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THE RECOVERY OF PLATINUM, PALLADIUM, AND GOLD FROM A CYANIDE HEAP SOLUTION, WITH USE OF ION EXCHANGE RESINS

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Abstract

Ion exchange resins were investigated for the effective recovery of platinum, palladium, and gold from a pregnant cyanide heap leach solution. Screening results indicated that strong-base anion exchange resins proved to be most effective in the recovery of these metal cyanides. Gold was selectively recovered with the use of Minix, while effective recovery of gold, platinum, and palladium over the base metals, copper, and iron, was achieved with Amberlite PWA-5. Successful elution of these metal cyanides was possible from both Minix and Amberlite PWA-5, using a 2M potassium thiocyanate solution at ambient temperature. Acidic thiourea was unable to elute platinum from either Minix or Amberlite PWA-5. Effective elution of palladium and gold were, however, achieved with use of acidic thiourea. Using these results, two conceptual flow diagrams for the recovery of these metals from cyanide solutions are proposed.

Introduction

The use of ion exchange resins for the recovery of gold and silver from pregnant cyanide leach solutions has been widely investigated over the past three decades as an alternative to the carbon-in-pulp (CIP) recovery process¹⁻⁷. Most of these investigations involved researching ion exchange resins, as an alternative to conventional activated carbon. Resins are superior to activated carbon with respect to loading capacity and adsorption kinetics, and they can also be modified for specific applications.

Leaching platinum group metals with cyanide, however, is a new concept and process option. The research available is very much limited to the recovery of platinum group metals from recycled automotive catalyst using cyanide⁸⁻¹⁴. The concentration of platinum group metals in these solutions is typically between 150 ppm and 500 ppm^{8, 12-14}, compared with 1 ppm when ore or concentrate is heap-leached with cyanide¹⁵. Other proposed processes to treat pregnant cyanide solutions that contain precious and platinum group metals include thermal hydrolysis, electrowinning, and zinc cementation^{8, 15}.

However, the operation of these processes is optimal when using high-strength solution, and they would prove ineffective in treating solutions originating from heap leach operations.

Ion exchange technology, however, has proved able to recover metals selectively in low strength solutions, achieving fast adsorption kinetics and high equilibrium loadings, with low energy requirements^{2-4, 16-19}. The success of ion exchange in the gold industry for recovering gold from cyanide solutions can thus provide a basis for recovering platinum and palladium from these solutions using ion exchange resins.

Background and theory

The solution investigated comes from the second stage of a 2-stage heap leach of ore or low-grade concentrate; it is not yet established which material will be used. The first stage involves a bioleach, in which the majority of copper, iron, nickel, zinc, and cobalt are extracted from the material using a sulphuric acid solution containing thermophiles and iron (II). The second stage consists of a dilute cyanide leach, in which the platinum and palladium, as well as gold, are extracted from the bioleach residue material.

As in the case of gold, the cyanide leaching reactions of platinum and palladium can take place at ambient conditions, and follow kinetics similar to those described by gold dissolution into cyanide media to form the aurocyanide, $\text{Au}(\text{CN})_2^-$, complex in solution^{15, 20}. Platinum and palladium will thus be present in the pregnant leach solution in their cyanide complexes as $\text{Pt}(\text{CN})_4^{2-}$ and $\text{Pd}(\text{CN})_4^{2-}$ respectively.

It is known from cyanide leaching practice and research that base metals such as copper, iron, zinc, cobalt, and nickel, are readily dissolved into the cyanide leach solution. The concentrations of these base metals in the pregnant solution, in most cases, are much higher than that of the gold, platinum, and palladium^{15, 21}. Thus the base metals can have a large impact on the downstream processes to recover the precious and platinum group metals selectively from the pregnant leach solution.

Experimental

Resin pre-treatment

The ion exchange resins used in the study were transferred to a column and first back-washed with distilled water to remove the smaller particles from the bed, then washed again with an excess amount of water for a period of 2 hours.

Next, the bed was treated with 6 bed volumes of a 2M sodium chloride solution over a period of 2 hours. This was done to ensure that all the active groups on the resin were in the chloride form.

