CHAPTER 10.13

Ion Exchange Technologies

Brandon Kern

Ion exchange processes can be used for extractive metallurgy in situations where ionic concentrations are relatively low, generally with a practical upper bound in the thousands of parts per million (tenths of a percent) of metal concentration in solution. Some overlap of application space exists between solvent extraction and ion exchange for metals extraction; at lower concentrations where solvent extraction encounters diminishing return, ion exchange processes find increased removal efficiency. As such, in many applications, ion exchange processes are considered for final polishing applications; however, some key areas have found good success in utilizing ion exchange for primary recovery. These applications are discussed in greater detail herein.

The techniques that use ion exchange technology in mining are multiple and diverse. Technology for mining applications is usually decided according to the recovering techniques being used, including in situ leach (ISL), alkaline or acid leaching, resin-in-pulp (RIP), and heap leaching. Each technique requires specific characteristics for the ion exchange resin (IER) technology.

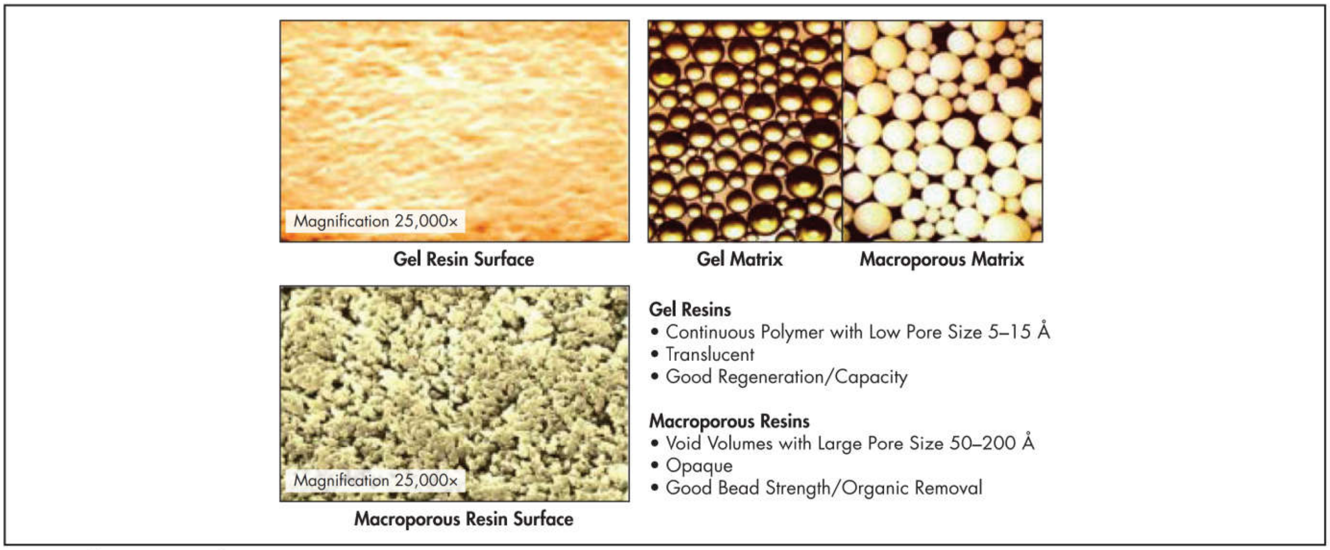
The bulk of ion exchange processes for primary recovery generally focus in one of three different areas: base metals mining (copper, cobalt, nickel), uranium mining, and gold recovery. However, the application of ion exchange also extends to the recovery of high-value, low-volume products, such as rare earth metals or platinum group metals, and more recently in the processing of lithium-containing brines. Depending on the desired separation, different IER types and chemistries are deployed. Generally, the major design parameters are resin morphology (gel versus macroporous), resin bead size and size distribution, and resin functional group.

