



Separation and recovery of some platinum group metals (PGMs) by means of selective photocatalytic reduction

by R.J. Kriek, W.J. Engelbrecht, and J.J. Cruywagen*

Synopsis

Selective photoreduction, using the semiconductor TiO_2 under UV irradiation, has been investigated as a possible recovery and/or separation method for the PGM's rhodium, palladium and platinum. The effect of pH value and concentration of the sacrificial reducing agent (ethanol) on the rate of reduction of Rh(III), Pd(II) and Pt(IV) ions have been determined separately for these ions as well as for some mixtures. Conditions favourable for the reduction and adsorption (or precipitation) on TiO_2 have been established. It has been found that already deposited Pt or Pd metal promotes reduction of Rh(III) ions at a pH value where it would not otherwise be reduced. A method for the separation of a mixture comprising Rh(III), Pd(II) and Pt(IV), based on photoreduction and adsorption on the TiO_2 , is proposed.

Introduction

The economic importance of the platinum group metals (PGMs) for South Africa is well known. According to current estimates about 72 per cent of the world's deposits (all PGMs) are found in South Africa and the country produces approximately 60 per cent (286 tons per year) of the world output of three of these metals, namely platinum, palladium and rhodium^{1,2}. The PGMs are extracted from ores but, because of their high value, are also recovered from industrial residues of variable composition and quality. The development of new technology for the extraction, recovery and separation of these metals is therefore of special interest.

Important criteria in designing separation processes are, amongst other things, high separation efficiency and minimum losses of noble metals. Ecological considerations are often crucial and can have a great effect on production costs. Current separation processes are quite complex and involve many steps, such as solvent extraction, ion exchange, precipitation, crystallization and distillation². The actual procedure followed, depends in most cases on the composition of the mixture. The solvents and other chemicals required in the separation techniques are quite often costly. Considerations such as these have led us to investigate selective photocatalytic reduction as a possible separation and/or recovery method for the PGMs.

The semiconductor TiO_2 was used as photocatalyst. Although TiO_2 is only a UV absorber it is not expensive, insoluble under most conditions, photostable and not toxic. Irradiation of TiO_2 results in the production of electrons in the conduction band and positive holes in the valence band which can be manipulated to accomplish redox reactions. In the case of water purification, for example, organic pollutants are oxidized in the presence of oxygen to form water, carbon dioxide and mineral acids³. The reduction of metal species with high redox potentials, on the other hand, can be accomplished in the presence of any suitable oxidizable substance. The photodeposition of metals such as gold, silver, palladium, platinum, iridium and rhodium on TiO_2 during UV irradiation has been reported by several authors⁴⁻⁷. The present paper deals with the photocatalytic reduction of ions of three PGMs, namely rhodium(III), palladium(II) and platinum(IV). Due to the exploratory nature of the work the systems investigated had to be kept as simple as possible. The characteristic photochemical behaviour of each element was first determined individually to establish conditions on which a separation procedure can be based. However, when mixtures of the metal ions were investigated, it was found that the reduction behaviours were different from those of the individual ions. For example, already deposited platinum strongly promotes the reduction of rhodium(III), a factor which impedes selective reduction. The method proposed for the recovery and separation of platinum(IV), rhodium(III), and palladium(II) is based on selective reduction but it also makes use of adsorption of species by TiO_2 from the solution at a suitable pH value. The method was considered to be particularly suitable for solutions of very low concentrations and to have the advantage of being simple and efficient.

Theoretical considerations

The difference between metals, semiconductors and insulators is well known (Figure 1)⁸.

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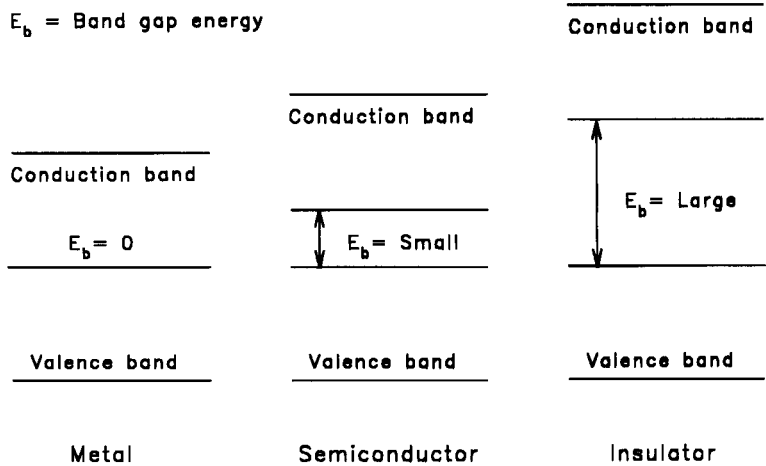


Figure 1—Band gap energies in metals, semiconductors and insulators

Acknowledgements

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- In metals the electrons completely fill the valence band and there is no energy gap between the valence and conduction bands. At absolute zero the conduction band is empty. Above absolute zero, electrons at the top of the highest occupied levels can gain thermal energy and move into the low-lying empty levels of the conduction band.

