to be very suitable as a wide bandwidth disinfectant for deionisers. (Result of work done by Degussa Technical Applications Department in conjunction with Chemiewerk Homburg). Using a peracetic acid solution of strength 0.2% (in water, with a reaction time of one hour), - a slime concentration of 10^4 - 10^5 /ml- including mould-was reduced to almost zero. The short rinsing time after using peracetic acid is of importance (typically about 45 minutes or 10-15BV).

In addition to the excellent disinfection action, peracetic acid (according to experiments) has a 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 followed for both cation and anion resin.

- Ensure anion resins are fully exhausted as peracetic acid performs best at a pH below 8.
- Make up one bed volume (BV)* of peracetic acid solution containing 0.2% peracetic acid.
- Inject 1BV of disinfectant at a flow rate of 5BV/h, with displacement discharged to drain.
- 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 5BV/h, followed by a fast flush for 30 minutes.
- Regenerate the resins once and return the unit to service.

FORMALDEHYDE

If formaldehyde is to be used as disinfectant the following procedure should be followed:-

- Make up 3BV* of formaldehyde solution containing 0.5% formaldehyde. Commercial formaldehyde (called formalin) contains 40% formaldehyde and should therefore be diluted approximately 80 times. Alternatively arrange the regenerant injection system to provide a solution of injection strength 0.5%. The ion exchange plant manufacturer will provide advice as to how this can be accomplished.
- Inject 1BV of disinfectant at a flow rate of 5BV/h discharging to drain.
- If possible, drain down the unit to a level about 50mm above the resin surface.
- Inject a second BV of disinfectant at the same rate and retain in the unit for a period of at least eight hours and preferably in the unit overnight. Formaldehyde should be detectable by smell at any drain valve.
- Flush the unit to drain using raw water until no formaldehyde is detectable at the drain by Schiff's Test.
- Regenerate the resins TWICE (double regeneration) and return the unit to service.

*1BV = 1 litre per litre of resin.

DISINFECTION TREATMENT PROCEDURES

SODIUM HYPOCHLORITE

Availability

The most convenient packout of sodium hypochlorite is in the form of small carboys/containers.

Preparation

For resin sterilisation a 1% available chlorine solution should be used. This is obtained by diluting the commercially available hypochlorite.

Treatment Procedure

- The column should be regenerated with brine before treatment in order to convert all resin to the exhausted form (a double or triple regeneration is often required). It should be ensured particularly that cation resin is fully exhausted before treatment so that there is no possibility of production of chlorine gas.
- The minimum volume of solution required to treat the bed is 3 bed volumes (i.e. 3 times the resin volume installed in the unit).
- The first bed volume should be passed through the bed at normal regeneration flow rate or approximately 4 bed volumes per hour (4BV/h).
- A portion of the second bed volume should be retained in the bed, but for no more than 2 hours.
- The third bed volume should be passed through the bed at the same rate as the first bed volume.
- The sodium hypochlorite should now be displaced at a rate of approximately 4BV/h with softened water and then rinsed thoroughly to drain to remove any trace of sodium hypochlorite. At least 8-10 bed volumes will be required.
- The resin should be triple regenerated before returning to service.

Caution

It should be noted that this form of treatment may cause slight de-crosslinking of the resin matrix and therefore frequent treatments are not advised.

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

In the case of anion resins, the oxidising effect of the sodium hypochlorite is on the amine groups and therefore disinfection in sodium hypochlorite should only be considered in extreme cases and then only on a once off basis.

Please note:

Suitable safety precautions should be taken when using sodium hypochlorite and drains into which the waste is to be discharged should be free from acids or other chemicals which may react adversely with the dilute hypochlorite discharge.

IRON (AND MANGANESE) FOULING

INTRODUCTION

Iron can be present in several different forms in water. For example in the case of un-aerated borehole water iron can be present in the ferrous state (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 either in the sodium or hydrogen forms.

In the case of hydrogen form cation resin representing the first stage of a demineralisation system the iron is removed from the water but eluted on regeneration with mineral acid. With softening resin the situation is different, as the ion exchange resin removes the iron from the water but the regeneration procedure using brine does not elute the accumulated iron from the 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 organo/iron complexes the complex is present as an anion and is therefore removed from solution by the anion resin.

Because the anion resin is being regenerated with caustic soda, whilst the organic matter may be substantially removed each regeneration cycle, the iron is retained on the resin. The accumulation of iron on the resin causes the anion resin to become iron fouled.

It is recommended that where the iron content of a water is higher than 0.5ppm some form of pre-treatment is used in order to reduce the iron level down to less than 0.1ppm.

Remedial Action

Cation Resin

When using sulphuric acid constantly while iron is present in the feedwater some accumulation of iron on the resin might take place causing a reduction in performance.

In these cases, treatment with hydrochloric acid should be considered providing the internal construction of the units and attendant pipe work make this possible.

In the case of accumulation of iron on base exchange softening resin, again either hydrochloric acid or sodium dithionite treatment may be considered.

SODIUM DITHIONITE TREATMENT

Sodium dithionite is a powerful reducing agent and when applied to an iron fouled resin bed will reduce any ferric iron present to the soluble ferrous form. Thus the bed can be freed from iron during a normal aqueous cycle.