The copolymer backbone of IERs (usually styrenic, but occasionally phenolic or acrylic polymers) dictate many of their physical properties. An exhaustive summary of the different types of ion exchangers and their properties is outside the scope of this handbook but is extensively reported by other sources (Dorfner 1991; Helfferich 1962; Kunin 1972). Important to mining applications, however, is the distinction between gel copolymer matrices and macroporous polymers. Gel polymers consist of a continuous polymer phase that can be swollen with water or process fluid to occupy roughly 50% of the resin's volume. This water of hydration is responsible for shuttling ionic species into and out of the resin bead to facilitate contact with the charged ion exchange functional groups contained therein. Macroporous IERs are produced of similar polymer that has microchannels and voids present throughout; these channels provide for increased access for contact between ions in solution and the resin's functional groups. In practice, gel copolymer IERs tend to offer higher total exchange capacity than their macroporous counterparts, whereas macroporous resins tend to have a higher overall mechanical stability and fouling resistance. For these reasons, gel resins are often the best choice for treatment of clarified solutions where their higher capacity can be utilized. Macroporous resins are best suited to applications where physical stability and fouling resistance are important, for example, RIP applications. Figure 1 illustrates some of the physical structure differences between macroporous and gel copolymers.

A wide variety of different ion exchange functional groups can be utilized for mining processes, each with their own specific advantages and applications. The most typical standard anion and cation exchange functional groups are shown in Table 1. A separate family of IERs use chelating agents that are bound to the copolymer matrix; these chelating resins can utilize a wide variety of different chemistries to selectively bind certain classes of metals and can have specific utility in mining applications.

Most hydrometallurgical applications of ion exchange involve the use of strong base anion exchangers because many of the metals of interest form stable anionic species in the leachate stream. Some metals separations, however, may require the use of selective IERs (generally referred to as *chelating* resins) that incorporate different chemical functionality tailored to selectively remove one particular species, frequently in the presence of high background concentrations of undesired species. Many of the developments in this area involve the creation of new resins with different functionality, higher capacity, and better selectivity, which may involve the use of different chelating chemistry or adsorbent technology.

Brandon Kern, Technical Service Specialist, Ion Exchange, The Dow Chemical Company, Midland, Michigan, USA



Gel Resins
- Continuous Polymer with Low Pore Size 5–15 Å
- Translucent
- Good Regeneration/Capacity

Macroporous Resins
- Void Volumes with Large Pore Size 50–200 Å
- Opaque
- Good Bead Strength/Organic Removal

Courtesy of Dow Water Solutions

Figure 1 Gel versus macroporous resins

Table 1 Typical functional group families of ion exchange resins

Ion Exchange Resin Class	Typical Structure
Strong acid cation exchanger	∿∿∿ SO_3H
Weak acid cation exchanger	∿∿∿ COOH
Weak base anion exchanger	∿∿∿ NR_2
Strong base anion exchanger (chloride form)	∿∿∿ $NR_3^+Cl^-$
Copolymer backbone (styrenic, acrylic, or phenolic)	∿∿∿

Table 2 Characteristics of resin families used in metallurgy

Chelating Resins	Strong or Weak Base Anion Resins	Strong Acid Cation Resins
Highly selective	Used when total anion removal needed	Used for total cation removal
Work in concentrated feeds	Effective for separation of metallochloride complexes in chloride matrices	Macroporous, Gaussian particle size distribution generally favored (fouling resistance)
Selectivity is tunable with pH	Frequently used for $AuCN^-$ and uranium applications	Medium to high levels of sulfonation (high capacity)

Some general descriptions of the different resin families used in metallurgical processes are described in Table 2.

URANIUM EXTRACTION USING ION EXCHANGE

Strong and weak base anion exchangers have been employed in uranium recovery from ISL systems since the 1950s (Benes et al. 2001). Early divergence in ISL philosophy led to the development of mainly carbonate leach systems in North America, whereas sulfuric acid leaching techniques have generally been preferred in other geographies. The role of the IER is to selectively fix uranium from the leach solution and then recover it by eluting from the resin for further processing. Strong base anion resins are now mainly used to recover uranium, both from acid leach and from carbonate leach circuits,whereas use of a cation exchange resin would permit loading of the UO_2^{2+} directly, and loading competition would exist between the uranium and other cations (iron, calcium, magnesium, etc.), which are likely at much higher concentrations. Recovery of anionic uranyl sulfates and uranyl carbonates allows for a higher-purity product as it mitigates interference from these other contaminants. The specific resin functional group and the polymer matrix can be varied according to the solution composition to further limit the influence of interfering anions.

