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ION EXCHANGE RESINS FOR URANIUM RECOVERY: THE DURABILITY QUESTION EXPLORED

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ABSTRACT

The recovery of valuable metals from solutions and slurries can be greatly improved by using ion exchange resins. Resins found application in the uranium industry, the precious metal industry, e.g. gold and the PGM's, and the base metal industry, e.g. copper, nickel and cobalt. The use of the resin-in-pulp (RIP) process improves overall metal recoveries from slurries, especially in the case of ores with poor filterability where the difficult and expensive solid-liquid separation step can be eliminated.

RIP technology allows the exploitation of low-grade pulps and tailings which may have been previously considered as economically unattractive. An added benefit of the RIP process is a reduction in the negative environmental effect of entrained metal in solid waste residues.

The loss of resin and the associated cost is an important consideration of a RIP project. Projects with low-grade pulps are especially sensitive to resin loss. Resin degradation occurs as a result of the harsh physical environment (abrasive pulps, pumping, screening and agitation) as well as osmotic shock due to the constantly varying chemical environment (extraction under mildly acidic conditions, elution under strongly acidic conditions).

It is not practical or economically feasible to evaluate the resin loss of each project in a large-scale continuous plant. For this reason, various accelerated laboratory-scale durability tests and demonstration plants have been developed to evaluate the relative durability of different resins. This paper investigates the various tests that are currently available and comments on their applicability.

INTRODUCTION

Ion exchange resins have been used to recover uranium since the 1950's. The first generation of uranium mines in the western world used mainly fixed-bed ion exchange. During the 1970's, several continuous ion exchange processes were adopted, such as the Porter System (Rossing Uranium in Namibia) and the NimCIX (Vaal Reefs South in South Africa). These fluidised bed systems had a significant advantage over fixed-bed systems in that they could handle some solids, thereby decreasing the need for perfect solid-liquid separation.

The majority of operating uranium plants employ sulphuric acid leaching, followed by upgrading and purification via ion exchange and/or solvent extraction, followed by final precipitation of yellow cake.

Countries in the Former Soviet Union states have always been using RIP for the recovery of both gold and uranium. The main advantage of RIP over fixed-bed and fluidised-bed operations is that no solid-liquid separation is required, resulting in a major capital cost savings. Furthermore, it has the potential to combine the recovery and purification steps, resulting in further cost savings. There is also the potential for improved recovery from ores that are difficult to settle and filter and where water balance issues prevent efficient washing. RIP allows the exploitation of low grade ore-bodies that may have been previously considered as uneconomical due to a simplified flowsheet and higher recoveries.

The use of RIP in the Western World for the recovery of uranium, gold and base metals is still very limited. Numerous studies have been conducted, with very positive outcomes. However, the expected resin loss remains one of the major uncertainties. The resin loss that is experienced on a plant is not only dependent on the durability of the resin itself, but also on the plant design, nature of the ore and management of the resin inventory. Various plant designs exist, including air-agitated pachucas and mechanically agitated tanks, arranged in either carousel or continuous configuration. The manner in which resin is transferred between stages is also very important and designs include airlifts, pumping and eductors. The geology of the orebody plays an important role, since uranium occurs in different types of minerals. Some ores are considered as harsh, especially siliceous ores such as the Witwatersrand gold ores in South Africa. Higher resin losses can be expected for a RIP operation on such ores. Other ores have a higher clay content and will be less harsh on the resin.

Resin suppliers have invested greatly to ensure the supply of improved products to the RIP market. In addition to confirming the chemical suitability of a product (loading and elution characteristics), the mechanical durability of products has to be tested. The overall suitability of a resin for a specific application has to be confirmed at an early stage of the project, since it is costly and impractical to test several resins on a large scale. For these reasons, a lot of developmental work has been done by resin manufacturers, test-laboratories and others around the world in the quest for quick, easy, inexpensive and accurate tools to predict the resin-loss for a full-scale operation. Failing that, such tools should at least provide a reliable manner of classifying different resins.

METALLURGY

A simplified flowsheet of a uranium RIP plant is shown in Figure 1. Leached pulp enters the plant via the first contactor, from where it moves via gravity to the next reactors, until barren pulp leaves the plant from the last reactor. Fresh/eluted resin enters the plant via the last reactor and moves counter-currently to the pulp through the plant.

Loaded resin from the first reactor is transferred to a dedicated elution column where it is contacted with sulphuric acid. The uranium-rich eluate continues to further purification via solvent extraction or directly to precipitation. Entrained acid and eluate is displaced through a waterwash, after which the eluted resin is returned to the last reactor of the adsorption section.

