

Adsorption Basics: Part 2

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This article focuses on adsorbents, including activated carbon, zeolite molecular sieves, and polymeric adsorbents, as well as ion exchange resins.

Adsorption is a unit operation that exploits the attraction of solutes in a liquid or gas to a solid surface. This attraction allows the solutes to be removed, or solutes with different affinities for the solid to be separated. The first article (1) of this two-part series presented the fundamentals of adsorption, including equilibrium and mass-transfer considerations, the mass-transfer zone concept, breakthrough, regeneration, and column sizing. This article, Part 2, covers the most widely used adsorbents — in order of decreasing commercial importance (2, 3): activated carbon, zeolite molecular sieves, silica gel, activated alumina, and polymeric adsorbents — with discussion of their physical characteristics, suitable applications, and regeneration requirements. Ion exchange, a process that is similar to adsorption and is used to remove ions from solution, is also reviewed.

Activated carbon

Activated carbon is a hydrophobic, highly porous adsorbent that is produced by roasting one of a variety of organic precursors, including coconut shells, wood, lignite, and coal. Roasting is followed by activation, which increases adsorption capacity by removing contaminants from the surface of the adsorbent. Activation can be done thermally, by heating the material to 1,000°C or higher to decompose impurities. Afterward, the material is exposed to an oxidizing atmosphere, which substantially increases the pore volume. Alternatively, chemical agents such as phosphoric acid can be used to activate the adsorbent.

Activated carbon products may be granular (*i.e.*, made directly from a granular precursor), pelletized, or crushed to a powder. Powdered activated carbon has shorter diffusion paths and thus higher mass-transfer rates. However, powdered carbon is suitable only for the treatment of liquids in a

batch mix tank, because the high pressure drop precludes its use in a column. Spent powdered carbon must be removed by filtration, and regeneration is not practical.

Activated carbon is a highly versatile adsorbent, because its performance characteristics can be tailored by varying the precursor, roasting conditions, and activation method. Consequently, products are available for a wide range of applications, for treating both gases and liquids. Liquid applications include potable water treatment (discussed later), groundwater remediation, industrial and municipal wastewater treatment, sweetener decolorization, and chemical and pharmaceutical processing. Examples of gas applications are removal of organics from waste gas, capture of gasoline vapor emissions from automobiles, and adsorption of gaseous radionuclides from nuclear power plants.

Key properties of four common types of carbon are shown in Table 1. Coconut-derived carbons are generally considered high in quality, as evidenced by their high hardness (so they experience less attrition), low ash content, low dusting tendency, relative ease of regeneration, and high iodine number. The iodine number is the milligrams of elemental iodine adsorbed per gram of carbon. This fundamental performance parameter is an indicator of activity, particularly for liquid applications. A higher iodine number is better.

Coal-derived products have higher ash content and are more prone to dusting. Lignite-derived carbons are lower in quality, with low hardness, high ash content, high dusting tendency, poor regenerability, and low iodine number. The large number of macropores allows better diffusion of large molecules, which makes lignite-derived carbons suitable for use with liquids. However, more macropores means less surface area per unit volume of adsorbent, and, in turn, lower capacity. Powdered carbon derived from wood also has a

large number of macropores, as well as low ash content and a high iodine number — but powdered carbons have several disadvantages, as outlined earlier.

To illustrate the effects of fluid properties and operating conditions on activated carbon performance, consider potable water treatment as an example. The objectives are to improve taste and to remove precursors of toxic products that would otherwise form during chlorination. Activated carbon is a hydrophobic adsorbent, and it typically removes large molecules more easily because they tend to be more hydrophobic than small molecules. Performance is better at acidic pH, because many contaminants in public water supplies are organic acids that originate from decaying plant matter, and the protonated form is more hydrophobic than the salt form. A rule of thumb is that the required bed size increases by 20% for every pH unit above 7 (4).