Carbonate Leaching

Most common in the United States are ISL systems involving dissolution of uranium into sodium carbonate solutions, as opposed to sulfuric acid solutions that are practiced in other geographies. The following reactions are relevant to uranium ion exchange in carbonate leach systems:

$$UO_2^{2+} + O_2 \Leftrightarrow UO_3 \qquad \textbf{(EQ 1)}$$

$$UO_3 + 2Na_2CO_3 + H_2O \Leftrightarrow Na_4UO_2(CO_3)_3 + 2NaOH \qquad \textbf{(EQ 2)}$$

$$2(R^+)_2CO_3^{2-} + UO_2(CO_3)^{4-} \Leftrightarrow (R^+)_4UO_2(CO_3)_3^{4-} + 2CO_3^{2-} \qquad \textbf{(EQ 3)}$$

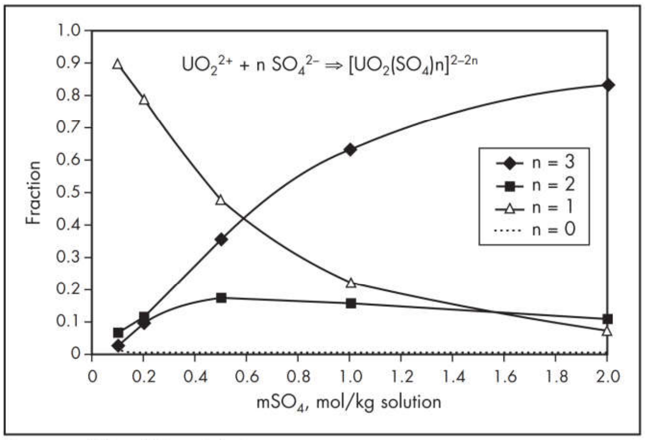
Alkaline conditions result in the formation of a quadrivalent anionic carbonate complex. This approach has dual advantages of both yielding a complex with very strong selectivity for both strong and weak base IERs as well as the avoidance of loading many metal impurities that do not form anionic complexes. Leaching of loaded resin is typically performed using an eluent composed of a mixture of sodium carbonate and sodium bicarbonate salts (Zaganiaris 2009).

Acid Leaching

Acid leach is practiced using sulfuric acid (H_2SO_4). Uranium in the leach solution forms uranyl sulfate complexes according to the following reactions:

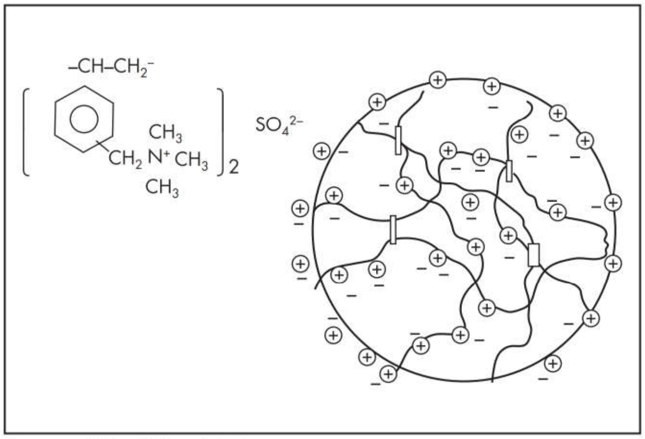
$$UO_2^{2+} + SO_4^{2-} \Leftrightarrow UO_2(SO_4) \qquad \text{Log } K_1 = 2.98 \qquad \textbf{(EQ 4)}$$

$$UO_2^{2+} + 2SO_4^{2-} \Leftrightarrow UO_2(SO_4)_2^{2-} \qquad \text{Log } K_2 = 3.83 \qquad \textbf{(EQ 5)}$$