After the sodium chloride pre-treatment, the bed was again washed with an excess amount of water over a 2-hour period to remove excess sodium chloride solution from the bed.

In the case when weak-base and chelating resins were used, the resin was washed with 5 bed-volumes of a 1M HCl solution prior to the sodium chloride pre-treatment. This was done to ensure that all the functional groups on these resins were protonated.

Stock solution preparation

Synthetic solutions were prepared using $K_2Pd(CN)_4 \cdot H_2O$ (99%, Sigma-Aldrich), $K_2Pt(CN)_4 \cdot H_2O$ (98%, Sigma-Aldrich), $KAu(CN)_2$ (99%, Sigma-Aldrich), $CuCN$ (99%, Sigma-Aldrich), $K_2Ni(CN)_4 \cdot H_2O$ (Sigma-Aldrich), $K_4Fe(CN)_6 \cdot 3H_2O$ (98.5%, Sigma Aldrich) and $ZnSO_4 \cdot H_2O$ (98%, Merck). The metal salts were mixed together as they were needed, in water. In the cases when $ZnSO_4 \cdot H_2O$ and $CuCN$ were used, the molar ratios of cyanide to metal salts used were 4 to 1. In the majority of experiments, the free cyanide concentration of the stock solutions was adjusted to 150 ppm, to keep the metal cyanide complexes in their most stable form, as $Fe(CN)_4^{6-}$, $Cu(CN)_4^{3-}$, $Cu(CN)_3^{2-}$, $Zn(CN)_4^{2-}$, $Ni(CN)_4^{2-}$, $Pd(CN)_4^{2-}$, $Pt(CN)_4^{2-}$ and $Au(CN)_2^-$.

Eluant solution preparation

The two eluting agents investigated were acidic thiourea and thiocyanate. In the case of acidic thiourea, thiourea (99%, Sigma-Aldrich) was mixed with concentrated HCl (32%, Ajax Laboratories), to create a solution containing 0.5 M thiourea and 0.1 M H_2SO_4 . The thiocyanate eluant was prepared using $KSCN$ (99%, Sigma-Aldrich). The free cyanide concentration of the thiocyanate eluant was adjusted to 150 ppm by the addition of $NaCN$. A 2M solution of thiocyanate having a pH of 12 was used in the investigation.

Analytical methods

The concentrations of metals in the stock solutions were measured using inductively coupled plasma – mass spectroscopy (ICP–MS). Metal cyanide solutions were prepared as described earlier, and were used as metal calibration standards in all experimental results reported in this study.

Silver nitrate titration was used for the measurement of free cyanide in solutions. Silver reacts with free cyanide in solution, to form $Ag(CN)_2^-$, the end point of the titration is reached when a silver-sensitive indicator detects an excess of silver in solution. The end point was taken to be the final colour change from canary yellow to salmon.

Results and discussion

Resin screening

The adsorption test work involved two screenings tests. In the first screening test 12 different resins, which included weak base, chelating and strong base resins were screened, to evaluate their platinum, palladium, and gold adsorption potentials, under the conditions of the leach solution. The composition of the leach solution used in the majority of adsorption test work, was derived from leaching work reported elsewhere¹⁵, as shown in Table I.

The solution was made up synthetically, and had a free cyanide concentration of 150 ppm, pH of 10.5, and temperature of approximately 22°C.

Table I-Composition of synthetic leach solution

Metal Concentration (mg/l)						
Au	Pt	Pd	Zn	Ni	Cu	Fe
0.1	0.15	0.4	5	5	10	10

The first screening test involved contacting the different resins with the one litre of solution in rolling bottles, at a resin-to-solution ratio of 1:2000, for 24 hours. The large resin-to-solution ratio was chosen to create a competitive environment between the active sites and the different metal anions in solution. The percentage recovery of each metal was then used to evaluate the anion exchange potential and selectivity of each resin for the respective metal cyanide anions after 24 hours of contact.

From the preliminary screening test, the four resins presented in Table II were selected for further experimentation. The selection was made on the basis of high recovery of platinum, palladium, and gold, as well as low base metal recovery.

