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Dow
Water Solutions

DOWEX™ Fine Mesh Spherical Ion Exchange Resins

For Fine Chemical and Pharmaceutical Column
Separations

A full range of particle sizes and cross-linkages helps ensure success from lab to plant

Column Separations

For the highest levels of selectivity, consistency, and reliability in column separations, there is no better choice than DOWEX™ spherical fine mesh ion exchange resins. DOWEX fine mesh resins are produced by selectively controlling the suspension polymerization technology we use to produce our standard 16 - 50 mesh industrial resins. The resulting resins consist of whole, spherical beads that provide excellent kinetic and packing properties in chromatographic separations... without the problems associated with ground or granular resins.

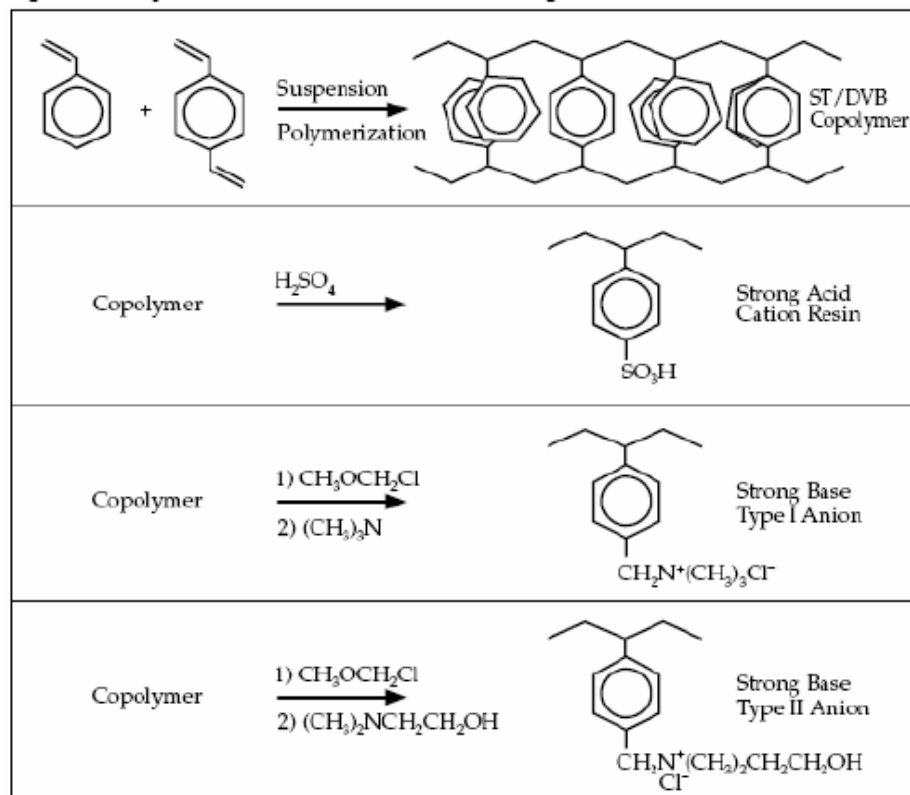
Dow offers a wide range of fine mesh resins for use in fine chemical and pharmaceutical column separations. Anion and cation resins with mesh sizes including 50 - 100, 100 - 200, and 200 - 400 are available. Cross-linkages (as measured by percent divinylbenzene content) include 2, 4, and 8. This broad product line permits selection of a resin offering the optimum levels of permeability, water retention capacity and total capacity for your particular application.

Dow's range of resin options not only enhances your ability to optimize laboratory separations but also simplifies the adjustments that are often necessary during process scale-ups. This can help ensure that your success in the lab translates into success in pilot-scale as well as full-scale plant operations.

DOWEX fine mesh resins are durable, pH tolerant, clean and predictable separations media that perform consistently and reliably in fine chemical and pharmaceutical column separations.