Courtesy of Dow Water Solutions

Figure 2 Uranyl complex ionic form as a function of sulfate concentration



Courtesy of Dow Water Solutions

Figure 3 Donnan potential

$$UO_2^{2+} + 3SO_4^{2-} \Leftrightarrow UO_2(SO_4)_3^{4-} \quad \text{Log } K_3 = 3.45 \quad \textbf{(EQ 6)}$$

And in addition,

$$HSO_4^{-} \Leftrightarrow H^+ + SO_4^{2-} \qquad K_d = 0.012 \quad \textbf{(EQ 7)}$$

Both divalent and quadrivalent anionic sulfate complexes are formed with uranium in acidic leach systems. Optimum pH for recovery of uranium is around 2 with a concentration in sulfate of approximately 0.25 mol/kg in solution; under these conditions, formation of the quadrivalent complex is preferred. Loading of the uranyl sulfate complexes onto anion exchange resins takes place according to the following reaction:

$$2(R^+)_2\,SO_4^{2-} + UO_2(SO_4)_3^{4-} \Leftrightarrow (R^+)_4UO_2(SO_4)_3^{4-} + 2SO_4^{2-} \quad \textbf{(EQ 8)}$$

ISL circuits in sulfate service are most commonly eluted using sulfuric acid. From Equations 1 to 3, the relative fractions of the different species can be calculated for a given concentration of uranium and SO_4^{2-} in solution. Figure 2 shows the ionic form of the uranyl as a function of the concentration of sulfate. This illustrates that the uranium enters the resin as a neutral species. At higher concentration in sulfate, the loading of uranium in the resin decreases, which corresponds to the point where uranium is in its negatively charged form.

The explanation for this phenomenon resides in the concept of the Donnan potential, described graphically in Figure 3. The Donnan potential has one immediate consequence for electrolyte sorption: It repels co-ions from the exchange resin and thus prevents the internal co-ion concentration from rising beyond an equilibrium value, which is usually much smaller than the concentration in the external solution. Thus, only the neutral species can easily enter the resin matrix while charged molecules or ions are repelled. This concept is the basis for the concept known as *ion exclusion*.

Chloride Effect

Uranyl sulfate enters in the matrix and forms a stable complex with the sulfate attached to the functional group. So the mechanism of uranium fixation can also be seen as a chelating mechanism. Chloride impairs the loading of the resin despite a lower affinity in comparison with the sulfate, which can be explained as the chloride causing an interruption in the formation of uranyl sulfate complexation with nearby sulfate groups. As such, modest increases in chloride concentration can result in significant impairment to loading capacity.

Silica Fouling

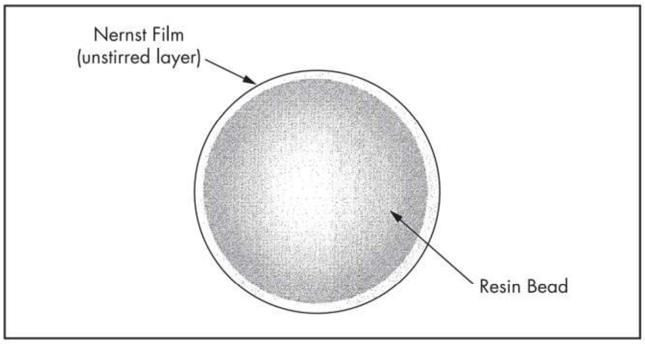
In many mining applications, silica fouling becomes critical. If silica fouling is not overcome, specific issues, such as irreversible fouling, impaired kinetics, resin coalescence and pressure drop, and so forth, ultimately may lead to shutdowns of production. Silica fouling is considered a priority in RIP technology because the ore is in contact with the lixiviant solution and the resin, the ore contains silica, and the concentration of silica is more important.

The mechanism of silica fouling is not completely understood, but many hypotheses exist. Silica is not very soluble at the low pH commonly used in the mining process. As pH of a system increases above about 8, silica solubility increases dramatically. Perhaps even more important is the morphology of the silica with varying pH; silica has many somewhat soluble gel phases at different pH levels.