Table II-Selected anion exchange resins

Name	Type	Functionality	Capacity (eq/l)
Dowex 21K XLT	Strong Base	Trimethyl Ammonium	1.4
Amberjet 4600	Strong Base	Dimethyl Ethanol Ammonium	1.25
Amberlite PWA-5	Strong Base	Tri-ethyl Ammonium	≥ 1.00
XZ - 91419 (Minix)	Strong Base	Tri-butyl Ammonium	0.3

From Table II, it can be observed that all four resins selected were strong-base anionic resins, with different functionalities and exchange capacities. This was done specifically to evaluate the effect of exchange capacity and functionality on the recovery of platinum, palladium, and gold during subsequent test work.

The second adsorption test work involved batch kinetic evaluations and discontinuous loading test, performed on all four resins, to select only the resins that performed most favourably, when considering process development.

Batch kinetic test work involved contacting the four resins with solution in rolling bottles, at a resin-to-solution ratio of 1:1000, for 48 hours. Samples were collected at predetermined time intervals to generate kinetic loading data for each metal on each resin. The percentage recovery and loading of each metal after 48 hours on each resin are presented in Table III.

Table III-Percentage recovery and loading of metals after 48 hours

	% Metal Extraction						
Resin	Fe	Ni	Cu	Zn	Pd	Pt	Au
Dowex 21K XLT	96	96	87	98	99	99	84
Amberjet 4600	98	96	85	96	98	99	83
Amberlite PWA-5	42	99	90	100	100	100	95
Minix	3	35	15	64	61	72	98
	Metal Loading (g/l)						
	Fe	Ni	Cu	Zn	Pd	Pt	Au
Dowex 21K XLT	9.56	4.82	8.66	4.92	0.39	0.15	0.08
Amberjet 4600	9.85	4.79	8.54	4.82	0.39	0.15	0.08
Amberlite PWA-5	4.16	4.95	9.51	4.98	0.40	0.15	0.09
Minix	0.34	1.75	1.54	3.18	0.25	0.11	0.10

From Table III it can be seen that high recoveries of gold (>80 per cent), platinum (>98 per cent) and palladium (>99 per cent), were obtained with Dowex 21K XLT, Amberjet 4600, and Amberlite PWA-5. Minix showed moderate recoveries for platinum and palladium (72 per cent and 61 per cent respectively), with a high recovery of gold (>95 per cent).

High recoveries of base metals were observed onto Dowex 21K XLT and Amberjet 4600. During the initial screening test the recoveries of base metals onto these two resins were also higher than for Amberlite PWA-5 and Minix. Amberlite PWA-5, on the other hand, showed lower iron recovery than both Dowex 21K XLT and Amberjet 4600, with higher recoveries of copper, nickel, and zinc. Minix showed low base metal recovery potential, with the exception of zinc, for which the recovery was 64 per cent, which was similar to the recoveries obtained for platinum and palladium.

Comparing the recovery with the loading of each metal on the four respective resins, an immediate concern was noticed. The low concentrations of platinum, palladium, and gold in the synthetic leach solution (Table I) caused their loadings to be much lower than those of the base metals, despite their high recoveries. For example, even if 98 per cent of the gold and 3 per cent of the iron are recovered using Minix, the iron loading (0.34 g/l) on the resin is still three times higher than the gold loading (0.1 g/l).

Thus to have an effective process, the resins selected should have low base metal loading potential, in order to produce a process stream that contains high amounts of platinum, palladium, and gold, with lower amounts of base metals.

A cumulative loading test was performed to determine the loading of each metal onto the four resins under discontinuous loading conditions. The experiment involved contacting fresh synthetic solution with resin, in a ratio of 1:1000, for 24 hours. After each contact, the resin was filtered under vacuum from the solution and re-introduced into fresh solution for another 24 hours on rolling bottles. This process was repeated nine times, and at each contact a sample was taken for analysis, from which the loading of each metal could be determined by a mass balance. The cumulative loading results of all four resins, after nine contacts, are presented in Table IV.