Higher feedwater hardness equates to greater polarity. Higher polarity causes a shift in the equilibrium distribution in favor of the solid, which increases adsorption. In addition, adsorption improves with a higher adsorbate concentration,

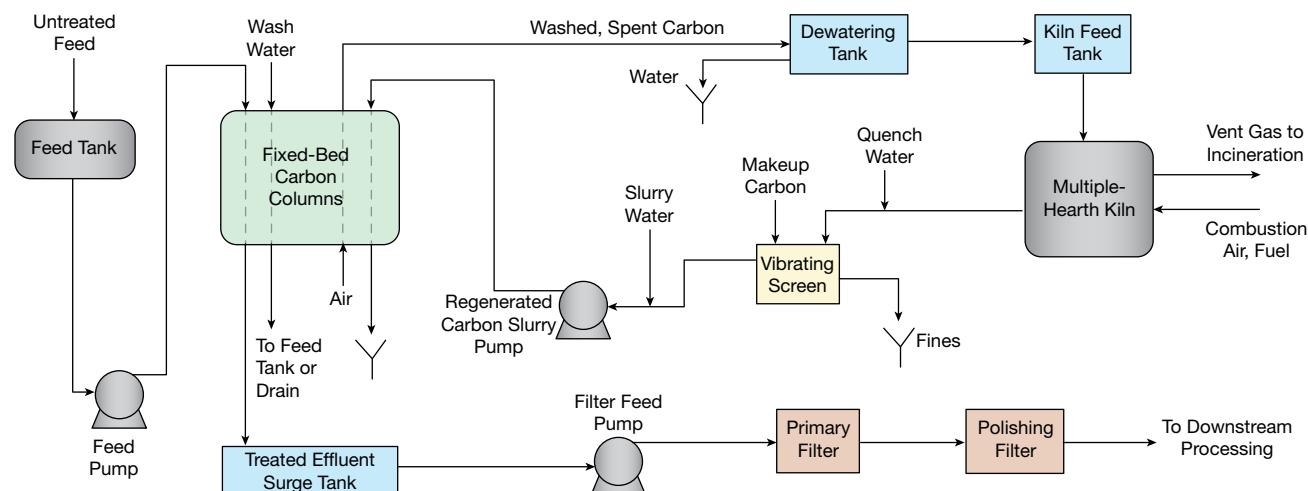
because the amount adsorbed at equilibrium is correspondingly higher. In water treatment service, activated carbon typically adsorbs about 10% of its weight in organics (4).

As with adsorption processes in general, water treatment with activated carbon typically improves with decreasing flowrate. The usual residence time is 15–30 min, although the faster mass transfer obtained with smaller particles allows higher flowrates. In general, 20×50 mesh particles (with diameters of 0.3–0.8 mm) can accommodate twice the flowrate of 12×40 mesh ones, which in turn can handle twice the flowrate of 8×30 mesh carbon. However, even though smaller particles can accommodate higher flowrates, they have higher pressure drops (4).

In an activated carbon liquid treatment plant (Figure 1), the treated column effluent is filtered to remove any carbon fines that pass through the screen supporting the carbon bed, or to catch particles that may escape because of a screen or gasket failure. A polishing filter may be necessary to remove fine particulates, or to protect the downstream process in the event of a primary filter malfunction.

Table 1. Activated carbon can be generated from a variety of precursors, including coconut, coal, lignite, and wood.

	Coconut	Coal	Lignite	Wood (Powdered)
Micropores (<20 Å)	High	High	Medium	Low
Macropores (>500 Å)	Low	Medium	High	High
Hardness	High	High	Low	N/A
Ash, %	5	10	20	5
Water-Soluble Ash	High	Low	High	Medium
Dust	Low	Medium	High	Very High
Ease of Regeneration	Good	Good	Poor	None
Bulk Density, g/cm ³	0.48	0.48	0.40	0.35
Iodine Number	1,100	1,000	600	1,000



