

The Relix process for the resin-in-pulp recovery of uranium*

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SYNOPSIS

The Relix process is based on direct contact between an ion-exchange resin and undiluted pulp, thus avoiding prior solid-liquid separation. The resin particles float near the surface of the pulp, forming an inverted fluidized bed with the pulp flowing downwards.

The basic idea was demonstrated on a full-scale pachuca tank at Stilfontein Gold Mine in 1970, followed by a small-scale demonstration run in a laboratory at the National Institute for Metallurgy. A pilot plant based on a throughput of 60 tons of ore per day was subsequently operated at West Driefontein Gold Mine for several periods over two years.

Although the plant proved operable from a mechanical point of view, the metallurgical performance was not up to expectation. The basic cause of the poor metallurgical performance was shown to be backmixing of both the resin and the pulp between stages. The values obtained for resin losses were inconclusive.

Further development of resin-in-pulp processes for the recovery of uranium should be focused on the performance of various techniques for the screening of resin from pulp.

SAMEVATTING

Die Relix-proses is gebaseer op direkte kontak tussen 'n ioonruilhars en onverdunde pulp waardeur voorafgaande skeiding van die vaste stof en vloeistof uitgeskakel word. Die harspartikels dryf naby die oppervlak van die pulp en vorm 'n omgekeerde fluïedbed met die pulp wat afwaarts vloei.

Die basiese idee is in 1970 met 'n volskaalse pachuca-tenk by die Stilfontein-goudmyn gedemonstreer en daarna deur 'n kleinskaalse demonstrasieloop in 'n laboratorium by die Nasionale Instituut vir Metallurgie. Later is 'n proefaanleg wat op 'n deurvoer van 60 ton erts per dag gebaseer is, vir etlike periodes oor 'n tydperk van twee jaar by die West Driefontein-goudmyn bedryf.

Hoewel die aanleg uit 'n meganiese oogpunt bedryf kon word, het die metallurgiese werkverrigting nie aan die verwagting voldoen nie. Daar is bewys dat die grondoorzaak van die swak metallurgiese werkverrigting die terugmenging van sowel die hars as die pulp tussen die stadiums was. Die waardes wat uit die harsverliese verkry is, was nie afdoende nie.

Verdere ontwikkeling van die hars-in-pulpprosesse vir die herwinning van uraan behoort toegespits te word op die werkverrigting van verskillende tegnieke vir die sifting van die hars uit die pulp.

Introduction

The most expensive process in modern uranium plants is the solid-liquid separation step after leaching. This step is claimed^{1, 2} to account for fully 50 per cent of the total capital cost of the plant, including that tied up in the steps from leaching to recovery of the product. It is therefore natural that attempts at reducing the cost of uranium plants should be focused on this step.

The original objective of the Relix process was to eliminate the solid-liquid separation step entirely and to combine extraction onto resin with part of the leaching process³.

The principle on which the Relix process⁴ operates is the contacting of ore pulp having a solids content of 50 to 60 per cent with beads of ion-exchange resin of a density that enables them to float in the pulp, as suggested by Read⁵ and Carman⁶. A central airlift circulates the pulp so that a fluidized bed of resin is formed below the surface by the downward-flowing pulp. The Relix process differs from those of Carman or Read: downward fluidization of the resin is caused deliberately, and the resin is transferred between stages by airlifts instead of by scoops or suction tanks.

The Resin-in-pulp Concept

Awareness of the possibility of resin-in-pulp (RIP) processes appears to have originated when the first ion-exchange processes were developed for the recovery of uranium. In the U.S.A. there was a technical incentive to adopt RIP techniques because of the non-filterability of some ores, whereas in other countries, particularly South Africa, metallurgists preferred to install plants based on complete solid-liquid separation before the ion-exchange or liquid-extraction step.

The earliest publication on RIP techniques⁷ covers most of the ideas that are still relevant. The basis of this approach is that the resin is controlled mechanically by restraining the beads with a screen. Ore pulp flows through a series of vessels in which baskets of resin are vibrated slowly to keep the screens clear. The resin is not moved, and the vessels are operated cyclically in a manner similar to that used with normal fixed beds. This approach is simply a mechanical modification of fixed beds so that solids in the pulp can flow through the resin. The observation is made in the same paper that the sand fraction of the pulp tends to blind the screens, and the use of a desanded pulp is suggested. Descriptions of the plant and operations of a commercial uranium mill based on pilot-plant data⁷ are given in later papers^{8, 9}. The product of a hydrocyclone sand-washing circuit contained 8 per cent solids that were nearly all less than 300 mesh, and this constituted the feed to the RIP section.

