Test Method for Ion Exchange Resin

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1 Preparation of stock solution

Prior to experiments, remove suspended solids, oils, and oxidizers from the stock solution brought into contact with the ion exchange resin.

2 Selection of resin brand

There are two methods for selecting a resin brand while checking adsorption behavior: the batch method and the column method. Each of these methods is described below.

2.1 Batch method

The batch method is a simple technique as an initial screening to test which resin brand suits the intended purpose.

Place about 10 mL of conditioned resin into a beaker or Erlenmeyer flask, and pour in the specified amount of stock solution. The preparation of the conditioning is described later on section 3.3. Stir or shake vigorously enough for the resin to float and rest for the resin to react about 0.5–2 hours. Then check the adsorption effect by filtering the supernatant from the resin slurry and analyze the filtrate. The adsorption rate can be calculated by conducting in fixed intervals.

Take a note on use of a magnetic stirrer as it may cause crushing of the resin.

2.2 Column method

Fill a column with 10–20 mL of the conditioned resin, and allow stock solution to flow at a flow velocity of SV 5–20. Sample a fixed amount of the treated liquid, analyze, and check effectiveness. Use a low flow velocity if the molecular weight of the adsorbed material is high, or if the viscosity of the liquid is high.

3 Determination of test conditions

Once a particular type of resins is chosen, conduct a column liquid passage test to check liquid passage conditions for industrialization purposes and to optimize regeneration conditions.

(As a special case, industrialization may be done using the batch method.)

3.1 Experimental equipment

In the case of a small-scale experiment for resin selection, as indicated above, it is convenient to use a 10–15 mm (dia.) glass column for the experiment, but for this

experiment, use a column with a diameter of about 20 mm or more, and set the height of the layer filled with ion exchange resin to 60 cm or higher. In addition, when performing backwashing, the column must have backwashing space roughly the same as the resin layer height.
(If the purpose is not to collect design data, then a scale smaller than the above conditions is fine, and there is no need to perform backwashing.)

Care is needed when using a PVC or acrylic column because there are restrictions on factors such as solvents used and experiment temperature.

Fix the column perpendicular to the support stand etc. Fig. 1 shows an example of the experimental equipment.

If there is no pump, the stock solution container can be placed at a location higher than the column, and liquid allowed to flow due to gravity.

When using a resin with large changes in volume due to the ion form, such as weakly acidic cation exchange resin or weakly basic anion exchange resin, there is a risk of column damage due to swelling pressure of the resin. Therefore, please consider to use a large-diameter glass column in such a case.

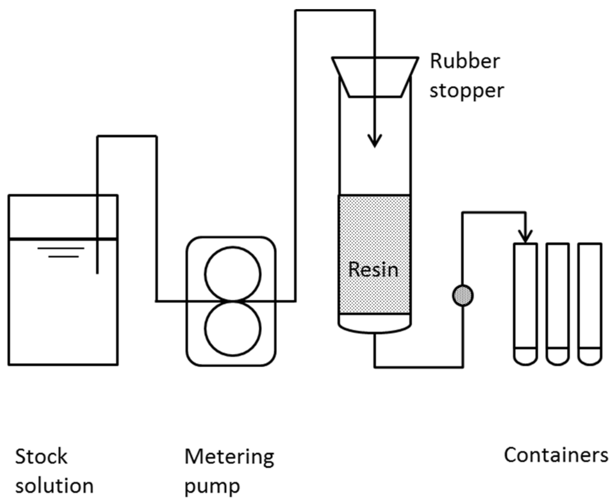

Fig. 1: Equipment

3.2 Resin filling method

When filling resin into the column, take the specified amount of resin, obtained by accurately measuring volume in water beforehand using a measuring cylinder, and add while in the slurry state into the column filled with water (Fig. 2).

At this time, be careful not to allow air bubbles into the resin layer. If air bubbles get in, insert a rod or similar tool, and remove the air bubbles.

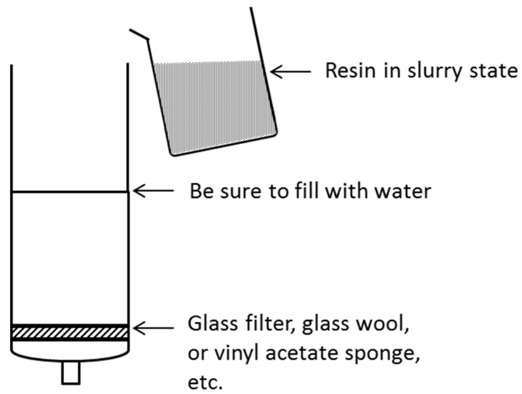

Fig. 2: Resin filling method

3.3 Resin conditioning

The ion form of a typical strongly acidic resin or strongly basic resin is a salt (Na or Cl) form. Also, even some weakly acidic resins and weakly basic resins have a salt form. Therefore, the following regeneration procedure must be performed as resin conditioning before use.

The amounts of regeneration agent used are as follows. These are twice the amounts for normal regeneration.

Strongly acidic resin	300–500 g-HCl/L-R
Strongly basic resin	300–500 g-NaOH/L-R
Weakly acidic resin	100–150 g-HCl/L-R
Weakly basic resin	100–150 g-NaOH/L-R

*When an ion exchange resin is first used, there is elution of trace amounts of impurities. The procedure for quickly reducing these elutes is also called conditioning. The procedure for this purpose involves repeating the regeneration and salt load steps 2–3 times.

3.4 Steps of ion exchange resin procedure

As a typical example of the regeneration/liquid passage procedures, the following shows the procedure steps. For details on each step, see "4. Experiment procedure" below.