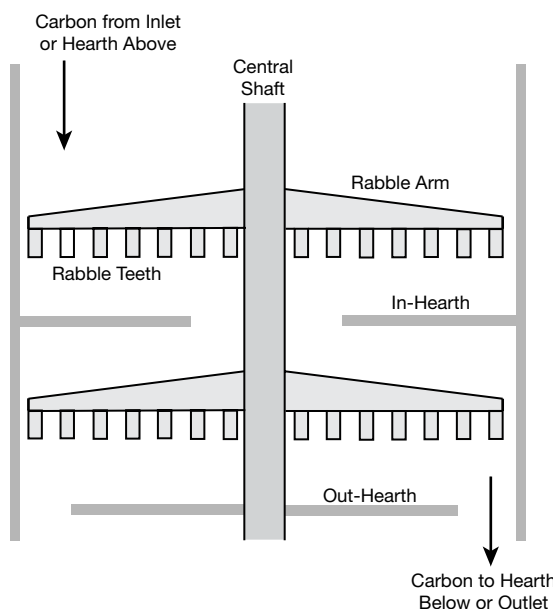
▲ **Figure 1.** In this process for treating a liquid with activated carbon, the treated column effluent is filtered before it moves to downstream processing. Spent, washed carbon is conveyed by air to a dewatering tank, then fed to a kiln for regeneration.

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In the process depicted in Figure 1, spent, washed carbon is conveyed by air to a dewatering tank, where water is allowed to drain by gravity over a period of several hours. When the kiln feed tank is nearly empty, the contents of the dewatering tank are dropped into it, making room in the dewatering tank for the next load. Carbon is continuously fed to the kiln by a screw conveyor.

Spent carbon is regenerated in a multiple-hearth kiln, which burns off the adsorbed materials. Multiple-hearth kilns use slowly rotating rabble arms to convey the carbon from one hearth to the next (Figure 2). The kiln may have 6–15 brick hearths situated within a refractory-lined steel shell. Carbon enters the top and falls through openings that are alternately located adjacent to the central shaft (an in-hearth) and the wall (an out-hearth). Carbon dries on the top hearths, pyrolysis occurs on the fired hearths in the middle (which are usually fired with natural gas), and burnoff finishes on the lower hearths. Temperatures reach as high as 1,000°C. As a rule of thumb, about 0.1 m² of hearth area is needed per kg/hr of carbon processed (5). Typically, about 5% of the carbon is burned and needs to be made up.

Regenerated carbon emerging from the exit end of the kiln is quenched with water, and then the slurry, along with makeup carbon, is passed over a vibrating screen. Water and fines go through the screen and are discarded. Carbon retained by the screen is again combined with water, then the slurry is pumped to an empty carbon column. An alternative to a kiln is regeneration with steam, which requires a lower capital investment but is less effective.



▲ **Figure 2.** In a multiple-hearth kiln, spent activated carbon is regenerated by burning off the adsorbed molecules. Rotating rabble arms convey the carbon from one hearth to the next. Source: Adapted from (5).

Zeolite molecular sieves

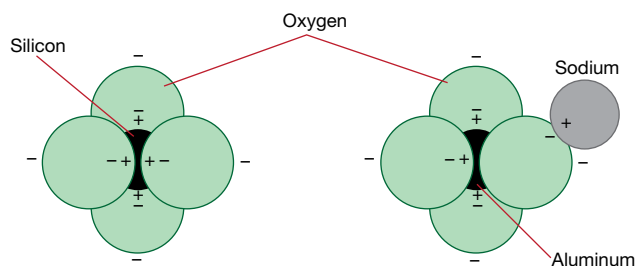
Zeolites are highly crystalline aluminosilicates made from tetrahedral building blocks comprising an atom of silicon or aluminum surrounded by four larger oxygen atoms (Figure 3). Each of the four outer electrons of a silicon atom pairs with one of the two electrons available on an oxygen atom, leaving the other oxygen electron to associate with another silicon atom, thereby extending the lattice. Because aluminum has only three outer electrons, another cation, such as sodium or potassium, is needed to form a stable structure.

There are both natural and synthetic zeolites; the latter are industrially more important. A range of physical properties and, in turn, performance characteristics can be obtained by varying the silicon-to-aluminum ratio, the cation used to achieve charge neutrality, and the crystallization and drying conditions (6). An example is Zeolite A (Figure 4), which has roughly spherical cavities that are each 11 Å in diameter. Other synthetic zeolites include Zeolite X, Zeolite Y, Zeolite L, Zeolite Ω , and Zeolite ZSM-5.