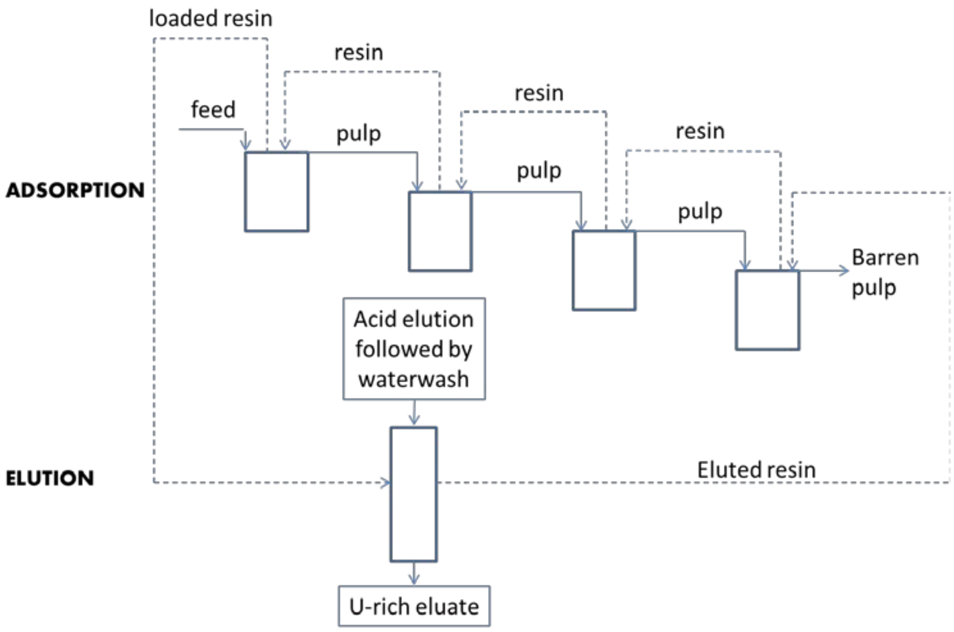

Figure 1: Schematic representation of RIP circuit

Resin Selection Parameters

The desirable characteristics of an ion exchange resin vary according to the application(3). In all cases, the general requirements are:

- High operating capacity;
- Efficient elution/regeneration;
- Service life durability.

A high operating capacity refers to the maximum uranium loading that can be achieved. At the same time, the co-loading of impurities should be minimised.

Efficient elution/regeneration includes the ability to use readily available and economical reagents to elute the valuable metal into a small volume, within a reasonable time, and to a low residual metal content.

The focus of this document is the third characteristic, namely service life durability. The life of a resin can be reduced by various factors, both chemical and physical.

Chemical factors include organic fouling, oxidation and osmotic shock. Resins, especially strong base anion exchange resins, are prone to organic fouling. Such organics may originate from a number of sources, e.g. the raw water used for dissolution of the uranium, the ore itself, oil-leaks from motors, and overdosing of certain chemicals upstream of the resin plant. Strong oxidants, such as free chlorine, will attack the backbone of the resin, thereby decreasing the stability of the resin-structure. Osmotic shock, due to swelling and shrinking when exposed to acidic and alkaline conditions, weakens the backbone of a resin.

However, breakage due to mechanical abrasion is the largest contributor to resin loss on a RIP plant, especially when combined with osmotic shock.

Particle Size Distribution

Resins used in RIP applications need to be of “reasonable” size. Larger resin beads allow faster separation from pulp over inter-stage screens, but larger beads has slower diffusion-kinetics of uranium, from the PLS into the resin during extraction, and out of the resin during elution. Slow reaction kinetics requires larger plants or higher resin inventories, both resulting in higher capital cost.

Large resin beads are more sensitive to osmotic shock, which is a result of swelling and shrinking of the resin when subjected to alternating acidic and alkaline conditions. Strong base resins typically swell 5-10% from the sulphate to hydroxide forms.

“Reasonable” is thus a compromise between the desire for fast kinetics of the loading/elution reactions (small beads), good mechanical strength (small beads) and the desire for quick separation from the pulp using large-aperture screens (large beads).

Different applications require different types of ion exchange contactor designs. Resins are supplied in different size-gradings, the choice of which depends on the contactor design. The differences are illustrated in Figure 2. For a “standard” grading, the size of the beads varies from 325 to 1180 µm, with the largest percentage being between 425 and 1000 µm. Modern fixed bed applications requires resins with a very narrow size distribution, e.g. 600-700 µm. RIP resins also have a narrow size distribution, but shifted to the right of the fixed-bed grade, with the majority or the resin beads being between 850 and 1000 µm.