Backwashing	Expansion rate is 1.3–1.5 times the resin layer height. 15–30 min
Settling down	5-10 min
Water draining	About 5 cm above the resin surface
Regeneration	See 4.4
Expulsion	1.5–2 BV at same flow velocity as regeneration step
Washing (1)	30–60 min at same flow velocity as liquid passage step
Sweetening on	1–2 BV at same flow velocity as liquid passage step
Liquid passage	See 4.8
Sweetening off	1–2 BV at same flow velocity as liquid passage step
Washing (2)	15–30 min at same flow velocity as liquid passage step

4 Experimental procedure

The following explains the experiment procedure for the column method, but steps 4.1 Backwashing – 4.2 Settling down can be omitted depending on the situation. In addition, if there is no need to recover liquid, there is no need to perform 4.7 sweetening on and 4.9 sweetening off.

4.1 Backwashing

This is performed to discharge, to the outside of the column, the suspended solid (SS) component mixed in during liquid passage and the resin crushed during use, and to improve contact efficiency with the regeneration agent by loosening the compacted resin layer. Perform backwashing with an upward flow at a flow velocity so that the resin layer expands by 1.3–1.5 times, until the water flowing out is transparent. The backwashing expansion rate varies in each case depending on the brand used and the water temperature, so set flow velocity based on DIAION Data Sheets.

4.2 Settling down

This step allows the resin layer, expanded due to backwashing, to naturally settle.

4.3 Water draining

After the settling down step is finished, this process drains off water at the top of the column to a position about 5 cm above the resin surface. This is done to avoid dilution of the regeneration agent.

4.4 Regeneration

This step converts a resin in the salt form to the regenerated form. The type of regeneration agent and the amount used varies in each case depending on the resin used.

Typical regeneration agent concentrations are as follows.

Strongly acidic resin	3–8%	HCl or H_2SO_4
Strongly basic resin	3–8%	NaOH
Weakly acidic resin		0.5–5% HCl
Weakly basic resin	0.5–5%	NaOH

Time needed to supply regeneration agent is roughly 30 min with a strongly acidic resin. In the case of a strongly basic resin, roughly 45 min is a good guideline.

Even if the regeneration level is raised to a fixed level or higher, it is not possible to obtain a regeneration effect corresponding to the amount of regeneration agent used. Therefore, it is typical to perform management using the following regeneration rates and amounts of regeneration agent used.

<table>
<tr><th colspan="2"></th><th>Regeneration rate</th><th>Amount of regeneration agent used</th></tr>
<tr><td colspan="2">Strongly acidic resin</td><td>50–70%</td><td>100–300 g/L-R</td></tr>
<tr><td rowspan="2">Strongly basic resin</td><td>Type I</td><td>30–50%</td><td rowspan="2">100–300 g/L-R</td></tr>
<tr><td>Type II</td><td>50–70%</td></tr>
<tr><td colspan="2">Weakly acidic resin</td><td>80–100%</td><td>60–120 g/L-R</td></tr>
<tr><td colspan="2">Weakly basic resin</td><td>80–100%</td><td>60–120 g/L-R</td></tr>
</table>

*The regeneration method above is for resin which has adsorbed impurities (unnecessary material). The resin may also be used in cases where valuable material is adsorbed using special chemicals. In those cases, there are two approaches: eluting the target material using an appropriate eluent and then performing elution; or performing elution and regeneration together.

4.5 Expulsion

This step is an extension of regeneration. Here, unreacted regeneration agent remaining in the column is expelled using pure water at the same flow velocity as regeneration. The amount of pure water used is 1.5–2 times the amount of ion exchange resin.

4.6 Washing (1)

This step is for completely removing regeneration agent remaining in the column. Normally, this is performed for 30–60 min at the same flow velocity as the liquid passage process. The process up to this step completes the regeneration process.

4.7 Sweetening on

When performing treatment with a special chemical liquid, water in the resin layer is steadily replaced due to the liquid being passed through. "Sweetening on" refers to this replacement step.

4.8 Liquid passage

Recovery as treatment liquid begins from the point where the chemical liquid at the column outlet reached or exceeded a certain concentration, and continues until ion leakage occurs and the specified liquid quality is no longer obtained. This process is called liquid passage. Flow velocity during liquid passage is SV20–50 in ordinary water treatment. In other cases it is SV10 or lower.

4.9 Sweetening off

In treatment with chemical liquid, this is the process of replacing the stock solution in the column with water after passing through the chemical liquid. The term applies until the outlet stock liquid concentration is at or below the specified concentration. This portion is recovered as treatment liquid or sweet water.

4.10 Washing (2)

In treatment with chemical liquid, this is performed in the same way as washing after regeneration. The amount of washing water used is about 1/2 that used after regeneration.

5 Check items for equipment development

Assuming that actual equipment will be developed, check factors such as liquid passage flow rate, temperature, treatment amount, regeneration level, service life, etc., and set optimal treatment conditions. Items to be checked are indicated below, but it is difficult to estimate life with ordinary column tests. Therefore, perform other tests such as accelerated cycle tests, long-term testing using a pilot plant, or a sample hanging test is actual equipment is in operation.

*Liquid passage conditions	*Regeneration conditions
-Treatment amount -Liquid quality -Flow velocity -Temperature -pH, etc.	-Chemical type -Amount used -Concentration -Flow velocity -Temperature

A pilot plant may be operated to recheck and evaluate design conditions, and to manufacture samples for distribution. Normally, the plant is 1/10 the scale of the actual equipment, and even a large pilot plant operates with resin amounts of about 50–100 L.