For adsorbate molecules to enter a cavity of the zeolite, they must fit through an aperture on the exterior. Unlike other types of adsorbents, which have a pore size distribution, the aperture size of a particular type of molecular sieve is uniform. Selectivity is determined by the size of the aperture — that is, molecules that fit through are adsorbed, while larger ones are not — and capacity is related to the volume of the cavity. However, aperture size is not an absolute barrier, because atomic vibration allows molecules slightly larger (about 0.5 Å) to squeeze through.

Atomic vibration varies with temperature, which affects the performance of zeolites. Capacity initially increases with decreasing temperature — as with other types of adsorbents (1) — but eventually the reduced vibration makes it more difficult for some molecules to pass through the aperture, and capacity decreases with continued cooling.

Molecular sieves, which are available in bead and powder forms, are classified by their aperture size. Designations include 3A, 4A, 5A, and 13X, with aperture diameters



▲ **Figure 3.** Zeolite molecular sieves consist of atoms of silicon or aluminum, each surrounded by four larger oxygen atoms. Aluminum requires a second cation, such as sodium, to pair with the fourth oxygen atom. Source: Adapted from (6).

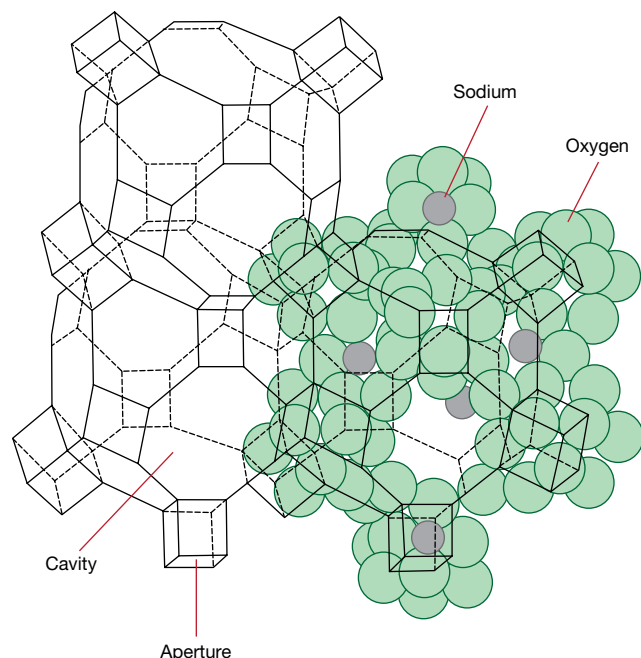
of 3 Å, 4 Å, 5 Å, and 10 Å, respectively. The 3A sieves are employed to remove water from gases and organic liquids, one of the most common uses of molecular sieves. Water-holding capacity is typically about 20% of the weight of the sieve, and the water concentration in liquids or gases can be reduced to as low as 0.035 ppm.

Applications include natural gas dehydration, dehydration of unsaturated hydrocarbons, removal of water from grain alcohol to obtain absolute ethanol, and drying of compressed air. Regeneration of the zeolite is performed in place with hot (200–300°C) gas; to avoid thermal shock, cooling to within 15°C of the service temperature is required before resuming treatment. In general, zeolite molecular sieves are more effective at water removal than silica gel or activated alumina (discussed next).

Silica gel and alumina adsorbents

Silica gel (SiO_2), a polar adsorbent made from colloidal silica, has a surface area ranging from 300 to 850 m^2/g , depending on pore size. Applications include removal of water and other polar compounds from liquid and gas streams (e.g., drying of compressed air in process plants), and humidity control in food products to prevent spoilage. Silica gel is regenerated by heating to 120°C for 1–2 hr (3, 7).

Activated alumina (Al_2O_3), another polar adsorbent, is obtained by removing water from hydrated colloidal alumina. This adsorbent is widely used as a desiccant to dry



▲ **Figure 4.** The crystalline structure of Zeolite A is shown here. The aperture size dictates the size of molecules that are adsorbed. Capacity is related to the size of the cavity. Source: Adapted from (6).

gases and liquids, and for the removal of fluoride, arsenic, and selenium from drinking water. Surface area is about 320 m^2/g , and moisture levels can be reduced to 1 ppm or lower (3, 8).