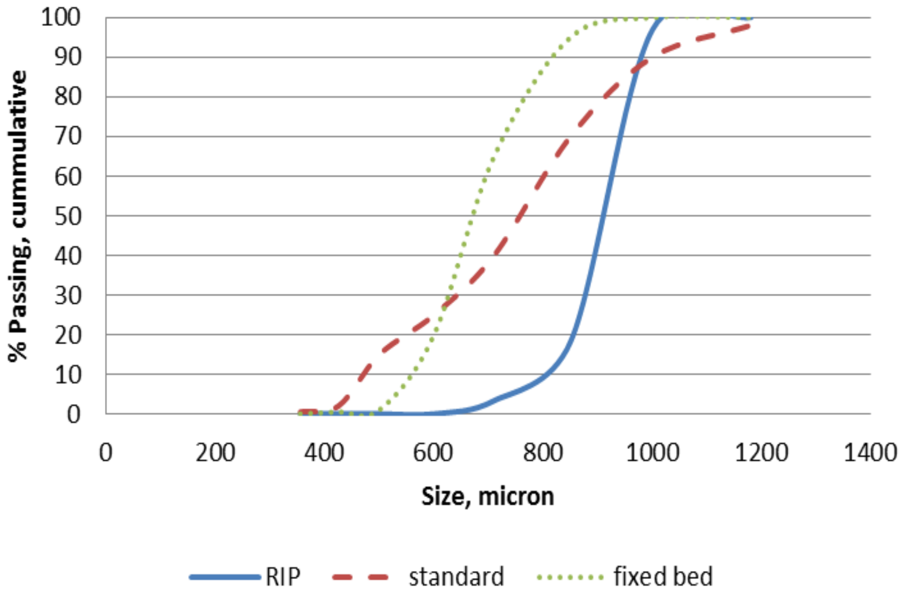

Figure 2: Particle size distribution of different resin gradings

Uranium ores are typically milled to 100% < 200 µm, with inter-stage screen-apertures set at between 400 and 600 µm. RIP grade resins typically have an average diameter of 850 to 1000 µm, with >90% of the resin having a size of 800-1300 µm. There is thus a diameter difference between the average resin bead and pulp particle of up to 600 µm.

Resin Loading Capacity

The maximum uranium loading onto a resin that can be achieved is dependent on a number of factors, including the uranium concentration of the pregnant leach solution (PLS), possible suppression of uranium loading due to the presence of competing anions (e.g. vanadium, chlorides) or fouling of the resin, and contact time. Generating an equilibrium loading isotherm, using a synthetic solution, is a quick and simple way of comparing the performance of different resins. Mintek uses a synthetic feed solution with a composition of 0.5 g/L U_3O_8, 1 g/L Fe^{3+} and 20 g/L SO_4^{2-}, at pH 1.8. This solution is representative of a PLS expected from a typical acid leach operation. Resin and solution are contacted in batch at different ratios for a period of 24 hours at ambient temperature. After 24 hours, the resin and solution are separated and analysed.

Freundlich isotherms have been found to generally give a good fit to the data, as shown in Figure 3. The data clearly shows that higher uranium loadings can be achieved with Resin B, making this resin more attractive, if judged purely on capacity.

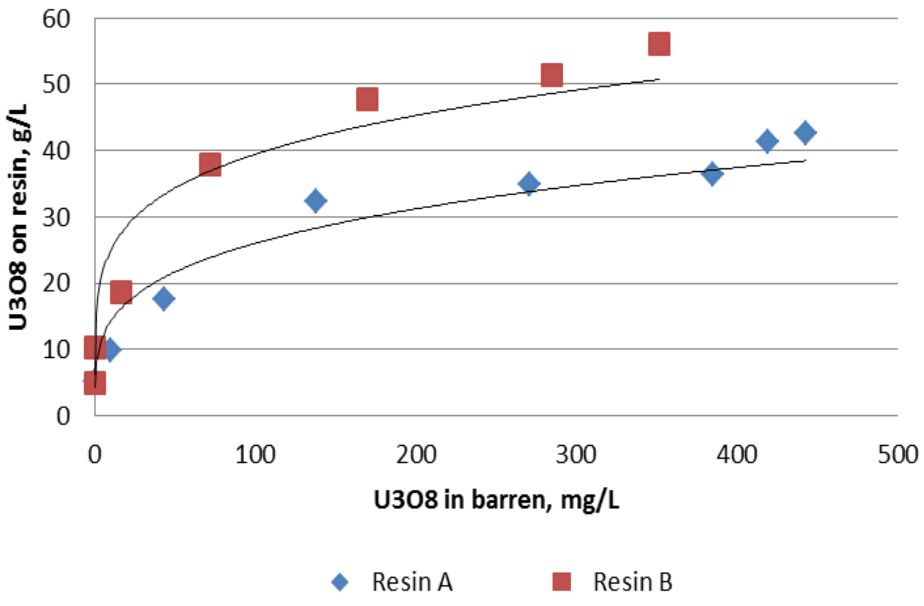

Figure 3: Equilibrium loading isotherm

Elution Characteristics

A suitable resin will be easy to elute, using readily available and inexpensive reagents, within a reasonable time. Acid leach uranium operations typically use sulphuric acid as eluant. Stripping (elution) isotherms for two different resins are shown in Figure 4, to illustrate the difference in behaviour of two different resins.

These isotherms were generated by contacting pre-loaded resin and 110 g/L H_2SO_4 in batch at different resin-to-solution ratios for a period of 10 hours. After completion of the test, the resin and acid were separated and the uranium concentrations determined. The final acid concentrations were ≥80 g/L, to ensure that stripping was not hampered by a lack of acid.