Strong, insoluble structure—DOWEX fine mesh resins are based on a microporous copolymer of styrene and divinylbenzene (DVB) that results in maximum resistance to oxidation, reduction, mechanical wear and breakage. What's more, this structure is also insoluble in common solvents. Figure 1 depicts the synthesis of DOWEX resins.

Figure 1. Synthesis of DOWEX ion exchange resins



DOWEX fine mesh resins are based on a microporous styrene/DVB copolymer. DVB content is controlled to produce resins with varying degrees of permeability. Introduction of the ion-active functional group to the copolymer results in strongly acidic cation exchange resins and strongly basic anion exchange resins.

Low in fines—DOWEX™ fine mesh resins are produced through direct synthetic manufacture. This results in whole, spherical resin beads that are cleaner and perform more reliably and consistently than ground resins. Ground resins consist of irregular shaped particles and contain “fines” or very fine particles. Fines are a particular source of processing difficulties. They produce dust when dry resins are handled. They plug frits and filters when the resin is used as chromatographic media or as a filtering aid. They extend settling times in batch processing applications. And they contribute to resin attrition due to physical losses.

Tight control of particle size and permeability—By controlling the suspension polymerization process, the particle size of DOWEX fine mesh resins is tightly regulated. The percentage of DVB used in the process is also controlled to determine cross-linkage (or permeability) of the resins. These factors result in a range of products with specific, dependable performance characteristics.

Dow Resin Nomenclature

The nomenclature used to identify DOWEX™ fine mesh resins is designed to communicate specific information regarding resin type and degree of cross-linkage. There are three resin types: strong acid cation exchange resins designated as “50W”, Type I strong base anion exchange resins designated as “1”, and Type II strong base anion resins designated as “2”.

Under the Dow nomenclature, one of these three designations appears in conjunction with an “X” number describing the degree of resin crosslinkage (the X number is the percentage of divinylbenzene [DVB] in the resin copolymer). Therefore...

DOWEX 50WX8 is a strong acid cation resin containing 8% DVB.

DOWEX 1X4 is a Type I strong base anion resin containing 4% DVB.

How to Evaluate and Select DOWEX™ Fine Mesh Resins

Table 1 contains basic information about the complete line of DOWEX™ fine mesh resins. Use this table to select the best resin candidates for your application.

Table 1. The full range of DOWEX resin options^{1, 2}

| Cation resins | Mesh size | Ionic form | Water retention capacity | Total exchange capacity | lbs/ft ³ | lbs/5 ft ³ drum |
|---------------------|-----------|-----------------|--------------------------|-------------------------|---------------------|----------------------------|
| | | | (%) | (meq/mL) | | |
| DOWEX 50WX2 | 50 - 100 | H ⁺ | 74 - 82 | 0.6 | 46 | 230 |
| DOWEX 50WX2 | 100 - 200 | H ⁺ | 74 - 82 | 0.6 | 46 | 230 |
| DOWEX 50WX2 | 200 - 400 | H ⁺ | 74 - 82 | 0.6 | 46 | 230 |
| DOWEX 50WX4 | 50 - 100 | H ⁺ | 64 - 72 | 1.1 | 48 | 240 |
| DOWEX 50WX4 | 100 - 200 | H ⁺ | 64 - 72 | 1.1 | 48 | 240 |
| DOWEX 50WX4 | 200 - 400 | H ⁺ | 64 - 72 | 1.1 | 48 | 240 |
| DOWEX 50WX8 | 50 - 100 | H ⁺ | 50 - 56 | 1.7 | 50 | 250 |
| DOWEX 50WX8 | 100 - 200 | H ⁺ | 50 - 58 | 1.7 | 50 | 250 |
| DOWEX 50WX8 | 200 - 400 | H ⁺ | 50 - 58 | 1.7 | 50 | 250 |
| Anion resins | | | | | | |
| DOWEX 1X2 | 50 - 100 | Cl ⁻ | 65 - 75 | 0.7 | 44 | 220 |
| DOWEX 1X2 | 100 - 200 | Cl ⁻ | 70 - 80 | 0.6 | 44 | 220 |
| DOWEX 1X2 | 200 - 400 | Cl ⁻ | 70 - 80 | 0.6 | 44 | 220 |
| DOWEX 1X4 | 50 - 100 | Cl ⁻ | 50 min. | 1.0 | 44 | 220 |
| DOWEX 1X4 | 100 - 200 | Cl ⁻ | 55 - 63 | 1.0 | 44 | 220 |
| DOWEX 1X4 | 200 - 400 | Cl ⁻ | 55 - 63 | 1.0 | 44 | 220 |
| DOWEX 1X8 | 50 - 100 | Cl ⁻ | 43 - 48 | 1.2 | 44 | 220 |
| DOWEX 1X8 | 100 - 200 | Cl ⁻ | 39 - 45 | 1.2 | 44 | 220 |
| DOWEX 1X8 | 200 - 400 | Cl ⁻ | 39 - 45 | 1.2 | 44 | 220 |

