LABORATORY TESTING OF ION EXCHANGE RESINS

1.0 INTRODUCTION

This is a Purolite guide to Chemists and Process Engineers on how to carry out simple evaluations of ion exchange and adsorbent resins in the laboratory to see if a particular process will work.

Depending on the results obtained and the depth of work carried out it may enable you to optimize some of the operating conditions. While it may not give you sufficient information to design a full scale plant, it should show if a process is viable and enable you to design a larger pilot plant if necessary.

Much development work by companies is carried out in secret to develop new and sometimes novel processes that can be patented. Much expensive development time can be saved by discussing your exact requirements with Purolite International Limited. We are willing to enter into confidentiality agreements with clients so that they are able to obtain the maximum from our help and support.

2.0 RESIN VOLUME

Where single beds are being tested approximately 100 ml of resin will be sufficient for an initial test. Remember on some applications where the inlet load is very low, it will require a very large amount of solution to be processed through the bed to reach exhaustion and this may make each test too long for you to complete the work quickly.

3.0 EQUIPMENT

To carry out initial trials a 25mm diameter glass or sometimes a plastic column is suitable. A glass column with a sintered glass bottom is ideal, but otherwise a column with a rubber stopper at each end with a glass tube through each stopper will work. If this type of simple equipment is used then a nylon cloth over the surface of each rubber stopper covered to a depth of 15-25 mm with small diameter glass beads (3mm diameter) at the bottom of the column will act as an adequate collection system.

If the column does not have a valve connection then rubber tubing and a screw clamp on the tubing can be used as an on/off valve and to control the flow rate.

4.0 RESIN STORAGE

Sometimes Purolite supplies resin to test, but unfortunately the trials cannot be carried out immediately. If you are storing resins prior to testing please ensure they are not left open to the atmosphere or allowed to dry out. They should also be kept away from strong sunlight and adverse temperatures.

5.0 PRECONDITIONING THE RESIN

Never test resins with very shallow bed depths. The minimum bed depth of 150 mm should be avoided if possible. In most applications the process will improve if the bed depth is increased.

Backwashing

Once the resin is loaded in the column the resin should be backwashed so that the resin bed is fully classified. This is carried out with demineralised water in an up flow manner. 10-15 minutes should be sufficient to classify the bed. The resin bed will expand and the larger particles will fall towards the bottom of the bed and the smaller beads will be nearer the surface when they are allowed to settle.

Following this process and depending on the particle size range of the resin, you will notice that the height of the resin will have increased. This is now the height (volume) of the resin you must use for all your calculations concerning the test work.

Rinsing

When samples are sent out from our store the resin is usually taken from production batches but it may have been in our store for some time. Before carrying out your trials rinse the resin with demineralised water to reduce any leachables that can arise in storage before carrying out the test. 5-10 bed volumes should be sufficient in many applications. In high purity applications please refer to Purolite.

None Water Applications

Unless a specially prepared product as been produced by Purolite, the resin will be delivered in a moist form and the beads will contain water. If the samples are to be tested in a non-aqueous application, or where the water content is very low or critical then the first step should be to displace the water with the process liquor. It is best to use treated product, otherwise your preconditioning treatment will actually be loading the resin with the material you want to capture on the beads. In some test work it is possible to use a suitable solvent such as acetone or alcohol to displace the water.

6.0 PROCESS PARAMETERS

Depending on the application the process conditions for the resin will vary widely.

In many water applications where the loading is small and conventional ion exchange is taking place then flow rates through the resin can be high (Up to 50 BV/h) and sometimes even greater flow rates are used.

In special process applications, or where highly selective removal is required then flow rates can be much lower (1-10 BV/h)

Where regeneration of a resin is undertaken this is normally carried out at relatively low flow rates to achieve maximum removal from the beads (1-6 BV/h) and is followed by a slow rinse at a similar flow rate to maximise the removal of the regenerant.

Finally a rinse at a higher or service flow rate is normally carried out.

In non-aqueous process applications the resin is often regenerated in the aqueous state. Under these circumstances the process liquor must first be displaced with water. This is often referred to as “sweetening off” and after regeneration the water is displaced with process liquor and is called “sweetening on”. These terms derive from the sugar industry where ion exchange resins and absorbents are used widely.

Parameters:

Resin volume	50 – 250 ml
Resin bed depth	150-600 ml (classified)
Service flow rate	2 – 50 BV/h (BV/h = Bed volumes per hour)
Regenerant flow rate	2 – 6 BV/h
Regenerant contact time	15 – 60 minutes
Slow displacement rinse	1 – 2 BV
Final fast rinse	2 – 10 BV

For regeneration chemicals, concentrations and quantities and details on sweetening on an off we strongly recommend you refer to Purolite for guidance.

7.0 REGENERATION

Many different ion exchange regeneration processes have been developed both in aqueous and non-aqueous applications. The simplest method of regeneration of regeneration is co-flow (Often referred to as co-current) where the regenerant passes downflow through the resin in the same direction as the service flow.

More widely used now is counter flow regeneration (often referred to as counter current or reverse flow). Here the regenerant passes in the opposite direction to the service flow and this results in much lower leakage from the bed. Counter flow regeneration is more difficult to set up in the laboratory and reference to Purolite International Limited for guidance is recommended. Parameters such as bed depth can be more critical as well as ensuring a means of retaining the bed in a fixed location has to be devised during the upflow passage of the testing solution or regenerant.

In co-flow regeneration the first stage of every regeneration cycle should be to backwash the bed. This relieves any compaction and can be used to remove any filtered out suspended matter. Solutions or water containing more than trace levels of solids should be removed by filtration prior to the test column.

Regeneration followed by a slow displacement rinse and then final rinse can then be carried out. In laboratory work it is often better if these three steps are carried out with demineralised water for regenerant dilution and the rinse stages as this ensures there is no ionic loading of the resin and makes calculations of capacity easier.

8.0 SERVICE OPERATION

Once a trial run has begun the resin should be allowed to continue to operate through to exhaustion. You should not stop the experiment in mid cycle. Most ion exchange reactions are reversible and hence once the solution is stopped the solution tries to reach an equilibrium and the ions come back off the resin into solution. This can cause premature exhaustion and leads to false results being obtained.

It normally takes two or three cycles from when the resin is first tested to obtain reliable information from your test work and you should aim to get three consecutive cycles giving consistent results before changing any operating conditions to try to optimise the performance.

Under normal conditions tests the bed must remain covered with solution. Never drain the column and introduce air into the bed as this will give poor performance and air bubbles are difficult to remove.

9.0 EQUIPMENT SET UP

Depending on the nature of the trial you may be able to feed the column by gravity or have to use a pump. If gravity feed is used then the pipework from the column can be arranged in a U shape so that it rises to a level above the top of the bed to keep the resin flooded at all times.

Where gravity or pumped systems are used it is essential that you have ready made up solutions of regenerant and demineralised water for the regeneration process made up and stored ready.

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