In a subsequent improvement¹⁰, the RIP operation

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was made continuous in the process sense, so that resin flowed continuously from an absorption vessel to an eluting process. The principle of retaining the resin beads between screens was still used, but, instead of the screens being moved, the upward flow of pulp had a slow air-induced pulse superimposed, which helped to clear the fixed screens.

A team at the Winchester Laboratories of the National Lead Company, under contract to the U.S. Atomic Energy Commission, was responsible for a parallel development in which continuous operations in both resin and pulp streams was used¹¹. A pilot plant was built, based on the use of air-agitated tanks to mix resin and pulp, followed by a vibrating screen to separate the two streams overflowing from the tank, in the same manner as a settler functions in a liquid-extraction contactor. A series of stages was connected so that resin and pulp flowed between the stages in countercurrent fashion. The report on this pilot plant¹¹ is notable for the systematic way in which the resin loss was measured. The team reported resin losses of 5 to 35 per cent per year, which were due only to the air agitation of resin-pulp mixtures and depended on the grade of the resin used. Tests on the same resin showed that the screening operation caused large increases in the attrition rate, ranging from 20 to 100 per cent per year for screening in water to between 30 and 300 per cent per year for screening in pulp. In test runs on the plant, the resin loss was reported to be 300 ml per ton of ore when Permutit SK resin was used.

A full-scale application of this pilot-plant work is described in a company pamphlet¹². This plant had been working since the latter part of 1966, when the old reciprocating-basket plant installed in 1957 was replaced with the vibrating-screen system. The vibrating-screen plant, like the reciprocating basket, depended on the desanding of the pulp by classifiers and cyclones before it was fed to the RIP circuit. Washing of the sand fraction required 2.5 parts of water for 1 part of leach pulp, which diluted the RIP feed to 7 per cent solids, all of which were below 325 mesh. This desanding process was clearly necessary because the ore was ground to only 5 per cent greater than 28 mesh. The larger particles in the ore were of the same size as much of the resin, and would have blinded the screens. No mention of the rate of loss of the resin or the make of resin used is made in the pamphlet. It is possible that the plant described¹² used a more modern resin than was used in the work reported earlier¹¹, since that work (in 1958) was regarded as 'perfectly satisfactory except for resin degradation'.

Work based on a different approach was carried out in South Africa at about the same time as the Australian work¹⁰ referred to above. In all previous operations, it had been found necessary to desand the pulp with cyclones or thickeners prior to feeding it to the resin, which was retained between screens. However, Read⁵ and Carman⁶ reported the results obtained in an extensive pilot-plant test of a contactor in which concentrated pulp was used without a desanding operation. The novelty in their approach was the elimination of screens, the resin being allowed to float on the surface of the pulp. The work was generally satisfactory, but it was

not scaled-up because of uncertainty in the industry (1960) and of mechanical difficulties in controlling the flow of resin through the plant. Wood fibres caused considerable trouble with the resin-transfer scoops. On the subject of resin loss, Read⁵ reports that the loss due to abrasion would not exceed that experienced at present as a result of poisoning in fixed beds, and concludes that, even in a continuous RIP process, chemical poisoning determines the life of the resin.

A further application of the principle used by Carman and Read is reported by Davison *et al*¹³. They made a pilot-plant study of the RIP recovery of gold from a raw leach pulp. The results appeared promising and showed that the RIP recovery of gold could be cheaper than the conventional process. It is not clear why the promising results were not followed by work on a larger scale. This is in contrast to the gold industry in the U.S.S.R., which was extending the RIP recovery of gold¹⁴ during the five-year plan of 1970 to 1975.

It is known that plants are now in operation in South Africa in which gold is recovered from pulp by extraction onto activated carbon.

Experimental Work

After some preliminary experiments on the Relix process in the laboratory, a small eight-stage pilot plant was run continuously at the National Institute for Metallurgy (NIM) for two weeks under cold-leaching conditions. This plant processed only 10 litres of pulp per hour, but ran satisfactorily and recovered about 98 per cent of the soluble uranium in the pulp.

From the hydraulic aspect, the feasibility of the concept depends on the ability of the resin to float in a band in the upper part of the pachuca. This becomes an inverted fluidized-bed in which the fluidizing medium is pulp that is flowing slowly downwards. The hydraulic concept was verified in a batch test on alkaline pulp and normal-size scrap resin in a single pachuca of 4.6 m diameter at Stilfontein Gold Mine in 1970. There was no flow of pulp through the pachuca.