¹ Standard sales packs of one and five cubic feet are available. For further information, call the Dow Customer Service Center, 1-800-232-2436.

² Smaller, laboratory-size quantities are available through suppliers of chromatography and fine chemical products.

While the chemical and physical properties of ion exchange resins are defined by specific measurements, actual resin performance also depends on the interrelationship between resin properties and the application conditions under which the resin will operate.

Following are brief discussions of three basic considerations that may apply to your resin selection process:

The impact of application conditions on resin operating capacity

Resins with greater ion exchange capacity will not necessarily yield the greatest operating capacity in a particular application. In fact, for a specific solution, resin cross-linkage may play a more important role than ion exchange capacity in determining solute uptake.

This is illustrated in Table 2. Uptake of erythromycin was measured for several fine mesh resins with varying levels of ion exchange capacity and cross-linkage. The illustration shows the two lower cross-linked strong acid cation resins (DOWEX™ 50WX2 and DOWEX 50WX4) provided higher erythromycin uptake than the tighter cross-linked resin (DOWEX 50WX8) even though their ion exchange capacities are lower.

Table 2. Uptake of erythromycin by resins with varying ion exchange capacities and cross-linkage

| Resin | Exchange capacity (meq/mL) | Erythromycin adsorbed ^{1,2} (mg/mL) |
|-----------------------|----------------------------|--|
| DOWEX 50WX2, 50 - 100 | 0.6 | 5.9 |
| DOWEX 50WX4, 50 - 100 | 1.2 | 11.7 |
| DOWEX 50WX8, 50 - 100 | 1.7 | 0.86 |

1 As adsorbed from a filtered fermentation broth with an initial antibiotic concentration of 1,000 micrograms per milliliter.

2 F.J. Dechow, "Separation and Purification Techniques in Biotechnology," Noyes Publications, Park Ridge, NJ, 1989.

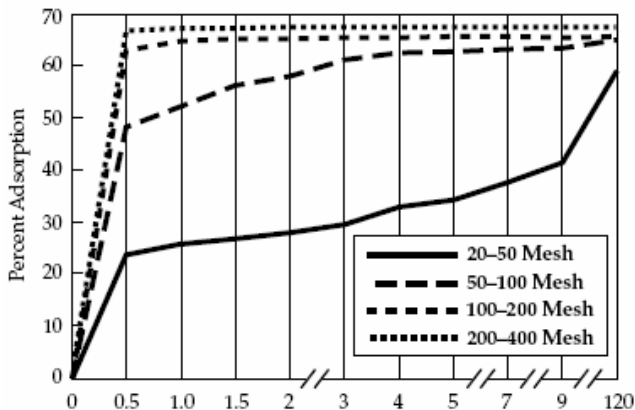
Solvent conditions can affect actual resin operating capacity as shown in this example where, despite greater ion exchange capacity, actual uptake of erythromycin by the tighter cross-linked DOWEX 50WX8 resin was less than the two resins with lower cross-linking.