We would recommend the following procedure for applying the sodium dithionite to a resin bed :-

- The sodium dithionite should be added to water (and not the reverse) so as to form a 4% solution.
- Caution should be shown when mixing the sodium dithionite because strong fumes of an obnoxious nature are evolved during the mixing process. Sufficient solution should be mixed so that when applied to the resin bed there is sufficient to fully immerse the whole of the resin. The resin should be agitated so that the sodium dithionite solution is evenly distributed throughout the bed.
- Air should not be used for agitation purposes as this will tend to oxidise the sodium dithionite.
- The dithionite should be allowed to remain in contact with the resin bed for a minimum of 3 hours but 6 hours if possible.
- After this period drain and rinse the unit thoroughly in a downflow fashion after pressuring the unit and then backwash for a full 30 minutes in order to remove any extraneous matter.
- After this last procedure the unit should be regenerated in the normal way prior to it being returned to service.
- Because of the relative instability of sodium dithionite solution a method utilising sodium tripolyphosphate has been found to be even more effective than using sodium dithionite alone.
- In this instance the solution should be made up of 2% sodium dithionite 2% sodium tripolyphosphate. The resultant solution retains its iron removal power for a period of up to sixteen hours because of its greater stability.
- In instances where a preventive procedure may be considered we would recommend the addition of a 1 gram of sodium dithionite to every 100 grams of sodium chloride used during the regeneration sequence.
- However, we would emphasise that the same precaution should be taken in both the preventing of oxidation of the sodium dithionite by addition immediately prior to brine injection and also in the method of addition to brine solution.

IRON (AND MANGANESE) FOULING

The general characteristics of sodium dithionite together with the precautionary procedures are as follows:-

Characteristics of sodium dithionite

Sodium dithionite will decompose under the influence of heat or moisture. For this reason sodium dithionite 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 since, on contact with water, this product decomposes quite rapidly forming gases which can ignite spontaneously.

For this reason Sodium Dithionite is classified as a flammable solid and is shipped under the appropriate caution label. Because of the above mentioned, any material which is spilled should be promptly cleaned up and the site washed with copious amounts of water. Partially used containers represent a definite fire hazard.

When fighting a sodium dithionite fire the burning material should be deluged with water since too little water may be worse than none at all. Carbon dioxide and dry fire extinguishers are valueless since the product provides for its own oxygen for combustion.

Caution

Full details of the recommended procedure for storing and handling sodium dithionite should be obtained from the supplier and these recommendations strictly adhered to so as to ensure full compliance with local Health and Safety Regulations.

HYDROCHLORIC ACID TREATMENT

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

However, where it is possible, 6% hydrochloric acid should be utilised and three bed volumes applied retaining the middle bed volume in contact with the resin for a period in excess of two hours (warming to 40°C is beneficial).

The resin should then be regenerated twice with 10% brine solution, before putting back into service.

ORGANIC FOULING

INTRODUCTION

It is well known that anion resins are susceptible to fouling by the humic and fulvic acids sometimes found in surface waters.

These organic species, because of the relatively large molecular weights, become trapped within the resin matrix (to a greater or lesser degree depending upon the resin) and specific procedures have to be employed to cause recovery of the original ion exchange properties of the resin.

The symptoms of organic fouling include long rinse requirements, poor capacity and, in the case of strong base resins, higher silica leakage.

Treatment

The most common forms of treatment involve the use of brine solution; the procedure is as follows :-

- The resin should be treated at the end of the normal exhaustion cycle.
- Three bed volumes of 10% w/v brine solution containing 2% w/v caustic soda should be prepared.
- One bed volume should be introduced into the ion exchange unit at a flow rate not exceeding 2 BV's per hour followed by a second bed volume - this second bed volume should be retained in the unit for as long as possible, but at least 4 hours. Some agitation, if possible, should be employed periodically 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 per hour and the resin thoroughly rinsed with clean water until free from brine.
- The resin should be subject to at least two complete regeneration cycles before being put back on line.

N.B. Brine at minimum 35°C should be employed or preferably as high as 60°C so as to produce a better organic elution effect.

Iron/Organic Complexes

This subject is covered to some extent in the section on iron fouling.

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

In these cases, it is advisable to consider treatment of 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 extremely important that all traces of hydrochloric acid are removed from the unit before introduction of the caustic soda regenerant.

It is important to ensure that the materials of construction are suitably resistant to hydrochloric acid.

OIL FOULING

CLEAN UP OF OIL FOULED RESIN

Introduction

The cleaning of resins fouled by oil is extremely difficult. If the resins are heavily fouled it may be impossible to clean them up sufficiently well in order to make them suitable for ongoing use. However the following procedure is recommended for lightly fouled ion exchange resins and inert polymers.

Procedure

- The procedure uses a low foaming nonionic-surfactant.
- Initially, the fouled resin should be thoroughly backwashed, the unit drained and then filled with a solution containing no more than 0.1% of surfactant.
- The treatment is more effective if the solution is administered at approximately 40°C. Using lower temperature may produce considerable foaming.
- More efficient cleaning action will result if air is introduced to the resin bed causing agitation while the resin soaks in the surfactant solution. Agitation should be continued for approximately half an hour. Following this the unit should again be thoroughly backwashed and rinsed downflow until foaming disappears completely. Here again it is helpful if the first part of the rinse utilises water at approximately 40°C.
- The resin should then be thoroughly regenerated and rinsed before proceeding to the next service cycle.

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Produced by Hamilton-Rio Design, Epsom, U.K.
PSB/101/5.96

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Printed in England