After discussions with various mining groups, it was decided that a pilot plant should be constructed at an operating uranium plant so that reagents could be obtained and the recovered uranium disposed of without costs being incurred. West Driefontein Gold Mine was selected as the most suitable site because the old uranium plant and much of its equipment were available at no cost to the project. The plant was based on some scrap pachuca tanks 1.2 m in diameter. The Atomic Energy Board and NIM took over the design, supply, and installation of pipes, pumps, instruments, and all other items of equipment according to a flowsheet that is shown in its final form in Fig. 1.

The early experiments were concerned mainly with the testing of the various screens and with checking that pulp would flow through the plant. A flow-rate of about 60 tons of dry ore per day was set as the throughput of the plant, and the resin-handling equipment was designed for a corresponding flow of about 1 litre of resin per minute.

A further innovation in the pilot plant was the continuous elution of resin in a Cloete-Streat contactor¹⁵

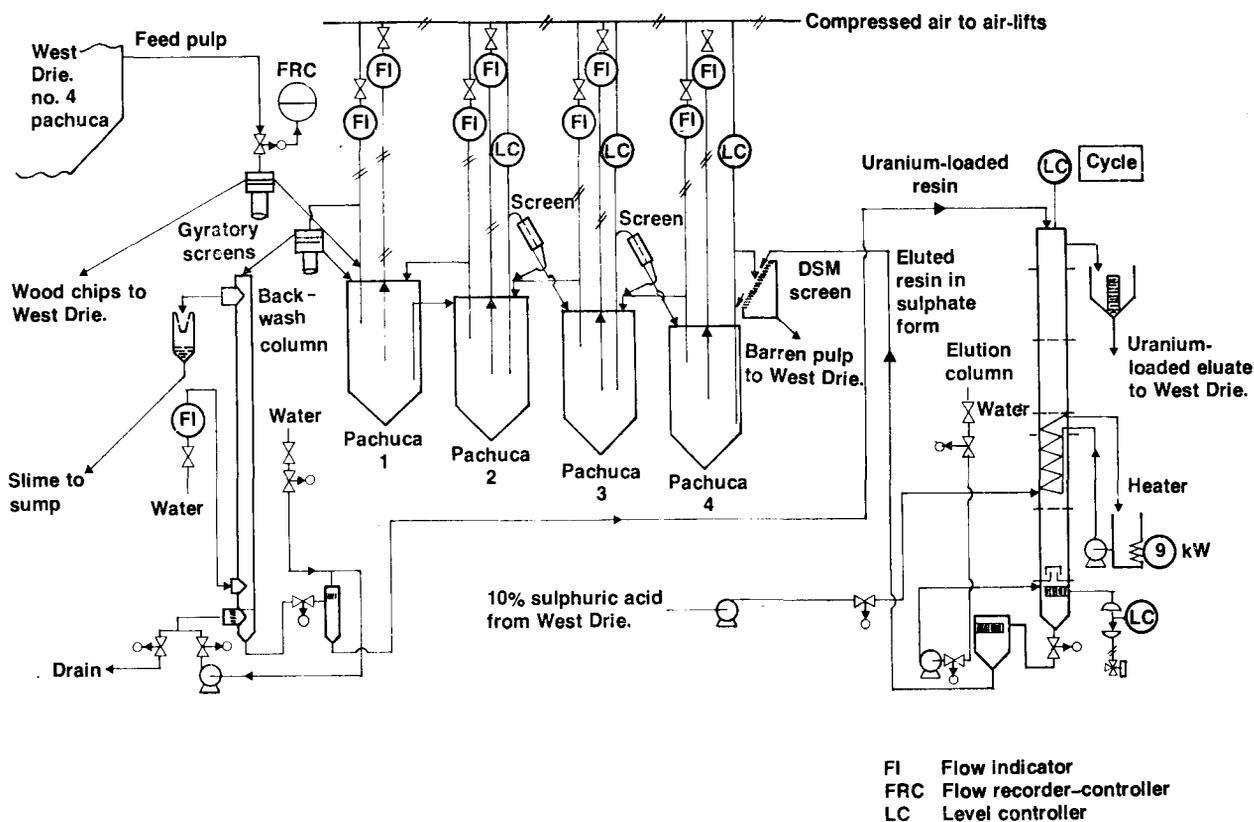


Fig. 1—Flowsheet of the Relix pilot plant, January 1973

with 10 per cent sulphuric acid. To build up confidence in the use of this technique, the two 25 cm-diameter elution columns installed were connected so that one extracted uranium from unclarified pregnant solution from the mine uranium plant while the resin thus loaded was eluted in the other column. Although this was an incidental part of the RIP project, it was the first actual pilot-plant demonstration of the proposed continuous ion-exchange process for the recovery of uranium^{15,16}, which was developed further as the NIMCIX process¹⁷. The successful runs during August and September 1971 proved that the elution process and the plant were satisfactory.