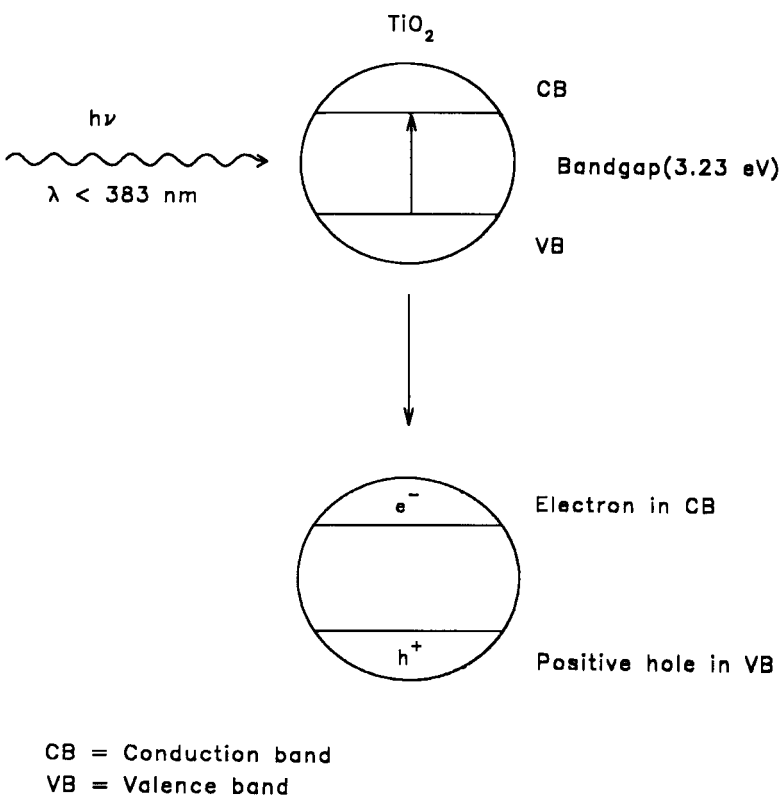


Figure 2—Excitation of an electron in TiO_2 on absorption of a photon with an energy greater than 3.23 eV

- In insulators and semiconductors the valence band is completely filled and an energy gap exists between this band and the next higher energy band. If the energy gap is large, there is little chance for the electron to be excited into the empty conduction band and the material is an insulator. If there is only a small energy gap the electron is rather easily excited into the conduction band thereby also creating a positive hole in the valence band as an additional carrier and the material can now conduct electricity and is called an intrinsic semiconductor.

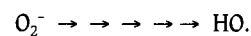
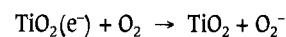
Anatase, one of the crystal structures of titaniumdioxide (TiO_2), is a common semiconductor photocatalyst. Anatase has an energy band gap of 3.23 eV which corresponds to wavelength 383 nm⁹. Therefore, when a suspension of TiO_2 is illuminated by light of a wavelength equal to or shorter than 383 nm, excitation of an electron from the valence band to the conduction band will take place producing a positive hole in the valence band and an electron in the conduction band (Figure 2). The electron and positive hole consequently migrate to the surface of the TiO_2 particle.

The electrons can then be donated to species adsorbed on the TiO_2 -surface, for example to the chloro-complexes of the PGMs thereby causing reduction. The positive holes on the other hand can accept electrons thereby causing oxidation of, for example, organic pollutants in water^{3,10}.

By changing the energy of the photons beyond what is needed to cause excitation across the band gap and/or by changing conditions of the solution in contact with the semiconductor particles, selectivity could be accomplished.

In order to accomplish either an oxidation or reduction the rate of recombination of the electron-hole pairs has to be reduced to such an extent as to allow utilization of both the electron and positive hole.

- To perform an oxidation by using the positive holes, available in the valence band, a sacrificial oxidizing agent (SOA) is needed to take up the electrons in the conduction band. Oxygen (O_2) is generally used as SOA and leads to the formation of oxygen radical anions, which through a series of further reactions form hydroxyl radicals¹⁰.