Polymeric adsorbents

Like activated carbon, polymeric adsorbents adsorb hydrophobic molecules from liquid or gas streams. They are well-suited for removing organics from dilute aqueous feeds, either for pollution abatement or product recovery. Polymeric adsorbents are not true adsorbents, because molecules are captured within the polymer structure as well as adsorbed onto the surface. However, they behave like adsorbents and can be described accordingly.

These polymers are typically supplied as spherical beads, with average particle diameters of 0.4–0.7 mm. They have surface areas of 400–1,200 m^2/g . As with activated carbon, surface area is higher if there is a larger number of smaller pores, but smaller pores may not be suitable for capturing large molecules. A polar solvent such as methanol, ethanol, isopropanol, or acetone can be used to regenerate a polymeric adsorbent.

The most widely used polymeric adsorbent (also called resin) is polystyrene crosslinked with divinyl benzene (PSDVB). Acrylic polymers, although not as popular as PSDVB resins, are also common; the monomer is either acrylic or methacrylic acid, and divinyl benzene is the crosslinker.

Acrylic polymers are sometimes preferred over PSDVB for recovery of large molecules because they have a large number of macropores, but capacity is lower. In addition, acrylics tend to have a higher affinity than PSDVB for less hydrophobic molecules, and some molecules that are very tightly held by PSDVB are more easily desorbed from acrylics. Greater crosslinking in both types of polymers creates a more rigid structure and less swelling upon changeover from water to the regenerating solvent (i.e., the eluting solvent). This change in volume can be as high as 25%, depending on the solvent.

Polymeric adsorbents are more physically robust than activated carbon — that is, they are less prone to attrition and fines generation. Polymers are easily regenerated, without the need for the high temperatures required to regenerate carbon. While initially more expensive, the long-term cost of polymeric adsorbents is usually lower because of their greater durability and avoidance of losses during regeneration.

On the other hand, activated carbon has a higher affinity for a wider range of molecules, which is an advantage for waste treatment applications. However, this lack of selectivity, as well as the difficulty in preserving the structure of adsorbed molecules during desorption, largely precludes the

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use of carbon for recovery of valuable components. Finally, activated carbon is superior for color removal, *e.g.*, in the decolorization of sugar solutions prior to crystallization.

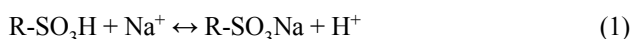
Ion exchange

As the name implies, ion exchange is the exchange of ions in solution with those immobilized on a solid support. In many applications, the objective is to deionize the feed liquid by exchanging cations with protons (H^+) and anions with hydroxyl ions (OH^-). Other ions can also be exchanged — for example, in a water softener, sodium ions replace calcium and magnesium in solution to prevent inorganic scale buildup. Additional applications include:

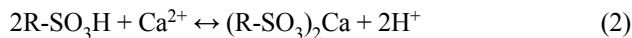
- treatment of boiler feedwater
- production of ultrapure water for semiconductor processing
- demineralization of food products, such as sugar, citric acid, apple and citrus juices, dairy products, and amino acids
- recovery of heavy metals (*e.g.*, gold, nickel) from waste streams
- recovery of uranium from leaching streams
- purification of pharmaceutical products such as antibiotics, vitamins, and other fermentation-derived compounds.

Ion exchange is carried out with the same polymers used as polymeric adsorbents, but with functional groups attached to obtain the required exchange capability. There are four general types of ion exchange resins: strong acid cation, strong base anion, weak acid cation, and weak base anion (Table 2, Figure 5). Exchange occurs as a result of a reversible chemical reaction, and the resin is named to describe its chemical behavior. As such, ion exchange resins are not adsorbents, but most sources discuss the two topics together.