At the end of 1971, it was felt that the process elements tested on the pilot plant could be attempted as a complete process on a shift basis. The early part of December 1971 was spent on training the operating staff and on bringing the plant on line in stages, uranium-loaded eluate being produced for the first time on 17th December, 1971.

The first objective of the project had been achieved, in that it demonstrated that the plant and process could be operated under steady conditions. In its final form, the plant ran completely automatically, the operator's duties being confined to occasional adjustment of the set points of timers and to the taking of samples. Since pumps and other moving parts had not been duplicated on this pilot plant, the operators were also responsible for carrying out minor repair and maintenance jobs. The results obtained during the period September 1972 to February 1973 are shown in Fig. 2.

It was anticipated that a comprehensive technical and economic evaluation of the Relix process could be made at this stage but, unfortunately, very few comprehensive conclusions could be drawn from the pilot-plant data obtained at West Driefontein. Thus, the technical and economic feasibility of the Relix concept can be said neither to have been proved nor disproved on the basis of that work. However, sufficient information was obtained to enable the problems that required further investigation to be defined.

Results and Discussion

Equipment

The pulp-handling systems in the pilot plant were relatively simple, but some problems were caused by blockages resulting from settling in stagnant zones and overflows. The pulp was screened on gyratory screens, 45° DSM screens, and simple inclined screens. The gyratory screens were subject to periodic failure, and some initial corrosion problems were experienced with materials in the DSM screens.

In any continuous ion-exchange plant, one of the major problems is the transfer of resin from vessel to vessel without breakdown of the resin. Positive displacement pumps crush the beads, and devices such as centrifugal pumps or ejectors, where the shear rates are high, abrade the resin. Two convenient non-mechanical techniques are the airlift^{12, 13} and hydraulic displacement^{19, 20}, which were used for all the resin handling in this plant.

The flow of resin between the pachucas was achieved

with variable-submergence airlifts, which behaved satisfactorily.

The backwash column was found to be essential for the removal of traces of slimes collected with the loaded resin, thus preventing blockage problems in the elution column and contamination of the concentrated eluate. An oversize screen was essential to prevent rubbish from blocking the backwash and elution columns.

The elution column performed satisfactorily and could be scaled up as required, as reported elsewhere¹⁷.

The flow of pulp to the plant was controlled automatically, and initially there were simple overflows through the rest of the pachucas. Interstage screening of pulp necessitated the use of controlled airlift pumps to replace the overflows. Apart from trouble with the magnetic flowmeter itself, this system was fairly satisfactory. High level in any pachuca started an alarm and cut off the pulp feed, which proved to be useful in avoiding floods. Direct control of the interstage flow-rate of resin was very difficult, but a measure of control was