Consideration of these factors, along with the Donnan effect described earlier, leads to an interesting discussion of the various portions of the polymer bead. If one dissects the main layers of the bead into the Nernst, or unstirred, layer and maintains the remainder of the bead as internal to the Nernst layer, a plausible hypothesis emerges relating to the depth of silica fouling on the resin. Silica generally accumulates in IERs in a layer very near the surface of the bead, consistent with the location of the Nernst film, as shown graphically in Figure 4.

Contacting Systems

These ion exchange systems can utilize different forms of resin contactors, depending on the application. Most common for uranium processing are fixed-bed systems configured in a lead–lag arrangement. In these systems, clarified leach liquor is passed through a lead column, followed by a polishing column, while a third column is being regenerated. As the lead column becomes exhausted, it is removed from service and eluted; consequently, the lag column takes the lead position and the third, freshly regenerated column is placed in the lag position. This arrangement ensures lower leakage (loss of uranium in the barren solution) and also results in improved loading of uranium onto the lead bed.



Adapted from Dorfner 1991
Figure 4 Layers of the ion exchange bead

In some instances, uranium ore bodies are of sufficiently high grade as to permit use of traditional grinding and leaching processes for uranium extraction. For such operations, uranium-containing ore is ground and leached with sulfuric acid, which leaches the uranium in the form of uranyl sulfate complexes. The resultant slurry may then be clarified prior to ion exchange by means or resin-in-leach (RIL) contacting, or the slurry (pulp) may be contacted directly with the ion exchange media via an RIP process. RIL applications may make use of many different fluidized-bed continuous ion exchange contacting systems, for example, the Higgins Loop (Higgins 1957) or the Porter system (Porter 1975). RIP processes generally use a series of stirred-vessel contactors through which the resin and pulp are exchanged in a countercurrent fashion. Uranium-rich pulp is fed into subsequent contactors from left to right, decreasing in grade through ion exchange in each of the contacting vessels. IER is screened and transferred from right to left, countercurrent to the pulp, such that as the resin loads, it is transferred into sequentially higher-concentration pulps, thus enhancing its loading efficiency. With RIP contactors, the physical stability and robustness of the selected IER is especially important, as physical contact with the pulp can crush and abrade resins with time (Yahorava et al. 2009). Additionally, larger-bead resin products are preferred as they are easier to separate from the pulp via screening.

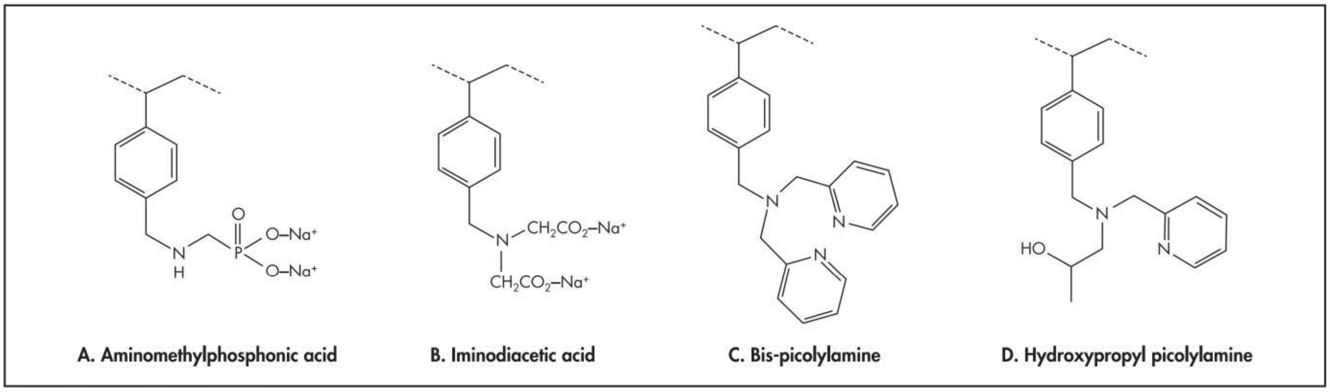
RECOVERY OF BASE METALS

Base metal recovery can be attained using a few different ion exchange approaches. Many of the transition metals react in the presence of high halogen concentrations to form anionic metal–halogen complexes. This feature can be exploited in the presence of hydrochloric acid, where metal–chloride complexes form based on their affinity for chloride complex formation and the concentration of hydrochloric acid in the solution. This topic is covered in greater detail elsewhere (Kraus 1956). Separation of chloride-complexed metals can be effectively achieved through the use of anion exchangers; some control of the metals that are removed versus those that stay behind in solution can be influenced by changing the concentration of hydrochloric acid (HCl) being used in the process.