The effective porosity of a resin can be affected by solvent conditions including ionic strength, pH, and organic modifiers. The availability of a range of DOWEX resins with varying degrees of cross-linkage is helpful in conducting size-in-solution evaluations to determine the optimum resin for a particular application.

Balancing solute uptake requirements and pressure drop

Solute uptake is influenced by particle size, with solute adsorption generally increasing as particle size decreases. This is illustrated in Figure 2 which shows uptake of L-tryptophan by DOWEX 1X4 resin of various particle sizes in a batch adsorption test. The standard 20 - 50 mesh resin shows a slower uptake isotherm than those observed for the finer particle size resins.

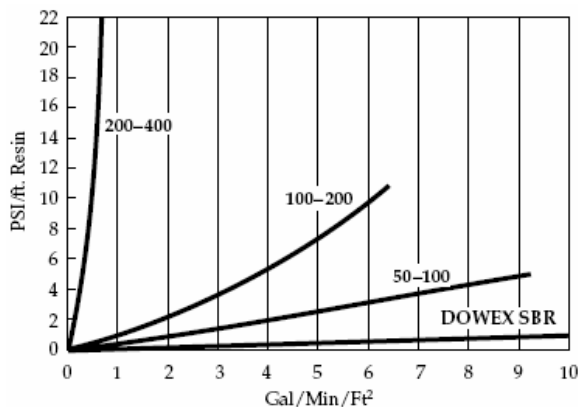
Figure 2. Influence of particle size on uptake of L-tryptophan by DOWEX 1X4 (1 eq resin/eq L-tryptophan, batch adsorption)



Uptake of L-tryptophan by DOWEX 1X4 fine mesh resins (mesh sizes 20-50, 50-100, 100-200, and 200-400) was compared in a batch adsorption test. The data show that solute uptake increased with decreasing particle size. The standard 20-50 mesh resin showed a slower uptake isotherm than those of the finer particle size resins.

However, simply selecting a resin based on small particle size can result in mechanical difficulties in some applications. Figure 3 shows that, as the particle size of a resin in a column decreases, the frictional loss or pressure drop of a liquid flowing through the column increases. While the kinetics of a particular process may be improved with a smaller particle size resin, increases in pressure drop must be taken into account when choosing equipment, establishing operational parameters and evaluating scale-up potential.

Figure 3. Influence of particle size on pressure drop, comparison of DOWEX™ SBR industrial-grade resin and DOWEX 1X2 fine mesh resin



Pressure drop of DOWEX 1X2 fine mesh resins (including 50-100, 100-200, and 200-400 mesh sizes) was compared to that of DOWEX SBR industrial-grade Type I strong base anion resin (16-50 mesh). The data show that pressure drop increased with decreasing particle size and the larger particle size industrial resin produced lower pressure drop than the fine mesh resins.

Certain procedures are helpful in evaluating resin solute uptake capabilities and pressure drops in a particular system. To measure solute uptake, use batch-wise experiments where the uptake of a solute of known concentration by a specific volume of resin is measured over time. This type of batch experiment is useful in determining the effects of particle size and in identifying the impact of factors (including pH, temperature, ionic strength and competing solutes) that contribute to chemical interactions at the active ion exchange sites, to overall process kinetics and to the speed with which ion exchange takes place.

When making pressure drop comparisons, study effluents that have the same viscosity as the process stream. Whenever possible, work with actual process streams to ensure that pressure drop observed during the comparisons will reflect that which is likely to be encountered in actual operations.

Factors that affect resin stability

The operational stability of ion exchange resins is largely defined by the particular process involved. Therefore, stability testing under actual process conditions is an important part of the resin evaluation and selection process.