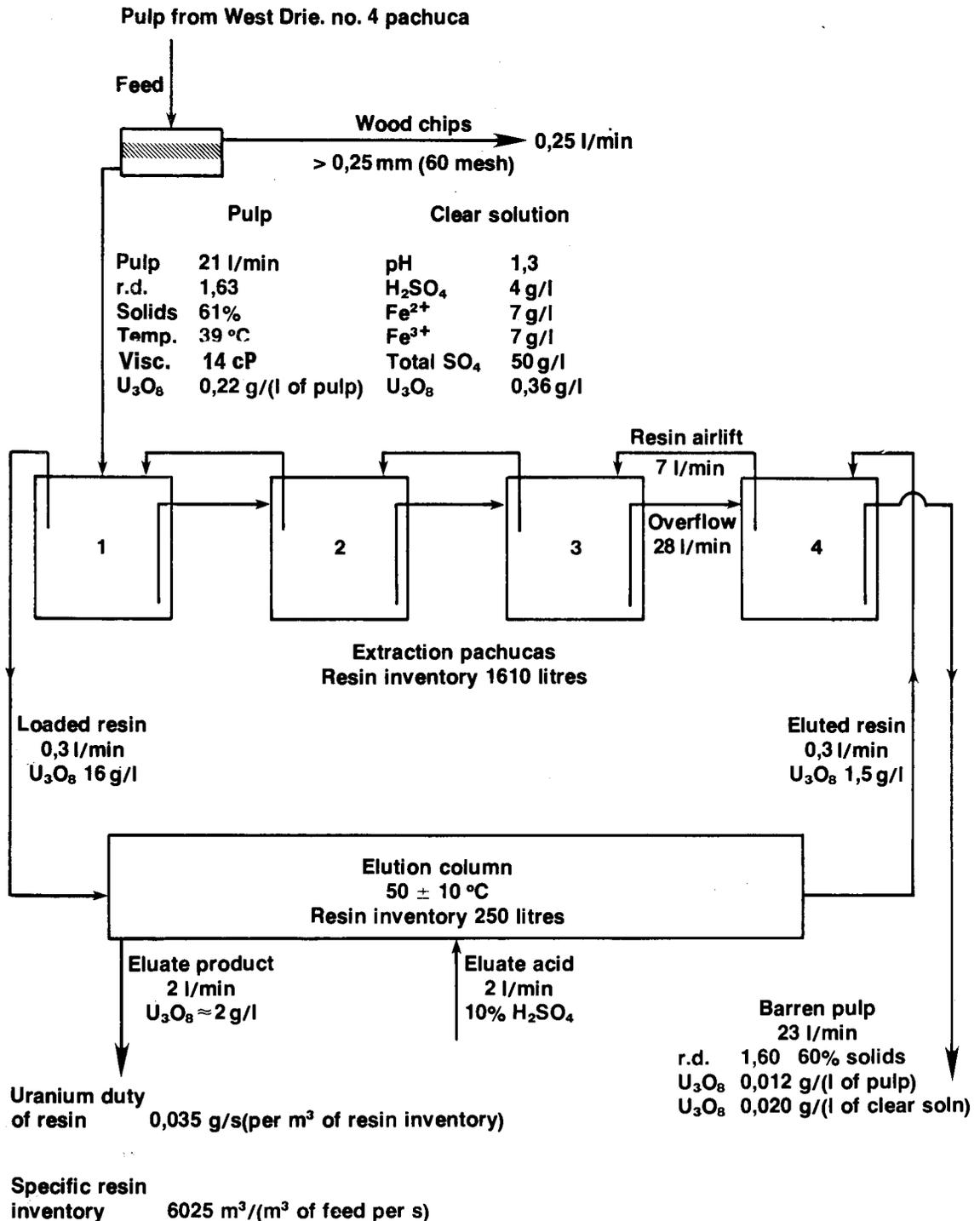


Fig. 2—Detailed flowsheet of the Relix process

achieved by alteration of the depth and flow-rate of the airlift discharging to the screens and by alteration of the depth of the resin airlifts in the other pachucas.

A very simple torque-sensitive switch at the top of the elution column caused the column to cycle only when full of resin²¹. The flow-rate of eluted resin could then be recorded by a count of the cycles since the volume of resin discharged per cycle was remarkably constant, as suggested by the theory of operation of this column²².

Loss of Resin

Although the loss of resin is generally thought to be a significant barrier to the adoption of resin-in-pulp techniques, operating data from resin-in-pulp plants overseas and the pilot-plant work of Read and Carman indicate that it can be kept under control.

At the outset of the project described here, various figures for resin loss in a number of overseas resin-in-pulp plants were compared, and a loss equivalent to 3 per cent of the resin inventory per month was set as an acceptable target for the Relix plant.

The first run on the West Driefontein pilot plant in January 1972 showed a catastrophic loss of 30 per cent of the resin inventory per month. It was concluded that this loss could have been caused by the poor condition of the scrap resin used or by the lack of a screen on the delivery of the apron-sump pump. The second run used Dowex 21K resin of resin-in-pulp size that had previously been used for only a few months. The apron and sump were lined with asphalt, and resin-scavenging screens were fitted to the sump-pump delivery and to the overflow for loaded eluate. This time the calculated loss was about 3 per cent of the resin inventory per month. These initial indications showed that the resin loss had been reduced to a reasonable level, but it was felt that this should be proved over a longer period of operation.

Measurements of resin loss during the last 18-week campaign on the pilot-plant are shown in Table I. In the evaluation of these measurements, it should be noted that both the Dowex 21K and IRA-425 resins were breaking up rapidly in the latter stages of the run, and that some resin was lost in a single accident.