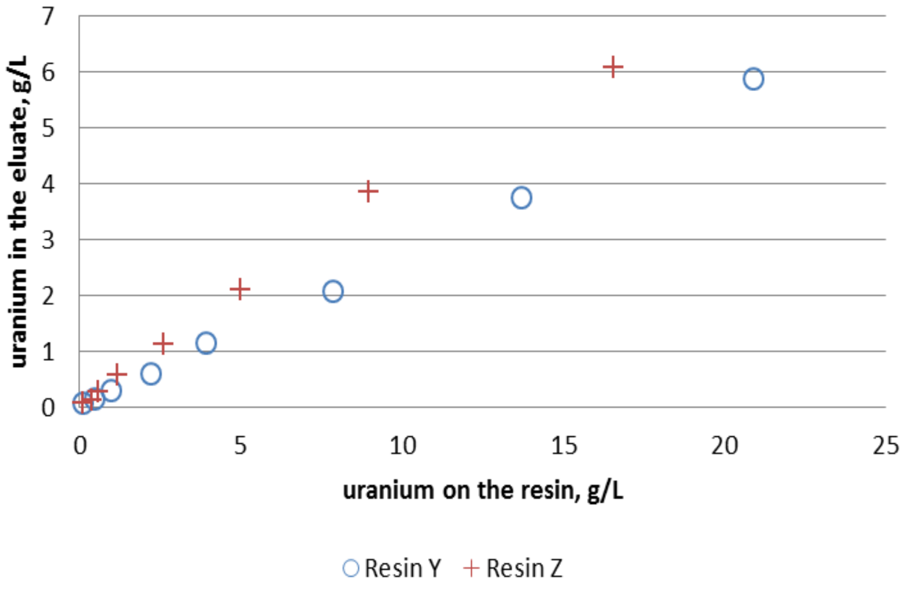

Figure 4: Stripping isotherms

The stripping isotherms for both resins were fairly flat, indicating that stripping is relatively unfavourable, but low residual loadings were achieved on both resins, meaning that it should be possible to achieve low barren concentrations when the resin is recycled to the adsorption circuit.

Higher eluate concentrations were achieved with Resin Z. This is desirable, as it will reduce the size of the downstream purification/precipitation equipment, thereby reducing both the capital outlay and reagent cost.

DURABILITY

The choice of resin for a RIP operation is not based on one single characteristic. It is rather a combination of high uranium loading capacity, low co-loading of impurities, ease of stripping using readily-available reagents, large particle size and, last, but certainly not least, mechanical strength.

It is not practical to evaluate the strength of all resins with promising chemical characteristics (loading capacity and elution) on a large-scale continuous plant, due to cost and time limitations. To overcome this problem, various laboratory-scale tests have been developed to evaluate the relative strength of different resins and provide an indication of the resin loss that can be expected on a full-scale plant.

Laboratory Scale Durability Tests

Ball Mill Tests

The Russian Ball Mill (RBM) test is used by Purolite to confirm the physical strength of the resin. A picture of the set-up is shown in Figure 5. The RBM parameter correlates to a specific resin loss expected on operating plants. The ratio is based on multiple data points that have been collected from a large number of (mainly gold) operations in the Former Soviet Union to which Purolite have been supplying resin since the 1980’s.

Figure 5: Russian Ball Mill (used by Purolite)

Mintek also performs a Ball Mill test(5), using a planetary ball mill with a somewhat different design, as shown in Figure 6.

Figure 6: Planetary ball mill (used by Mintek)

Both mills work on the same principle, regardless of the details of the design. A known volume of resin (pre-screened to >600 µm) is placed in a steel cylinder, together with a grinding medium (a set number of steel or ceramic balls with known diameter) and a known volume of water. The cylinder is rotated at a set speed for a set time, after which the resin is separated from the grinding medium and the remaining volume of resin beads with diameter >600 µm measured. The numbers are used to express the strength of the resin:

$S = V_1/V_2 \times 100$

Where:
S = mechanical strength
V_1 = volume of resin with; size >600 µm after milling;
V_2 = initial volume of resin with size >600 µm.

The ball mill test parameter cannot necessarily be used to predict the resin loss for all plants, due to the variations in plant design and ore characteristics that is found in different operations around the world. The conditions of the ball mill test are also much harsher than that experienced on an actual plant operation. Resin breakage occurs mainly during pumping and screening of resins and to a lesser degree due to agitation and collision.

The ball mill test does however remain a useful tool that is quick and inexpensive. It can be used to compare resins and eliminate very poor performing resins at an early stage, before spending time, effort and money on piloting of weak resins.

Flotation Cell Tests

A Denver Flotation Cell, as shown in Figure 7, is another method used to test resin durability(9). During this test, the resin is subjected to both osmotic shock (caused by swelling and shrinking of the resin during pH swings between acidic and alkaline conditions) and mechanical abrasion.

Figure 7: Denver flotation cell

The resin, pre-screened to >600 µm, is stirred in the Denver flotation cell for 60 minutes at 1500 rpm. Sand may be added, to simulate the abrasive environment provided by pulp. Upon completion, the resin is separated from the water and/or sand and transferred to a column where it is subjected to alkaline and acid treatments. Alkaline treatment consists of 3 bedvolumes (a bedvolume is the volume of solution equal to the volume of resin in the test) of a 2 mol/L NaOH solution, followed by a waterwash to remove excess caustic. The resin is then contacted with 3 bedvolumes of 2 mol/L H_2SO_4, followed by a waterwash to remove excess acid. One cycle consists of three distinct steps, i.e. (1) stirring in the flotation cell, (2) alkaline treatment, and (3) acid treatment. The resin volume is measured after each cycle and the relative loss, as a percentage of the volume at the beginning of the cycle, is calculated. Several cycles (at least 5) are required for a proper comparison. This test provides a good indication of the relative strength of different resins, but is rather cumbersome and time-consuming.