Strong acid resins (Figure 5a) are functionalized with sulfonic acid (SO_3H). The exchange reaction for a monovalent cation, using sodium as an example, is:

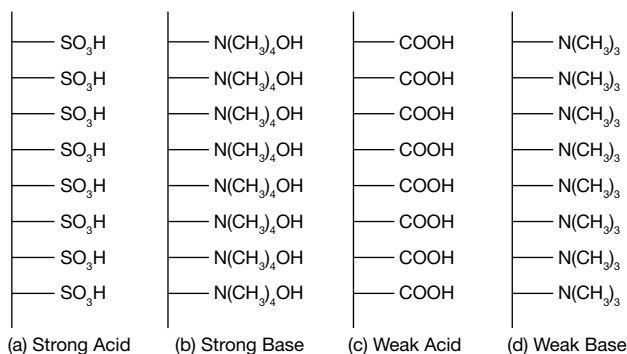


This reaction converts the resin from the hydrogen form to the sodium form. For a divalent cation such as calcium, the exchange reaction converts the resin from the hydrogen to the calcium form:



Like strong acids in general, strong acid resins are highly dissociated, so the cation is available for exchange over the entire pH range. Consequently, strong acid exchangers can split neutral salts. These resins are regenerated with a strong acid such as hydrochloric or sulfuric acid, which drives Reactions 1 and 2 in reverse. Because strong acid resins are highly dissociated, a considerable excess of regenerating acid is needed for complete regeneration, about 130% of the stoichiometric amount.

Maximum reversible swelling, upon conversion from the sodium form to the hydrogen form, may be as high as 10%, depending on the degree of crosslinking — more highly crosslinked resins experience less swelling. As with polymeric adsorbents and ion exchange resins in general, some swelling is desirable to increase the intraparticle diffusion rate; the downside is that a larger column is needed to accommodate a given amount of resin. Exchange capacity ranges from about 1.5 to 2.5 equivalents per liter (eq/L).



▲ **Figure 5.** Ion exchange resins are formed by attaching functional groups to a polymer backbone.

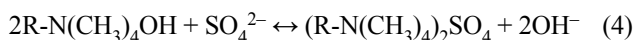
Table 2. The four general types of ion exchange resins are strong acid cation, strong base anion, weak acid cation, and weak base anion.

	Strong Acid	Strong Base	Weak Acid	Weak Base
Functional Group	Sulfonic Acid ($-SO_3H$)	Quaternary Ammonium ($-NR_4OH$; R is usually CH_3)	Carboxyl ($COOH$)	Most Common: Tertiary Amine ($-N(CH_3)_3$)
Operating pH	Entire Range	Entire Range	Above 6	Below 7
Able to Split Neutral Salts?	Yes	Yes	No	No
Amount of Regenerant Required (% of stoichiometric)	130	130	100	100
Reversible Swelling	$\leq 10\%$ ($Na^+ \rightarrow H^+$)	10–30% ($Cl^- \rightarrow OH^-$)	50–100% ($H^+ \rightarrow Na^+$)	20–50% (free base $\rightarrow Cl^-$)
Exchange Capacity, eq/L	1.5–2.5	0.5–1.5	3–5	1–3

Strong base resins (Figure 5b) are functionalized with the quaternary ammonium group NR_4OH , where R is usually CH_3 . Being strong bases, these resins are also highly dissociated over the entire pH range, and can also split neutral salts. For a monovalent anion such as chloride, the exchange reaction is:



Here the resin is converted from the hydroxide form to the chloride form. For a divalent anion such as sulfate, the exchange reaction is:



These resins are regenerated with a strong base, such as sodium or potassium hydroxide; as with strong acid resins, about 130% of the stoichiometric amount is needed for complete regeneration. Swelling, upon conversion from the chloride to the hydroxide form, ranges from about 10% to 30%, again depending on the degree of crosslinking. Exchange capacity varies from 0.5 to 1.5 eq/L.

Table 3. Relative equilibrium constants for Dowex 50, a strong acid PSDVB resin with 8% crosslinking. The reference ion is Li^+ , assigned a value of 1.00. Data are from (7).