One would therefore expect to be able to contain the resin losses in a full-scale plant provided that the follow-

ing conditions are met:

- (a) a tough macroreticular strong-base or weak-base resin is used,
- (b) all streams of barren pulp, loaded eluate, and sump drainings pass out of the plant through screens so that the resin can be recovered, and
- (c) the feed pulp is screened of all wood chips.

From Table I it can be seen that the type of resin used was changed a number of times. These changes, together with the very short periods of continuous operation, do not permit precise evaluation of the resin loss.

Extraction Process

The main interest in this project was in the extraction side of the process. Equilibrium determinations were made for all the resins used and are shown in Fig. 3. The following conclusions were drawn from the equilibrium graph.

- (a) The equilibrium curve was depressed when the leach was changed from that using manganese dioxide additions to that using ferric iron additions.
- (b) The capacity of a weak-base resin is not significantly different from that of a strong-base resin.

In addition, it was shown that fairly large variations in the silica loading of the resin do not affect its capacity for uranium.

A major defect in the early campaigns was the extensive backmixing of the pulp, which resulted from the resin air-lifts having a throughput of about 150 per cent of the pulp feed so that the four stages were equivalent to one badly stirred tank. In the later runs, the diameters of the resin air-lifts were reduced from 50 to 25 mm, which brought their flow down from 36 to 8 litres of mixture per minute. This reduced the backmixing of the pulp to about 33 per cent of the pulp feed.

Backmixing of the resin was also a problem in the July 1972 runs, which was indicated by a large change in the uranium concentration from the eluted resin to pachuca 4, compared with small changes in resin concentration between pachucas 1, 2, and 3. Screens were introduced in November 1972 to control the backmixing of resin in the pulp from stages 2 and 3, and this resulted in an improvement in metallurgical efficiency. However, it is possible that these screens resulted in increased losses of resin due to abrasion.