The cumulative loss per cycle for four different resins(2) is compared graphically in Figure 8. The results show that the highest loss over 5 cycles was obtained with GT2, while the lowest loss was seen for GT1.

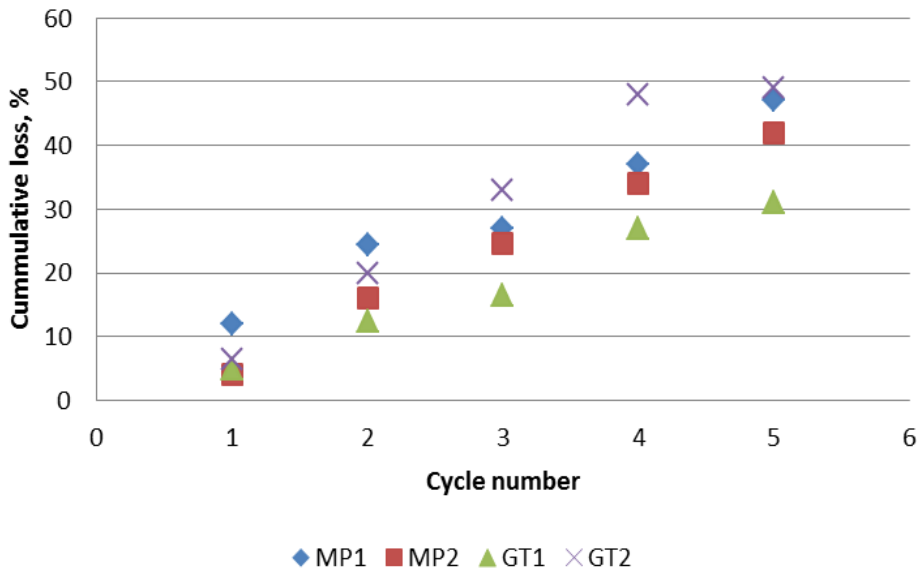

Figure 8: Cumulative loss of different resins, Denver flotation cell

Compression Tests

Compression testing(5) is used to determine the resin behaviour under load and the maximum stress the resin can sustain for the duration of the load (constant or progressive). It gives an indication of the elasticity and plasticity characteristics of the resins as well as the point of transition between these two states.

- The elasticity region is the range of applied load in which the resin bead returns to its initial form upon removal of the load. The slope of the curve in this region is termed the Young's Modulus.
- In the plasticity region, permanent deformation (cracking) has occurred and the resin bead does not completely return to its initial form upon removal of the load.
- Fracture is the point of irreversible deformation. At this point, compressive forces are greater than internal inter-molecular forces and are sufficient to cause cracking of the bead. The applied load at the point of fracture is termed the Break Load.

A picture of the LF Plus Materials Testing Machine is shown in Figure 9.

Figure 9: Compression test machine

Resin beads (100 beads per test) with a diameter of 850 µm are tested. Individual resin beads are placed on the compression plate and encircled with water. The compression plate is then lowered slowly until it contacts the resin bead, after which compression of the bead is done at a constant speed of 0.5 mm/minute and force of 0.5 N. The compression is automatically stopped once a fracture within the resin bead is detected. A typical stress-strain curve for the force applied on a material is shown in Figure 10. The applied load is plotted against the percentage strain that a resin undergoes. The strain is an indication of the deformation relative to the initial diameter of the resin (in this case 850 µm).