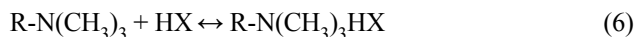
Cation	Relative Equilibrium Constant
Li^+	1.00
H^+	1.27
Na^+	1.98
UO_2^{2+}	2.45
NH_4^+	2.55
K^+	2.90
Rb^+	3.16
Cs^+	3.25
Mg^{2+}	3.29
Zn^{2+}	3.47
Co^{2+}	3.74
Cu^{2+}	3.85
Cd^{2+}	3.88
Ni^{2+}	3.93
Ca^{2+}	5.16
Sr^{2+}	6.51
Ag^+	8.51
Pb^{2+}	9.91
Ba^{2+}	11.5
Tl^+	12.4

Weak acid cation resins (Figure 5c) are functionalized with carboxyl groups. These resins behave like weak acids, with little dissociation at low pH and increasing ionic character as the pH increases. Capacity, a strong function of pH, is very limited below about pH 6 because the lack of dissociation leaves few ions available for exchange. Weak acid resins will not split neutral salts, but they are effective with the salt of a weak acid and a strong base, e.g., sodium bicarbonate:



Because weak acid resins have a higher affinity for H^+ ions than strong acid resins, regeneration is easier; usually a stoichiometric amount of acid is sufficient. Weak acid resins have capacities ranging from about 3 to 5 eq/L, and they swell as much as 100% upon conversion from the hydrogen form to the sodium form.

Weak base anion resins (Figure 5d) are functionalized with a primary, secondary, or, most commonly, tertiary amine (e.g., $\text{N}(\text{CH}_3)_3$). Weak base resins do not contain ionizable groups, but instead act as acid adsorbers:



Like weak acid exchangers, performance varies strongly with pH. Capacity is greatly diminished above pH 7, then increases with decreasing pH. Unlike strong base resins, complete regeneration is achieved with a stoichiometric amount of base. Weak base resins have capacities of about 1 to 3 eq/L, and they swell 20–50% upon conversion from the free base to the chloride form.

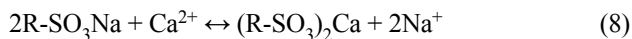
The ion exchange reaction is reversible, and the equation for the equilibrium constant is written in the usual way. For example, for exchange of Na^+ and H^+ using a strong acid resin (from Reaction 1):

$$K_{eq} = \frac{[\text{R-SO}_3\text{Na}][\text{H}^+]}{[\text{R-SO}_3\text{H}][\text{Na}^+]} \quad (7)$$

Equilibrium constants characterize the affinity of one ion vs. another for a particular resin. As an illustration, Table 3 gives relative equilibrium constants for exchange of several cations using a strong acid exchange resin, with the reference ion Li^+ assigned a value of 1.00.

The relative affinity of one ion over another is indicated by the ratio of the equilibrium constants. For example, consider a strong acid cation exchanger in the sodium form used as a water softener. From Table 3, both Ca^{2+} and Mg^{2+} will replace Na^+ on the resin because each has a higher relative equilibrium constant than Na^+ . The exchange reaction for Ca^{2+} is:

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The equilibrium constant for this reaction is the ratio of the relative equilibrium constants given in Table 3 for Ca^{2+} and Na^+ , or $5.16/1.98 = 2.61$. An analogous reaction can be written for Mg^{2+} , with an equilibrium constant of $3.29/1.98 = 1.66$. The softener is regenerated with aqueous sodium chloride (*i.e.*, brine), which drives these reactions to the left.

In general, the equilibrium constants given in Table 3 for monovalent cations increase with decreasing size of the hydrated ions, because ease of access to the resins improves. Typically, divalent cations are more tightly held than monovalent ones and the affinity is greater than suggested by the equilibrium constants (8).

Ion exchange operation

Figure 6 is a flowsheet for a deionization process that uses two cation and two anion columns. The diagram shows Cation 1 and Anion 1 as the online columns, as indicated by the flow path depicted in green, while Cation 2 and Anion 2 are being regenerated. When breakthrough occurs, the online column is removed from service and regenerated, and the freshly regenerated column is placed in operation.

More columns might be used in an actual commercial installation, to provide a guard, an installed spare, or higher throughput with parallel banks (1); for clarity, these are omitted from Figure 6.