Table IV-Cumulative loading results after nine contacts

Metal	Loading (g/l)			
	Dowex 21K XLT	Amberjet 4600 Cl	Amberlite PWA-5	Minix
Fe	20.0	10.0	n/a	n/a
Ni	7.1	6.6	6.5	n/a
Cu	8.2	5.6	2.0	n/a
Zn	14.2	11.4	20.8	1.6
Pd	1.3	1.1	1.6	0.0
Pt	0.7	0.6	0.8	0.1
Au	0.1	0.1	0.3	0.7
Total	51.5	35.5	31.9	2.4

From Table IV, it is clear that Dowex 21K XLT and Amberjet 4600 loaded significant amounts of base metals, up to 20 g/l of iron and 7.1 g/l of nickel. This observation is consistent with the literature^{2,4,5,16}, which states that non-selective adsorption occurs when using anion exchange resins containing high exchange capacities and small functional groups. These resins are able to load significant amounts of multivalent metal cyanide anions such as those of $\text{Fe}(\text{CN})_4^{6-}$, $\text{Cu}(\text{CN})_4^{3-}$, $\text{Cu}(\text{CN})_3^{2-}$, $\text{Zn}(\text{CN})_4^{2-}$, and $\text{Ni}(\text{CN})_4^{2-}$.

The reason for this is the high ionic density of these resins, which have a high affinity for higher charged anions such as those mentioned previously, as opposed to a monovalent anion such as $\text{Au}(\text{CN})_2^-$. This is clear from Table IV, with Dowex 21K XLT and Amberjet 4600 both having low gold loadings of 0.1 g/l.

Amberlite PWA-5, on the other hand, contains a larger functional group and has a lower exchange capacity than both Dowex 21K XLT and Amberjet 4600. This effect of this can be seen from Table IV, with no iron loading and low copper loading onto Amberlite PWA-5 after nine contacts.

The lower exchange capacity and larger functional group of Amberlite PWA-5 makes the resin unable to compensate the loading of the multivalent $\text{Fe}(\text{CN})_4^{6-}$ and $\text{Cu}(\text{CN})_4^{3-}$ anions. Riveros *et al.*¹⁶, concluded that anion exchange resins containing tri-ethyl functionality, had high affinity towards lower-valent metal cyanide anions. This explain the high loadings obtained for $\text{Zn}(\text{CN})_4^{2-}$ and $\text{Ni}(\text{CN})_4^{2-}$ $\text{Pd}(\text{CN})_4^{2-}$, $\text{Pt}(\text{CN})_4^{2-}$, and $\text{Au}(\text{CN})_2^-$ onto Amberlite PWA-5.

Minix proved to be very selective towards the monovalent $\text{Au}(\text{CN})_2^-$ anion. The highest gold loading of all the resins investigated, 0.7 g/l, was obtained with Minix. This was expected, because Minix were developed specifically as a gold-selective resin for use in cyanide solutions. It can also be seen that no base metals except for zinc (1.6 g/l), were present on Minix at the end of the nine contacts.

From the batch kinetics and continuous loading results, it was concluded that non-selective adsorption occurred onto both Dowex 21K XLT and Amberjet 4600, and that these two resins had high base and precious metal loading potentials. Amberlite PWA-5, however, achieved high loadings of palladium (1.6 g/l), platinum (0.8 g/l), and gold (0.3 g/l), with little to no loading of copper and iron. Minix selectively recovered all the gold from the solution over all other metal cyanides in solution. Thus from these results it was decided to continue experimentation with only Amberlite PWA-5 and Minix, because of the non-selective nature of Dowex 21K XLT and Amberjet 4600.

Effect of different parameters on adsorption

The effect of pH, platinum, palladium, and gold concentrations and base metal concentration on the adsorption potential of Amberlite PWA-5 and Minix was investigated. The test was performed in rolling bottles contacting resin with solution in a ratio of 1:1000 for 48 hours. In this section the results found will only be discussed in a qualitative manner, with no presentation of results in graph or table form.