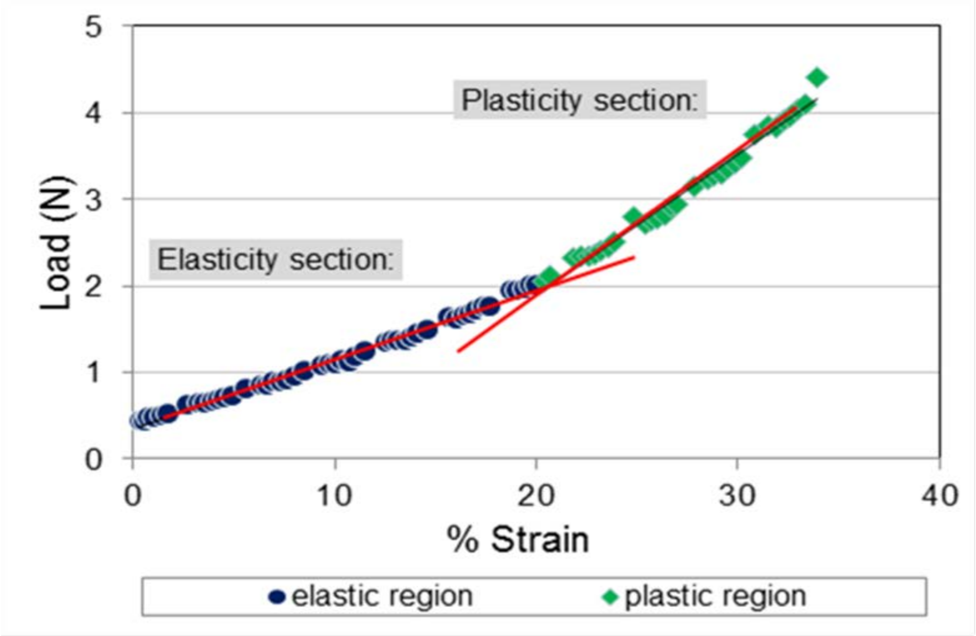

Figure 10: Diagram of a stress-strain curve, showing the relationship between stress (force applied) and strain (deformation) of a ductile material

The variation in characteristics of three different resins is illustrated by the data in Table 1 and Figure 11.

Table 1: Compression test data for 3 different resins

	Resin D	Resin E	Resin F
Young's Modulus (slope of first linear portion)	0.052	0.082	0.132
Break load, N	1.4	4.4	5.3
Break load, % Strain	18	33	28

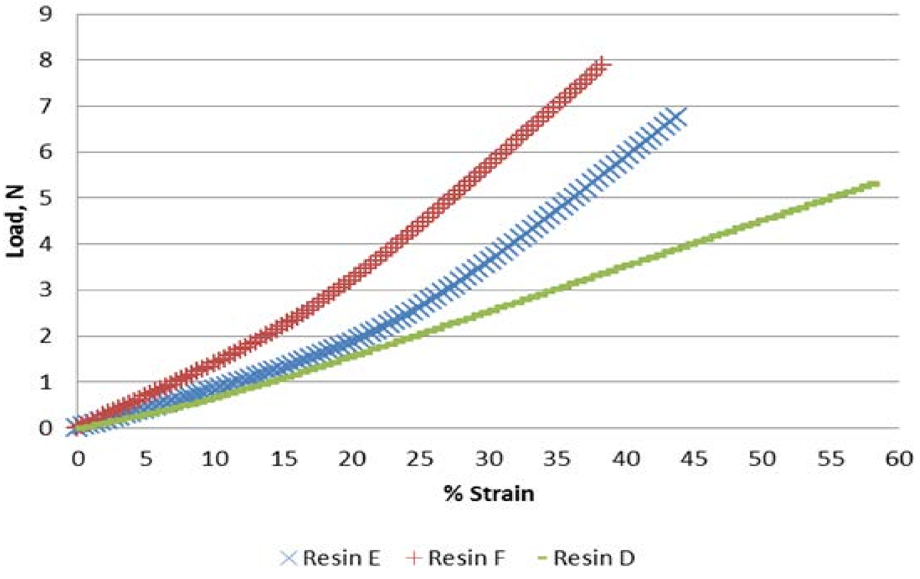

Figure 11: Compression test: Load vs % strain

Based on these results, Resin F can be classified as the strongest of the three resins. It had the highest Young's Modulus, meaning that it could withstand the highest load before permanent deformation, i.e. it was the most elastic. Resin F was also able to withstand the highest load before breaking. Resin D was the weakest of the three resins.

Too much elasticity is also not desirable, since a highly elastic resin may squeeze through the apertures in a screen and cause blockages. Exactly how much elasticity is required is still under investigation.

This type of test provides information on a number of aspects of the physical strength of a resin. Due to the automated nature of the test, it is quite independent of operator-skills. However, a large number of resin beads has to be tested to obtain a reasonable average and interpretation of the results requires skills and experience.

Mechanical Agitation

The results of investigations into the optimum conditions of mechanical agitation have been published by Mixtec(2). The aim of mixing the pulp-resin slurry is to ensure a uniform distribution of the resin and the solids in the pulp, allowing maximum uranium extraction and minimum resin breakage. In RIP, there are two different systems that must be catered for. The fast-settling slurries require a high degree of agitation at the bottom of the tank to provide uniform suspension. Buoyant resin needs good surface movement to draw it into the pulp. Typical mixing practice is to employ low-shear high-flow hydrofoil impellers. Mixtec's special impeller geometry consisted of a large ratio down pumping impeller to suspend the solids and a smaller up-pumping impeller to draw in the resin. This configuration was tested in tanks with diameters varying from 300 mm to 1200 mm.

It was found that resin loss increased with a decrease in tank diameter, from ~4% in the largest diameter tank to 70% in the smallest tank. This was ascribed to the fact that the impeller and other factors were scaled down in the smaller tank, but the resin particles remained the same, resulting in a higher collision rate and impact, causing higher resin losses.

This type of test is useful to provide information on the effect of agitation and agitator design on resin breakage. Since agitation is only one of the aspects that may cause resin breakage, this test can be used to compare the relative strength of different resins, but cannot be used in isolation to predict resin losses.