Ion exchange resins are tolerant of a wide range of chemical conditions and can be washed over the entire pH range without ill effects. In addition, ion exchange resins are tolerant of most inorganic and organic solutions with the exception of strong oxidizing agents.

Solutions of nitric or chromic acid, bleach or chlorine-generating solutions, as well as peroxide should be avoided. Contact with strong oxidizing agents such as nitric acid will result in rapid oxidation. With the right set of conditions, this can result in a potentially explosive reaction.

Slower degradation with oxygen may be catalytically induced. Therefore, exposure of resins to metal ions (including iron, manganese and copper) should be minimized in oxidizing environments.

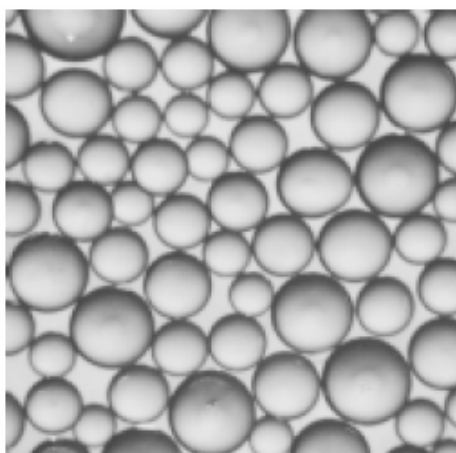
The physical stability of resins is also affected by the method of product handling. Deep resin beds, small diameter beds, very high flow rates and frequent pumping or agitation all can contribute to resin attrition breakage.

Attrition breakage due to mechanical factors can be minimized by understanding the physical conditions imposed by the system and process. For example, if the chemical environment is altered to change solvent retention capacity, the resin may undergo rapid shrink-swell. If the resulting osmotic shock weakens the chemical morphology of the resin beads, attrition breakage can result. In most cases, breakage due to shrink-swell results from a combination of osmotic shock and column mechanical effect, which is described on pages 7 and 8 of this bulletin under "Hints for Packing Laboratory Columns".

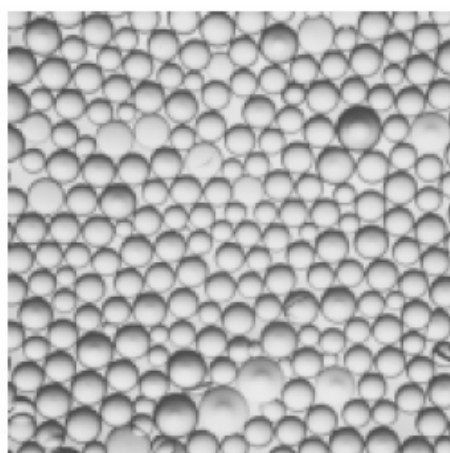
To evaluate resin stability for a particular process, use actual process samples to create a system model. Expose a small column of each candidate resin to multiple recycles of loading, recovery and regeneration using the actual product feed and follow-up chemical conditions. Use microscopic examination to measure the stability performance of resins subjected to the modeled conditions. As shown in Figure 4, such examination can detect cracked, broken and powdered, or discolored resin particles.

Figure 4. Resin photomicrographs showing the effect of particle size on osmotic stability after 10 cycles 10% NaOH and 10% HCl

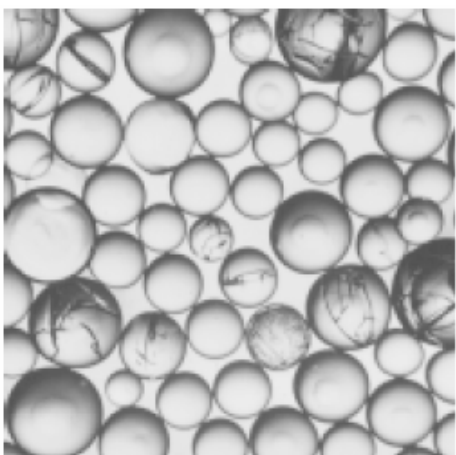
Osmotic model: A cycle of four bed volumes of NaOH (10%), followed by two bed volumes of distilled water, followed by four bed volumes of HCl (10%), followed by two bed volumes of distilled water. Resin particle cracking and breakage under these test conditions was significantly lower in the smaller particle size resin.