Other options for base metals recovery include the use of IERs that possess selective chelating ligands (Mendes and Martins 2004). Some chelating ligands, such as aminomethylphosphonic acid or iminodiacetic acid can be characterized as having improved selectivity for divalent and multivalent cations over monovalent species such as sodium and potassium. As a result, these resins can be used for selective metals recovery in the presence of brines. Other chelating ligands have high specificity for select classes of metals. For example, bis-picolylamine (BPA) functionalized chelant resins have demonstrated exceptional selectivity for copper and nickel (Grinstead 1984); and hydroxypropyl picolylamine (HPPA) functionality offers similar selectivity but improved regeneration characteristics (Grinstead 1979). The chemical structures of these functional groups are illustrated in Figure 5.

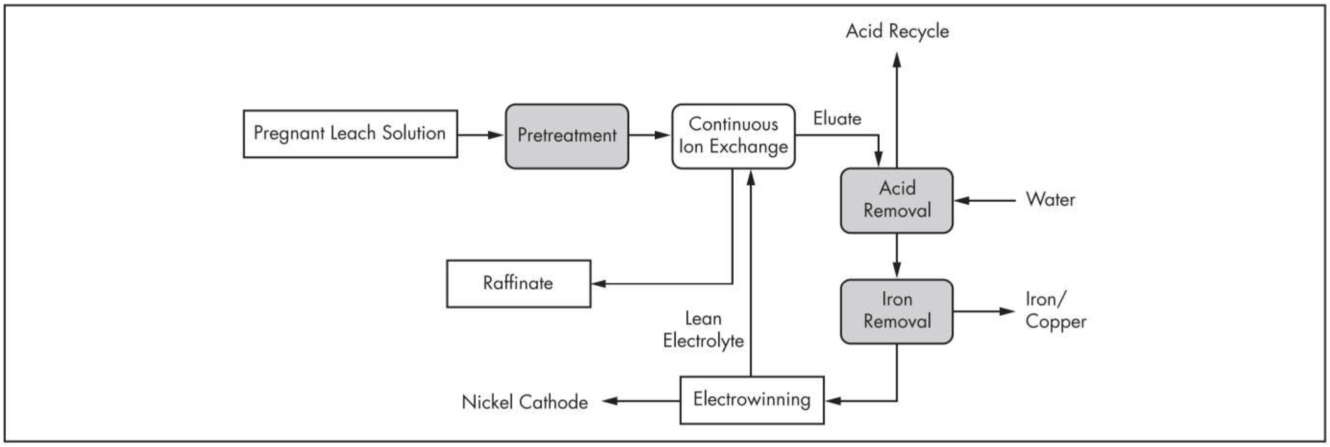
Lateritic Nickel Recovery by Ion Exchange

The hydrometallurgical processing of laterites to melting-grade nickel is complicated because of the high concentration of iron present in the pregnant leach solution (PLS). The use of chelating IERs for the selective removal of nickel from this high-iron PLS offers several advantages over more traditional techniques; for example, it allows for the selective removal of nickel from lateritic PLS with most iron reporting to the barren. Contaminating iron is then efficiently scrubbed from the resin using reducing agents and polished from the eluate with iron-selective resins. The resulting high-purity nickel eluate is ready for downstream electrowinning or precipitation without the need for further concentrating. Figure 6 details the individual development steps for a typical nickel laterite project and where iron removal is required. This approach to lateritic nickel recovery is facilitated by a BPA-functionalized ion exchanger that has particularly high selectivity for nickel and



A. Aminomethylphosphonic acid B. Iminodiacetic acid C. Bis-picolylamine D. Hydroxypropyl picolylamine

Source: Kern and Hwang 2014
Figure 5 Chemical structures of chelating functional groups



Courtesy of Dow Water Solutions

Figure 6 Laterite process utilizing ion exchange

copper present in acidic feeds. The picolylamine functional group binds copper especially strongly, such that any copper loaded from the nickel laterite ore does not interfere with the nickel elution step, which can utilize up to 20% sulfuric acid as an eluent. Stripping of copper from the resin requires use of an alkaline ammonium sulfate solution. The ammonia competitively binds the copper ions into a coordination complex that interrupts the interaction of the BPA functional group with the copper ions, facilitating its removal from the resin.