Effect of pH

The effect of pH on the loading of platinum, palladium, and gold onto both Minix and Amberlite PWA-5 over the pH range 9 to 11 was minimal. The observation was consistent with findings in the literature², that pH had little effect on the adsorption of anions onto strong-base anionic resins.

Effect of base metal concentration

The investigation of the effect of base metal concentration, was performed using only the base metals nickel and zinc in their cyanide complexes as $\text{Ni}(\text{CN})_4^{2-}$ and $\text{Zn}(\text{CN})_4^{2-}$. This was done because of the higher loadings obtained for these metals onto both Minix and Amberlite PWA-5 compared with copper and iron. The concentrations of platinum, palladium, and gold were kept constant, while the concentrations of zinc and nickel were varied between 5 mg/l and 20 mg/l.

The loading of $\text{Pt}(\text{CN})_4^{2-}$ and $\text{Pd}(\text{CN})_4^{2-}$ on Minix decreased by 45 per cent and 40 per cent, respectively, decreasing from 5 mg/l of nickel and zinc in solution to 10 mg/l. However, no further decrease in the loading of platinum and palladium was observed as the zinc and nickel concentration were increased above 10 mg/l. The $\text{Au}(\text{CN})_2^-$ loading on Minix, however, was not affected over the entire zinc and nickel concentration range investigated. Similar decreases in the initial loading rates of $\text{Pt}(\text{CN})_4^{2-}$ and $\text{Pd}(\text{CN})_4^{2-}$ onto Minix were also observed up to a concentration of 10 mg/l zinc and nickel in solution, after which the initial loading rate stayed constant as the base metal concentration was increased above 10 mg/l. Although the loading of $\text{Au}(\text{CN})_2^-$ was not affected by zinc and nickel concentration, a 20 per cent decrease in the initial loading rate of $\text{Au}(\text{CN})_2^-$, was observed as the concentration of nickel and zinc were increased from 10 mg/l to 20 mg/l in solution.

Loading of $\text{Pt}(\text{CN})_4^{2-}$, $\text{Pd}(\text{CN})_4^{2-}$, and $\text{Au}(\text{CN})_2^-$ onto Amberlite PWA-5 was not affected up to a concentration of 10 mg/l of nickel and zinc. However, as the concentration of nickel and zinc was increased to 20 mg/l, a slight decrease in loading was observed for all three anions. A linear decrease in the initial loading rates of $\text{Pt}(\text{CN})_4^{2-}$, $\text{Pd}(\text{CN})_4^{2-}$, and $\text{Au}(\text{CN})_2^-$, was also observed as the concentrations of nickel and zinc were increased from 5 mg/l to 20 mg/l. Thus it can be concluded that zinc and nickel concentrations have a significant effect on both the loadings and initial loading rates of $\text{Pt}(\text{CN})_4^{2-}$, $\text{Pd}(\text{CN})_4^{2-}$, and $\text{Au}(\text{CN})_2^-$ onto both Minix and Amberlite PWA-5.

Effect of precious and platinum group metals concentration

The effect of platinum, palladium, and gold concentration was investigated by keeping the concentrations of the base metals shown in Table I constant, and only increasing the platinum, palladium and gold concentrations to 2-3 times their concentrations in Table I.

Increasing the platinum, palladium, and gold concentration resulted in a linear increase in both loading and initial loading rates of these three metals. Platinum, palladium, and gold loading increased by 0.27 g/l, 0.60 g/l, and 0.20 g/l respectively, with increases in their initial loading rates of 100 per cent, 100 per cent, and 95 per cent respectively.

Similar trends in both the loading and initial loading rates of platinum, palladium, and gold were observed for Amberlite PWA-5. Platinum, palladium, and gold loadings increased by 0.30 g/l, 0.85 g/l, and 0.2 g/l respectively, with a 200 per cent increase in the initial loading rate of all three anions.

Elution

Two eluting agents, potassium thiocyanate and acidic thiourea, were investigated for the elution of platinum, palladium, and gold cyanides from both Minix and Amberlite PWA-5. The elution conditions that were used are shown in Table V.