Effect of Silica

Silica fouling of strong base anion exchange resins during the recovery of uranium from acid-leach liquors is a well-known problem(8,10). Recent investigations have shown that silica fouling mainly occurs on the outer rim of the resin beads, thereby inhibiting the free diffusion of uranium in and out of the resin during the adsorption and elution steps. If left untreated, the level of silica increases throughout the resin bead, severely affecting the metallurgical performance of the resin. Removal of silica via periodic treatment with caustic soda (NaOH) is required to restore the resin's operating capacity for the adsorption of valuable metal. The resin on an acid-leach uranium plant is subjected to significant osmotic shock, as it is subjected to strongly acidic conditions during elution (110 g/L H_2SO_4) as well as strongly alkaline conditions during caustic treatment to remove silica. The resulting swelling and shrinking weakens the resin, making it more prone to degradation. Interestingly enough, some improvement in resin strength was observed for silica fouled resins during laboratory tests. It has however also been found that higher silica levels makes the resin more brittle.

It is not expected that the presence of silica will have a significant effect on resin degradation, provided silica fouling is handled appropriately.

Large-Scale Durability Tests and Pilot Plant

Mintek and Bateman Engineering ran a demonstration plant for Harmony Gold at one of their gold mining operations in South Africa in 2010(1,4). The plant comprised of an acid-leach circuit, the Mintek/Bateman developed MetRIX continuous RIP plant, as well as an elution and tailings treatment circuit. The demonstration plant was operated for 47 days.

In addition to evaluating various mechanical aspects of the plant, the degradation and volume loss of resin were closely monitored. Fresh resin, as well as resin that had been abraded during the RIP piloting, are shown in Figure 12 (fresh) and Figure 13 (abraded). The pictures shows that the resin used during the demonstration plant fractured into irregular pieces. Resin loss is ascribed to the loss of any pieces that are smaller than the aperture of the screens used to separate resin and pulp.