Continued development of resins for this application has introduced the HPPA functionality, which functions in a similar capacity. This resin possesses a somewhat lower affinity for copper than the BPA functionality, which in turn permits stripping of copper from the resin using 20% sulfuric acid. This feature makes the sequential elution of copper, nickel, and cobalt from the resin more challenging, as the difference in selectivity between the different metals is less pronounced. However, the ability to acid-strip copper makes this resin significantly more convenient to use in conventional acidic copper-processing applications.

Ion Exchange for Processing Gold

In the operation of many cyanide-leached gold mining facilities, activated carbon is used as an economical means for separation of gold using carbon-in-pulp contacting approaches. However, IERs offer some advantages over carbon for gold processing. Specifically, current gold-selective IERs have a higher selectivity of gold cyanides over base metal cyanides, thereby allowing for higher-purity gold products as a result of processing. Additionally, the higher capacity and faster kinetics of ion exchange lend themselves to improved processing (Lewis and Bouwer 2000). These benefits are especially pronounced in the processing of poor-grade or carbonaceous (preg-robbing) ores.

In cyanide leach systems, gold exists as an aurocyanide complex $[Au(CN)_2]^-$. Being an anion, these species can be readily removed from solution via capture onto an anion exchange resin. Protonated tertiary amine weak base anion resins can be used for this application, and the sorbed aurocyanide species can be eluted from the resin using a solution of sodium hydroxide. Following elution of the weak base moiety using base, the resin needs to be reprotonated through treatment with dilute acid. Alternatively, a quaternary amine strong base anion resin can be employed. Although these resins lack the requirement of post-elution acid treatment, they can be more difficult to regenerate. Regeneration requires an acidified thiourea solution to break the ionic interaction between the aurocyanide complex and the functional group.

One potential drawback to standard anion exchange resins (especially weak base resins) is that other anionic metallocyanide complexes compete for loading sites with the aurocyanide complex. As such, specially functionalized anion exchange resins have been developed that optimize preferential selectivity for loading of aurocyanide species over other metallocyanide anions (Johns and Marsh 1993). These resins allow for a significant improvement in the quality of gold extraction from comparably poor quality leachate.

ION EXCHANGE SYSTEM DESIGN

Although the applications of ion exchange for liquid separations are numerous and varied, there are some general guidelines that hold true across multiple applications. Because the ion exchange process is fundamentally an equilibrium interaction between the resin's functional sites and the solution's ionic composition, flow characteristics that impact residence time and contact efficiency between the resin and the solution are important parameters to understand. Although there are, of course, exceptions to every rule, the following are some general guidelines that broadly apply to a variety of ion exchange applications.

Column Design and Flow Considerations

Ion exchange depends on efficient contact between solution and resin. The kinetics of the ion exchange process itself are generally quite rapid, so the rate-limiting step is often related to the efficiency with which the resin is contacted by solution. Generally, these flow characteristics consist of a combination of bulk flow rate (expressed in bed volumes per hour [BV/h] or empty-bed contact time) and linear flow velocity. Most IER systems operate in a broad range between 5 and 25 BV/h, depending on the characteristics of the solution being treated. For example, a system designed to treat a flow rate of 50 m^3/h with a bulk flow rate of 10 BV/h would require 5 m^3 of installed resin.