Table V-Eluting conditions

Eluting Agent	Concentration (M)	Temperature (°C)	pH	Flow (BV/h)
Acidic thiourea	0.5 M Thiourea, 0.1M HCl	60	-	10
Potassium thiocyanate	2	22	12	10

The resins were eluted with 20 bed volumes of each eluting agent under the conditions described in Table V. The percentage elution of each metal after 5, 10, 15, and 20 bed volumes are presented in Table VI.

Table VI-Percentage elution of metals with the respective eluting agents

	% Elution											
	Potassium thiocyanate											
	BV 5			BV 10			BV 15			BV 20		
	Au	Pd	Pt	Au	Pd	Pt	Au	Pd	Pt	Au	Pd	Pt
Minix	32	80	88	58	85	93	74	86	95	83	87	95
Amberlite PWA-5	17	61	50	47	96	94	68	100	100	81	100	100
	Acidic thiourea											
	BV 5			BV 10			BV 15			BV 20		
		Au	Pd	Pt	Au	Pd	Pt	Au	Pd	Pt	Au	Pd
Minix	31	63	0	83	93	0	100	98	0	100	100	0
Amberlite PWA-5	27	29	0	78	80	0	97	95	0	98	95	0

From Table VI, it can be seen that potassium thiocyanate was unable to fully elute all the metals from Minix within 20 bed volumes. The elution of platinum and palladium, however, was rapid, with 80 per cent of palladium and 88 per cent of platinum eluted within the first five bed volumes. From bed volume five onwards, however, only the elution of gold increased, with little improvement in the elution of platinum and palladium.

The elution from Amberlite PWA-5 with potassium thiocyanate was more complete than was observed for Minix. After only 15 bed volumes, platinum and palladium were completely eluted from the resin, with 81 per cent elution of gold after 20 bed volumes.

From Table VI, it can be seen that acidic thiourea was not able to elute platinum from either Minix or Amberlite PWA-5 after 20 bed volumes, at the conditions used for the elution. The reason for this is unclear, and should be investigated further.

Complete elution of gold and palladium from Minix, however, was achieved within 20 bed volumes of acidic thiourea. The elution of these two metals from Minix was fast, with almost complete elution of both metals after 15 bed volumes.

The elution of palladium and gold from Amberlite PWA-5 was slower than for Minix and the metals could not be eluted fully after 20 bed volumes of acidic thiourea. After 20 bed volumes, 98 per cent of the gold and 95 per cent of the palladium were eluted from Amberlite PWA-5. The problematic elution of the platinum cyanide complex from both resins when using acidic thiourea is being investigated further, with changes in both the thiourea and hydrochloric acid concentration during the elution process.

Conceptual flow diagrams

From a process development view, when considering both the adsorption and elution results, two possible flow diagrams could be drawn up. The first flow diagram involves a lead-lag configuration, where the gold is selectively recovered from the leach solution using Minix, and the platinum and palladium are then recovered from the Minix column effluent using Amberlite PWA-5. Literature on gold recovery using Minix indicated that effective elution of gold from the resin could be achieved with acidic thiourea. Although the elution of the platinum cyanide complex is problematic when using acidic thiourea, this complex loads poorly onto Minix, and thus minimal losses can be expected. Efforts to find an effective elution method for the recovery of metals from Amberlite PWA-5 are still ongoing. The flow diagram for the first conceptual configuration is presented in Figure 1.