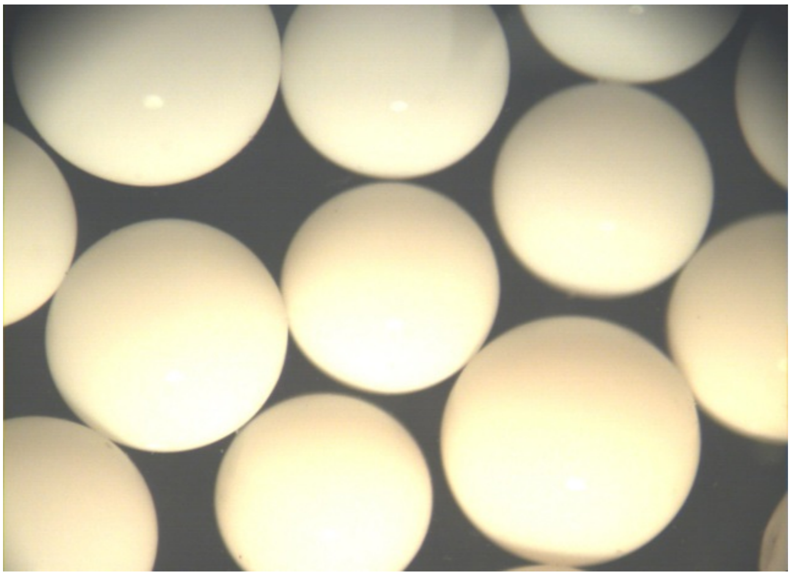

Figure 12: Resins viewed under a microscope, fresh

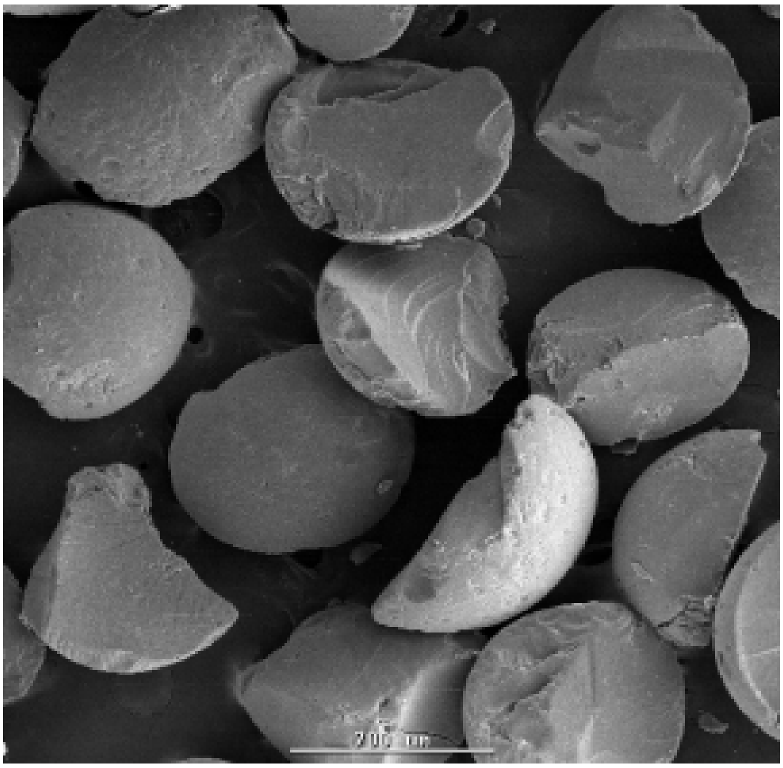

Figure 13: Resins viewed under a microscope, showing the effect of abrasion

FINANCIAL EFFECT OF RESIN LOSS

Techno-economic evaluations have been done on various flowsheet configurations. A recent study by Bateman(6) found that the optimum flowsheet can be largely based on the uranium grade of the ore. At uranium concentrations of less than 900 mg/L U_3O_8, a processing route comprising RIP for bulk uranium recovery, followed by solvent extraction (SX) for purification, was found to be the most economical (RIP-SX). At higher uranium concentrations (>900 mg/L U_3O_8), the most economical route would consist of counter-current decantation (CCD) followed by SX for uranium upgrading and purification (CCD-SX).

The Bateman study also investigated the effect of various parameters, such as resin loss, on the Operating Cost (Opex) of a project. The resin loss assumption was based on results of demonstration-scale durability trials. The study found that the Opex was relatively insensitive to variations in resin loss. The annual resin loss was found to constitute <4.4% of the total Opex of a RIP-SX processing route.

The relative cost of resin replacement in a RIP-SX plant was compared with the cost of replacement of flocculent and organic (the two major reagents) in a CCD-SX plant (Figure 14). The annual resin replacement will vary between projects, since it depends on various factors, including the nature of the ore (soft or harsh), throughput, and the resin used (resin price, mechanical strength, capacity). Based on current available information, it is expected that the annual resin replacement rate would be less than 100% of total inventory. The graph shows that the cost of resin replacement (RIP-SX plant) will equal that of the cost for flocculent+solvent replacement (in a CCD-SX plant) at an annual resin replacement rate of just under three times the inventory. It must be noted that the cost of solvent replacement in the relatively small SX plant of the RIP-SX plant is not included here.

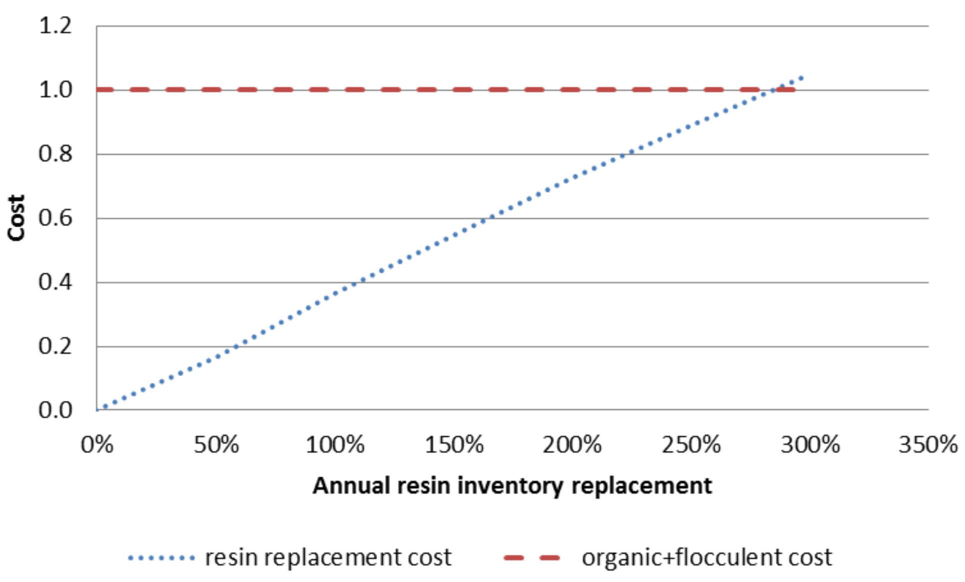

Figure 14: Cost of resin replacement vs organic+flocculent replacement

CONCLUSIONS

There is a need for a laboratory-scale test or tests that can be used to compare different resins for their mechanical durability. These tests must have the minimum requirement for manpower, material and time, i.e. they should be quick and inexpensive.

The main laboratory-scale tests that are currently used by various laboratories include:

- Flotation cell: tests effect of osmotic shock in conjunction with abrasion due to agitation;
- Ball mill: tests effect of mechanical impact;
- Compression test: evaluates breaking weight and elasticity.

These tests have been found to be useful for the comparison of the mechanical strength of various resins, as well as the elimination of non-performers at an early stage. However, the conditions of these tests are quite harsh and resin losses determined by the laboratory-scale tests are not necessarily representative of resin losses that will be experienced on full-scale plants.

True correlations between resin loss experienced on full-scale operations and laboratory tests will only be possible as more information from actual plants becomes available.

Recent economic studies have shown that the operating cost of a RIP uranium plant is relatively insensitive to resin loss. It has been shown that the resin replacement cost of a RIP circuit is less than the reagent (flocculent and solvent) consumption cost on a plant using solvent extraction for uranium recovery at annual resin replacement values up to three times the total resin inventory.

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