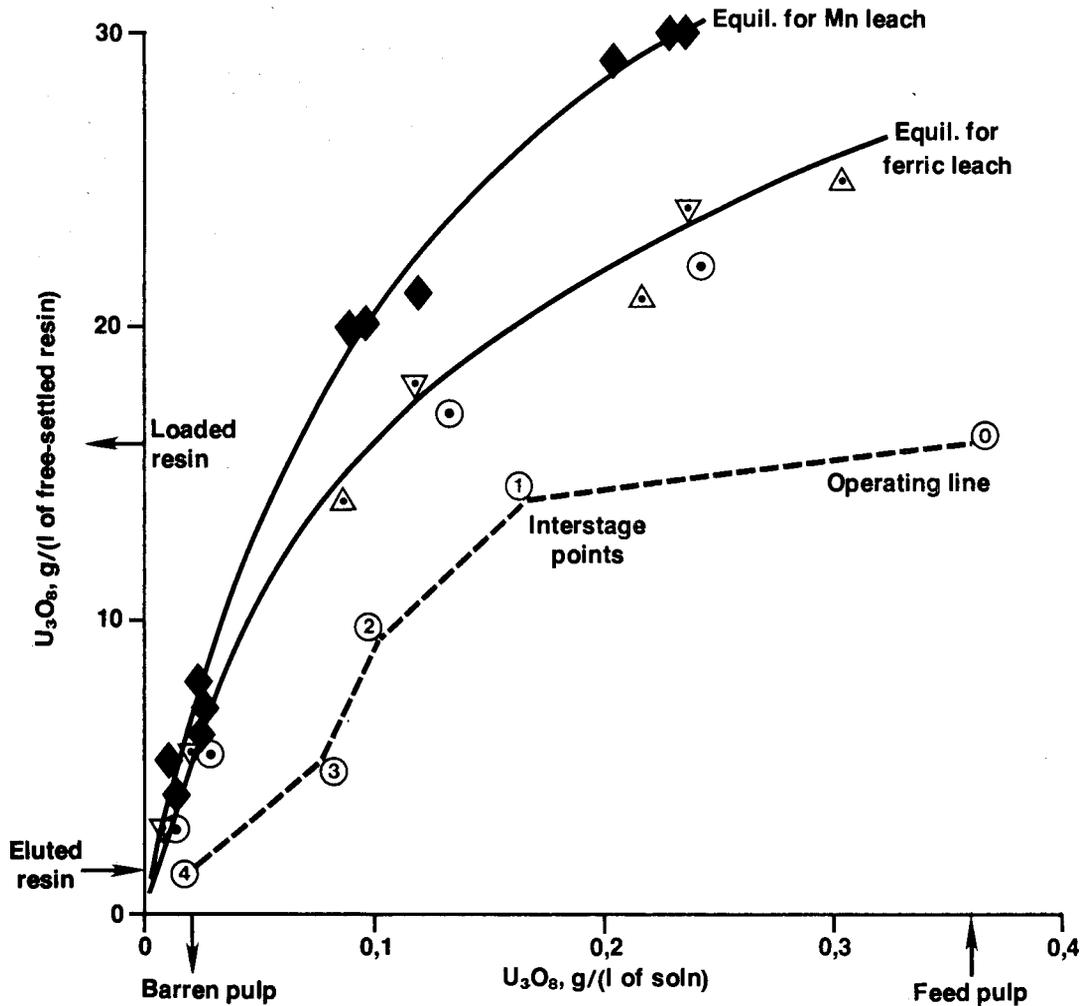
TABLE I
MEASUREMENT OF RESIN LOSSES

Date measured	4/8/72	1/11/72	23/11/72	6/12/72	19/2/73	Totals
Resin used	Dowex 21K	Dowex 21K	Dowex 21K	Bayer CA9247HL	Dowex 21K and old R & H IRA 425	Dowex and R & H (not Bayer)
Volume, litre	1246	1114	1024	1982	1859	1859 (1246)
Resin lost, litre	33	132	90	32	249	504
Number of days in use	23	44	19	8	63	149
Rate of resin loss per month, %	3	8	14	6	6	6 (8)

Fig. 3 shows a typical operating diagram during January and February 1973. An intensive sampling campaign showed that the uranium concentrations were constant, so that the plant appeared to be at steady-state.

The operating line for a plant at steady-state is not normally curved (as in Fig. 3), and it was therefore decided to investigate the steady-state mass balance in the system by simulation on a computer. Copelowitz

and Loveday²³ found that curved operating lines were obtained when feedback of resin or pulp occurred. So that the correct amount of resin (about 0,4 l/min) would be transferred between stages, the air-lift pumps also transferred 7 litres of pulp per minute, or about one-third of the pulp feed. Because the quantity of resin recovered by screens between pachucas 2, 3, and 4 was not measured, the feed-back of resin in the pulp from each stage could not be estimated. These feed-backs



Equil. point	Resin type	Silica loading g/(l of resin)	pH	Soln concn, g/l			
				Fe ²⁺	Fe ³⁺	Mn	SO ₄
◆	Dowex 21K	9	1,6	1	3	5	34
⊙	Dowex 21K	38	1,5	4	3	tr.	37
▽	New Bayer CA9247HL	0	1,5	4	3	tr.	37
△	Mixed Dowex 21K and IRA-425	26	1,9	7	7	tr.	55

Note: 1% SiO₂ by mass $\hat{=}$ 3 g of SiO₂ per litre
(air dried at 110 °C) (wet-settled)

Fig. 3—Typical operating diagram during January and February 1973

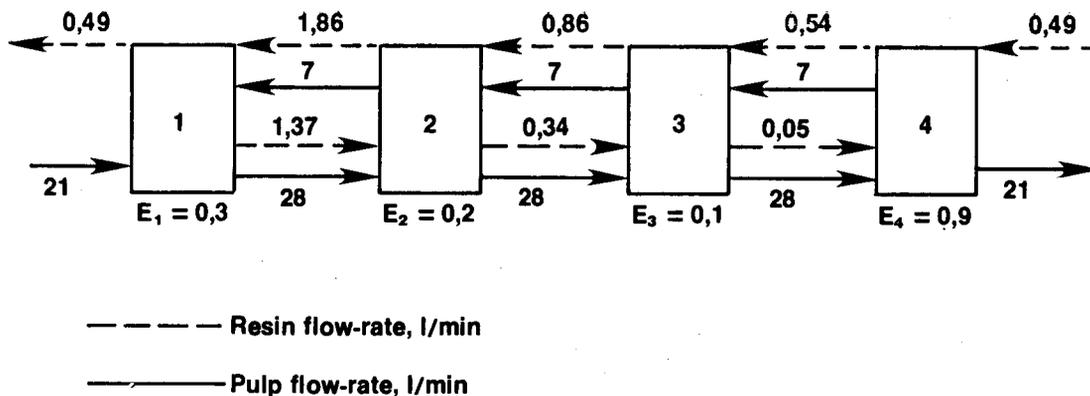


Fig. 4—Steady-state flows and efficiencies

were adjusted to simulate the operating line in Fig. 3 and, because it was not possible to obtain this line only from the adjustment of feed-backs, a Murphree stage efficiency was introduced for each stage. This stage efficiency is directly related to the inventory of resin in that stage via the kinetics of loading²³. The steady-state flows and efficiencies obtained by simulation to yield the operating diagram of Fig. 3 are as shown in Fig. 4.