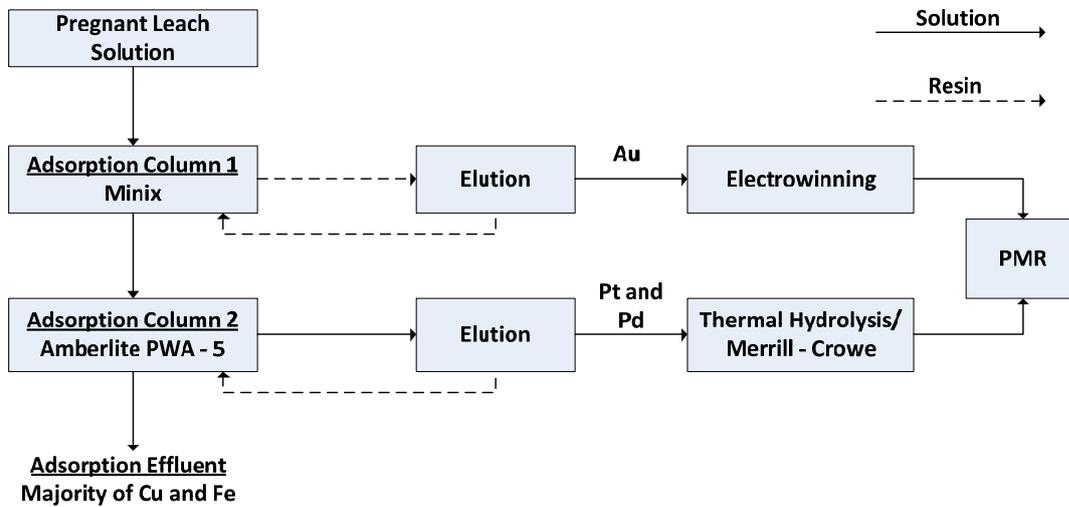


Figure 1-Conceptual flow diagram – 1

The flow diagram in Figure 1 provides a means where a high-grade gold stream could be produced that can then be processed using electrowinning. From the second adsorption stage, a process stream containing the majority of platinum and palladium will be produced, which can be treated by a number of recovery processes, ultimately producing a stream that can be sent directly to a precious metals refinery.

The second conceptual flow diagram involves the recovery of all three metals, i.e. platinum, palladium, and gold, using only Amberlite PWA-5. Adsorption results indicated that gold can also be recovered effectively with the use of Amberlite PWA-5. The second conceptual flow diagram is presented in Figure 2.

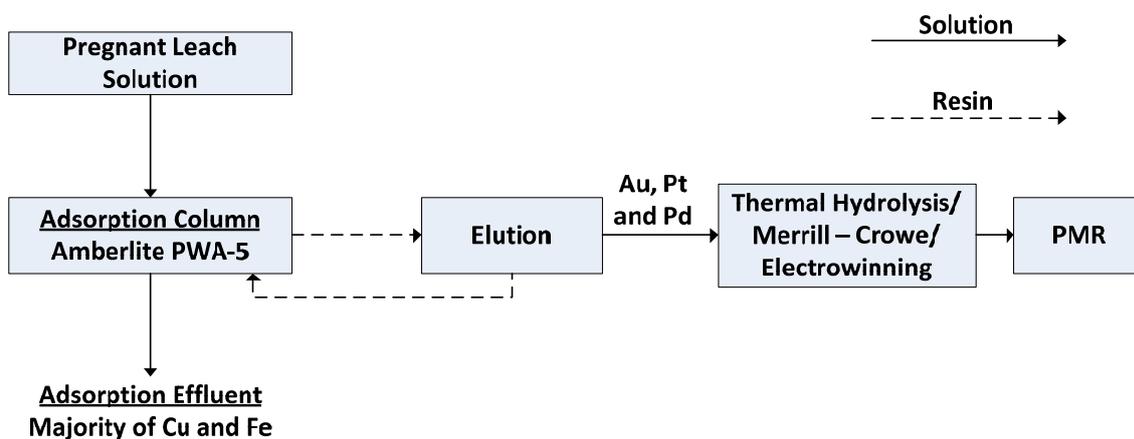


Figure 2-Conceptual flow diagram – 2

This flow sheet will produce a process stream containing the majority of gold, platinum, and platinum coming from the cyanide heap leach. The metals can be recovered by a number of processes, including zinc cementation, electrowinning, and thermal hydrolysis.

Conclusion

The use of strong-base anion exchange resins for the recovery of platinum, palladium, and gold from cyanide solutions has generated promising results. Both the adsorption and elution of these metals could be performed selectively from a dilute solution, using different kinds of strong-base resins.

Much research, however, is still needed to be able to compare this process, technically and financially, with existing PGM and gold recovery processes. Additional research into possible process alternatives, especially for the recovery of platinum and palladium from high-strength eluate solutions, is also required.

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