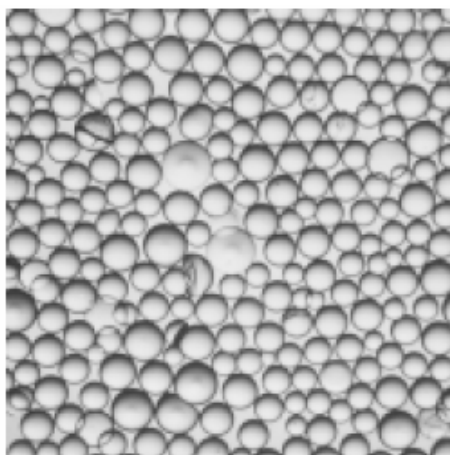
DOWEX™ 50WX8 20-50 Before



DOWEX 50WX8 50-100 Before



DOWEX 50WX8 20-50 After



DOWEX 50WX8 50-100 After

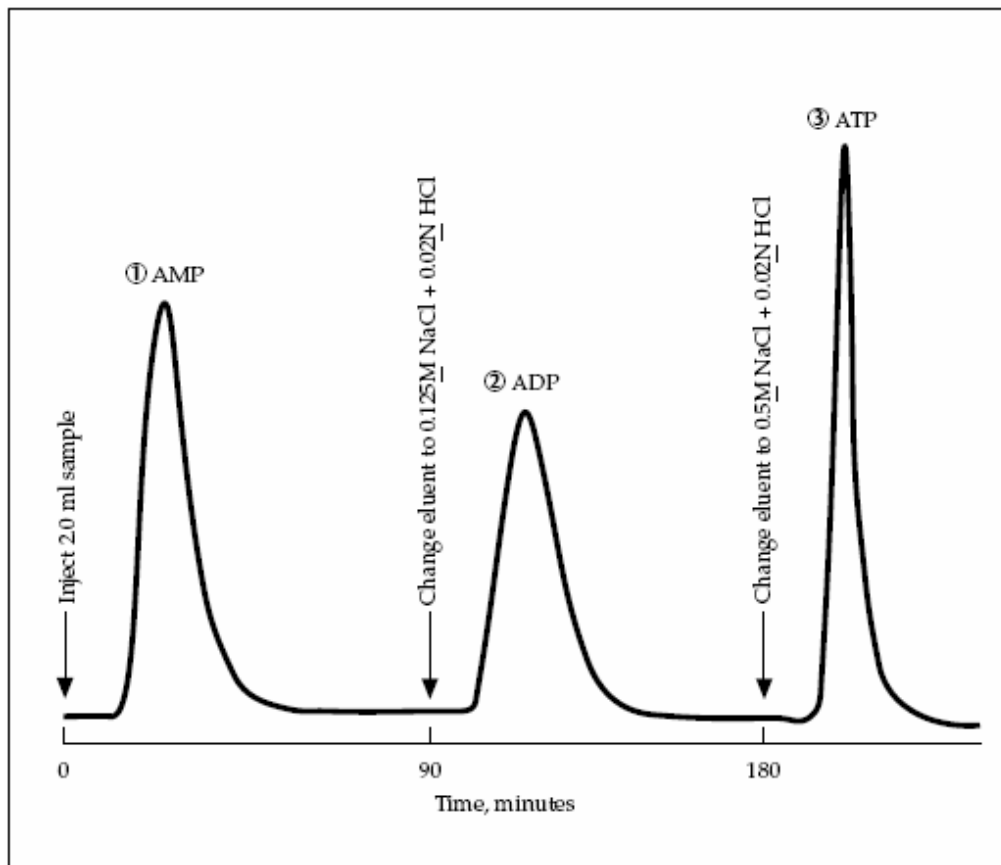
Figure 4 specifically compares photomicrographs of DOWEX 50WX8 resins, mesh sizes 20–50 and 50–100, after the resins were osmotically shocked with 10 cycles of 10% NaOH and 10% HCl.