The high feed-back of resin from stage 1 is consistent with the fact that pulp leaving stage 1 was not screened. The simulation shows a significant feed-back of resin from stage 2, despite the presence of a screen. This inferred flow of resin could well be due to the fact that the screen between stages 2 and 3 had a mesh aperture of 1 mm (i.e., slightly larger than the resin beads), whereas the screen between stages 3 and 4 had an aperture of 0,57 mm, which was smaller than the resin beads.

Additional resin was introduced in an attempt to improve the metallurgical efficiency for the last runs to give a total inventory of 1860 litres. The specific resin inventory during the period January to February 1973 was 5754 m³ of resin per cubic metre of feed per second. This is well above the 1000 m³ applicable to the continuous ion-exchange pilot plant that was under test at Hartebeesfontein Gold Mine.

An attempt was made to measure the resin inventory in each stage by the addition of resin containing radioactive gold (Au-198) and the determination of the proportion of radioactive resin after 2 hours of mixing. The inventories determined showed poor metallurgical efficiency, but, as the simulation study was undertaken only after all the experimental work had ceased, the underlying cause was not known during the experiments. Clearly, a higher overall efficiency could be obtained with more stages, but the efficiency of the four-stage system was far lower than the ideal value because of severe backmixing of pulp and resin and the consequent maldistribution of the resin.

Conclusions

The following conclusions can be drawn from the operating data gathered.

(1) The reliability of the plant was satisfactory as a

pilot plant. The mechanical breakdowns that occurred were of a minor nature.

- (2) The values obtained for resin loss were inconclusive.
- (3) The backmixing of both resin and pulp was considerable. As verified by a computer simulation of the process, this backmixing was responsible for the inadequate metallurgical performance of the plant.

Future Development

It is necessary to compare the proposed Relix process with the alternative of a small conventional 'screen type' resin-in-pulp process following leaching. In the Relix process, the residence time for leaching may be up to eight times the contact time required for the absorption of uranium on the resin. Thus, if the resin is distributed throughout the leaching pachucas, an excessive resin inventory may result, which could more than offset the savings obtained from the utilization of the leaching vessels as resin contactors. Resin transfer and separation devices are common to both systems.

The use of the original concept of an inverted fluidized bed did not result in efficient phase separation, perhaps because the agitation required to keep the pulp in suspension was too severe to permit phase separation by this principle. Interstage screening of the pulp was used to control backmixing of the resin, and resulted in a system virtually identical to that in a conventional resin-in-pulp plant.

Screening may increase the abrasion of resin, but, if the inverted fluidized-bed system could be made to work, screening could be avoided.

Consequently the following recommendations are made.

- (1) The effect of various types of screening techniques on the loss of resin should be investigated and compared with the loss of resin in an agitated pulp. These results will quantify the incentive to make the simple concept of an inverted fluidized-bed operate satisfactorily.
- (2) If there is an incentive for avoiding or reducing the use of screens, more-fundamental studies should be conducted on the principle of an inverted fluidized bed of resin-in-pulp.

Acknowledgements

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Coal trade and transportation

The future world demand for steaming coal – cited by many as the world's major alternative source of energy in the next decade – and growing handling and transportation problems will be the major subjects for discussion at CoalTrans 81, the First Coal Trade, Transportation and Handling Conference, which is to be held in London on 29th and 30th September, 1981.

Organized by the journal *Bulk Systems International*, CoalTrans 81 will provide a specialist forum to establish the handling and transportation improvements required to permit unrestricted development of the world's coal resources.

With the participation of government officials, mining executives, ship owners, coal-terminal operators, and coal users, the conference will examine transportation and handling weaknesses that, if ignored, will seriously

restrict the volume rates of supply currently indicated by the demand for steaming coal.

Internationally recognized speakers will identify the prime geographical and industrial expansion areas in coal demand, assess the rate at which current demands can be expected to rise, and comment on the changes in vessel design, ship routing patterns, and port and terminal handling systems that will be required if potentially disastrous disruptions within the overall supply chain are to be avoided.

For the full programme and registration details, write to The Conference Secretary, CoalTrans 81, McMillan House, 54 Cheam Common Road, Worcester Park, Surrey KT4 8RJ, England. Telephone: 01-330 3911. Telex: 8953141.