Another component of flow rate is the liner velocity, which refers to how quickly the solution is passing a given point in the resin bed. Flow velocities generally cover a broad range between 5 and 60 m/h. In a fixed ion exchange column, there is a stagnant film or unstirred layer of liquid around the individual resin beads, even as the bed is under flow. The size of this stagnant film layer depends on the solution velocity across the resin bed, with higher velocities generally resulting in smaller film layers. It is diffusion of ionic species into and out of this stagnant film layer that accounts for a sizable portion of the kinetic rate for ion exchange. Because smaller stagnant films yield shorter diffusion lengths to reach the ion exchange functionality of the resin bead, higher flow velocities can yield more efficient ion exchange kinetics. However, this effect must be balanced with other factors such as IER bead size, overall kinetic hindrance, and the size of ionic species being removed. Generally, most systems operate within a broad linear flow range of 5 to 60 m/h. Slow flow rates run the risk of inefficiency because of solution channeling, while operation on the fast end of the range risk excessive pressure drop across the resin bed. For a defined bulk flow rate, the linear flow rate can be modified by changing contact equipment dimensions. For example, a long, narrow IER bed has a faster linear flow velocity than a wide, short bed that contains the same amount of resin.

Most IERs suggest a minimum resin bed depth ranging from 0.75 to 1 m. Maximum resin depth is usually dictated by operational limitations on pressure drop across the vessel and functional limitations on equipment size. Generally, commercial ion exchange column dimensions are optimal at a height-to-width ratio (H:W) between 2:1 and 4:1. Pilot systems and other applications with lower flow rates generally have a H:W ratio exaggerated beyond 4:1 to maintain the necessary minimum resin depth as specified.

Other Treatment Factors

Besides flow rate and treatment volume, other system design considerations include composition of both the feed solution and the desired regenerant, as these dictate any materials compatibility or corrosion concerns when specifying equipment. In general, most IERs are sensitive to oxidative environments and undergo de-crosslinking or other oxidative damage in their presence. As such, potential oxidizing agents (hypochlorite, permanganate, etc.) should be avoided for contact with ion exchange. Nitric acid in particular can be damaging to IERs and under certain circumstances can react to form unstable compounds. Therefore, the use of ion exchange for treating solutions containing nitric acid is not advised because of safety concerns.

With the exception of RIP systems and fluidized beds, some type of pretreatment is generally required for the removal of suspended solids. Suspended solids can quickly become entrapped in ion exchange beds, increasing pressure drop and compromising contact between the solution and the resin. Sand filters or some other backwashable media filtration are generally preferred for many hydrometallurgy applications, though the use of bag filters or replaceable cartridge filtration may also be feasible depending on the application and filtration needs. Higher solids applications may require the upstream use of a technique capable of handling higher solids content, such as a hydrocyclone or dissolved air flotation.

Cost Considerations

Ion exchange systems for mineral processing applications can vary greatly in cost and scope, from simple systems treating small flow rates to huge, multi-vessel automated systems treating hundreds of cubic meters of solution per hour. As such, system capital expenditure for treatment in the mining space can range from thousands or tens of thousands of dollars to tens of millions of dollars, simply based on the size of the system and its complexity. Extent of process automation, equipment design (fixed bed versus moving bed, RIP, etc.), flow rate for treatment, materials compatibility and corrosion control, pretreatment requirements, and process redundancy are all factors that have a significant impact on the capital requirements for ion exchange system implementation. Furthermore, regeneration frequency and efficiency, chemical handling, and waste and wastewater management factors need to be considered in determining operational costs for an ion exchange facility. Discussions with original equipment manufacturers regarding their ion exchange systems early in the planning phase can help manage cost expectations through evaluation of these relevant factors.

SUMMARY

IERs can be used for a variety of different separations in extractive metallurgy, particularly in cases where low-grade ores are being processed or otherwise low concentrations of metal ions are being recovered. The three major application areas (uranium, gold, base metals) have been covered in greater detail in this chapter. Other application areas in mining use IERs, although in many of these cases, the ion exchange processes tend to be used for secondary recovery of side streams, as opposed to the main technology for primary recovery. However, in some of these applications, ion exchange has found a comfortable niche, including the processing of platinum group metals, fractionation of rare earth metals, and lithium extraction from brines. As lower-grade ore-processing gains momentum as a result of depletion of higher-grade stocks, ion exchange technologies will likely find continued adoption for their ability to effectively process the lower-concentration leachates that result.

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