Visual inspection reveals that, in this case, the smaller particle size resin exhibited greater stability under the test conditions. The criteria used to determine the success or failure of resins tested in this fashion may vary by application.

Hints for Packing Columns

Laboratory scale columns can be used to gain valuable process development and resin feasibility information. For example, Figure 5 shows the step-wise gradient elution of a series of three nucleotides...AMP, ADP, and ATP... illustrating how a column can be used to model and design a separation process.

Figure 5. Separation of a nucleotide test mixture (AMP, ADP, ATP) using DOWEX 1X8, 200 - 400 fine mesh resin and a step-wise gradient



Column: 1.5 cm x 30 cm
Resin: DOWEX 1X8, 200-400 mesh
Initial Eluent: 0.02N HCl
Flow: Downward 2.00 ml/min
Detector: UV spectrophotometer; wave length = 254 nm
Injection: 2.00 ml AMP, ADP, ATP mixture; concentration of each nucleotide is equal to 0.167 mg/ml.

Although lab column runs can provide valuable comparison data in selecting resins and in establishing process operating parameters, they are not directly scalable without taking system design into account. What's more, lab column studies should not be used as a substitute for developing and operating a pilot system prior to scale-up. Following are basic guidelines for packing lab columns:

Fine mesh resins can be used in common glass columns, but columns that resist pressures of approximately 3 kg/cm² or greater are best.

WARNING: Under certain conditions, some resins can expand with sufficient force to break a glass column. Lab columns equipped with over-fitting safety tubes or glass columns with plastic safety shields are recommended for laboratory evaluations. Columns should have internal diameters no less than 1.5 cm. Smaller columns can produce wall effects that can disturb chromatographic separation and affect resin breakthrough points.

Prior to column packing, completely hydrate and exercise the resin by washing it using a mobile phase that will cause the greatest swelling. Next, slurry-pack the column taking care to remove air bubbles. Do not allow the column to run dry. Settle the packed bed by passing mobile phase at a flow rate slightly higher than the rate of normal operation (e.g., if the operational flow rate is 2.0 mL/min, settle the column bed at a flow rate of 2.5 mL/min).

Once the column is packed, cycle the system between the eluting and regenerating mobile phases to determine if significant shrink-swell occurs. Add make-up resin or adjust volume at this time before investing a real sample in the evaluation. Lab columns with adjustable plungers can simplify this fine-tuning process.

To determine optimum loading and elution rates, first load about 10% of the column volume (for concentrated samples) or a volume amount containing about 75% of the theoretical exchangeable capacity for a dilute feed. Loading flow rates of 0.5 bed volumes per hour are a good starting point. Fine-tuning requirements will depend on the concentration of the components in the feed, the required product purity and the ease of separation.

To Order Resins, or to Learn More...

Standard one and five cubic foot sales packs of DOWEX™ fine mesh resins are available by calling the Dow Customer Service Center: 1-800-232-2436.

Smaller laboratory-size quantities are available through suppliers of chromatography and fine chemical products.

General References

F.C. Nachod and J. Schubert, "Ion Exchange Technology," Academic Press, Incorporated, New York, NY, 1956.

F. Helfferich, "Ion Exchange," McGraw-Hill Book Company, New York, NY, 1962.

F.J. Dechow, "Separation and Purification Techniques in Biotechnology," Noyes Publications, Park Ridge, NJ, 1989.



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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