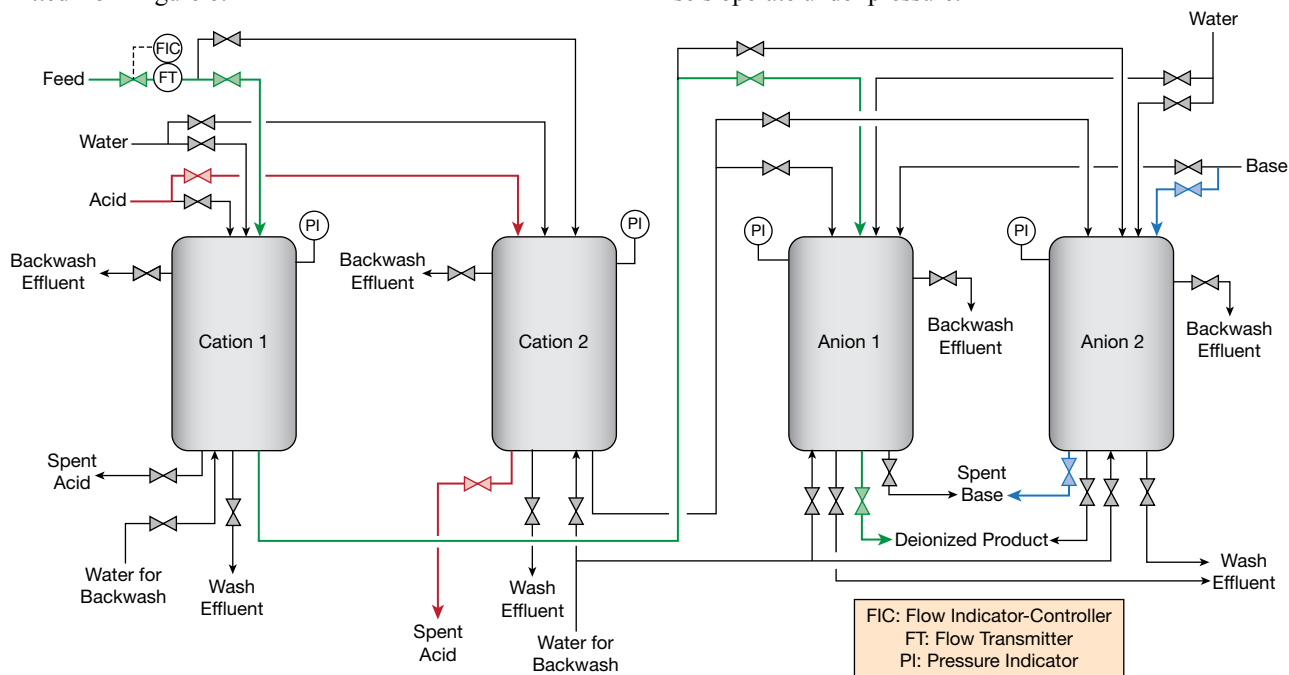
The flowsheet allows implementation of the following operating sequence, which is typical for ion exchange processes:

1. Run process liquid to breakthrough.
2. Rinse with water.
3. Regenerate with acid (cation column) or base (anion column).
4. Rinse with water.
5. Backwash with water, from bottom to top. (This fluidizes the bed, which removes any air pockets that may have formed, thereby increasing efficiency.)

For some applications, including purification of some fermentation products, the pH of the cation column effluent can be monitored to detect breakthrough. When a proton is exchanged for another ion, a salt is converted to its acid form, reducing pH. Conveniently, breakthrough is accompanied by an increase in pH, which is easily detected.

Similarly, the anion column breakthrough can be monitored by conductivity in some cases. The anion resin removes the remaining ions from solution (cations were removed in the upstream cation exchanger), so the effluent conductivity is very low. Upon breakthrough, anions appear in the effluent and the conductivity increases.

In modern plants, the numerous valves on the flowsheet in Figure 6 are operated automatically by a process computer or programmable logic controller (PLC). Note the absence of interstage pumps, which are not needed because the vessels operate under pressure.



▲ **Figure 6.** In this ion exchange process, the two cation columns alternate between service and regeneration, as do the two anion columns. The colored lines indicate active flow and open valves for Cation 1 and Anion 1 in service, while Cation 2 and Anion 2 are being regenerated. Red, green, and blue signify acid, process liquid, and base, respectively.

Concluding remarks

Adsorption is a versatile unit operation, and a wide variety of adsorbents are available for a diverse range of applications. By applying the basic principles and other information provided in this article and in Part 1 of this series (1), adsorption can be part of an economic purification or waste treatment process, reducing the solute content of gases or liquids to ppm levels.

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