The hydroxyl radical, with a standard reduction potential of + 2.85 eV, is a very strong oxidizing agent and then assists in the oxidizing process¹¹.

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- In the same way a sacrificial reducing agent (SRA) is needed to supply electrons to the positive holes to allow the electrons in the conduction band to be used for the reduction of the PGMs. In our investigation ethanol and sucrose (cane sugar) were used as SRA's.

Experimental

Reagents and solutions

All solutions (stock and reaction mixtures) were prepared with deionized water obtained from a Millipore Milli-Q system.

Stock solutions were prepared from Pt(metal), PdCl₂ (Fluka Chemie AG and PGM Chemicals (Pty) Ltd) and RhCl₃·3H₂O (Merck and PGM Chemicals (Pty) Ltd).

Platinum metal was dissolved in boiling aqua regia with an excess of hydrochloric acid to expel nitrogen compounds and then evaporated to form chloroplatinic acid, H₂PtCl₆·6H₂O.

To H₂PtCl₆·6H₂O, PdCl₂ and RhCl₃·3H₂O a calculated amount of 34 per cent HCl was added before the solution was made up with deionized water to prevent unwanted hydrolysis reactions. All stock solutions were 1 M in Cl⁻.

Degussa P25 TiO₂ (Anatase) was used as photocatalyst, the amount of TiO₂ in suspension being 2 g·dm⁻³. Sodium hydroxide solutions were prepared using NaOH pellets (Merck) and were standardized with potassium hydrogenphthalate. HCl was standardized with NaOH and borax. Ethanol (Merck) and commercial cane sugar were used as sacrificial reducing agents.

Apart from the PGMs which were added as their chloride salts the reaction mixtures comprised TiO₂ and ethanol (or sugar) and water. The pH value was adjusted by the addition of either sodium hydroxide or hydrochloric acid.

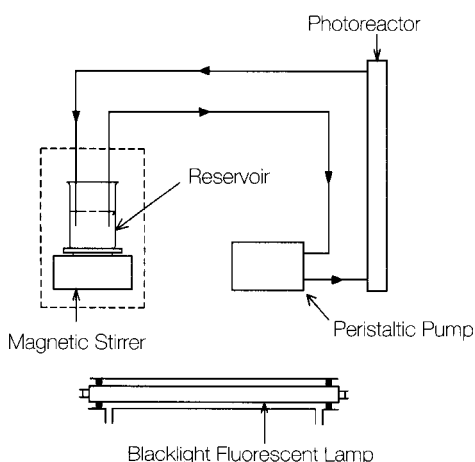


Figure 3—Diagram of the photoreactor

The concentrations of the metal ions in solution were determined by inductively coupled plasma (ICP) emission spectroscopy using a Varian Liberty 200 instrument.

Design of the photoreactor

A diagram of the reactor model¹² chosen for our laboratory investigation is shown in Figure 3.

The photoreactor consists of a blacklight fluorescent lamp within a glass cylinder, with an inlet and outlet. The reactor is sealed at the ends with O-rings. The blacklight fluorescent lamp emits light between 300 and 425 nm with a peak at 350 nm which is below 383 nm and therefore efficient for electron excitation. A peristaltic pump circulates the solution or suspension of 500 ml from a reservoir through the photoreactor under continuous illumination. The pumping speed throughout the investigation was kept at 400 ml·min⁻¹.

The suspension in the reservoir is continuously stirred by means of a magnetic stirrer and during a run samples are periodically taken from the reservoir for analysis.

Results and discussion

Effect of the concentration of the SRA (Ethanol) on the reduction rate of Rhodium(III)

To investigate the effect of ethanol on the reduction rate of Rh(III) the pH value was kept constant at 1.3 and the ethanol concentration varied from 0 to 2.1705 mol·dm⁻³. The results in Figure 4 show that the reduction rate of Rh(III) increases as the concentration of the SRA (ethanol) is increased.

Without ethanol no reduction of Rh(III) takes place which demonstrates the necessity of a SRA to provide indirectly the electrons required for the reduction process. It is seen that the rate of recombination of the electron-hole pair is slowed down by increasing the concentration of the SRA, thereby giving the excited electron a longer lifetime which leads to a higher reduction rate of Rh(III).

An ethanol concentration of 0.13 mol·dm⁻³ (curve no. 5) was chosen as standard for the subsequent experiments.

Individual Photocatalytic Reduction of Rh(III), Pd(II) and Pt(IV) and the Influence of pH Value

The conditions for these experiments were the same as before. Only the pH value was varied by addition of sodium hydroxide while the concentration of the SRA was kept constant at 0.13 mol·dm⁻³.

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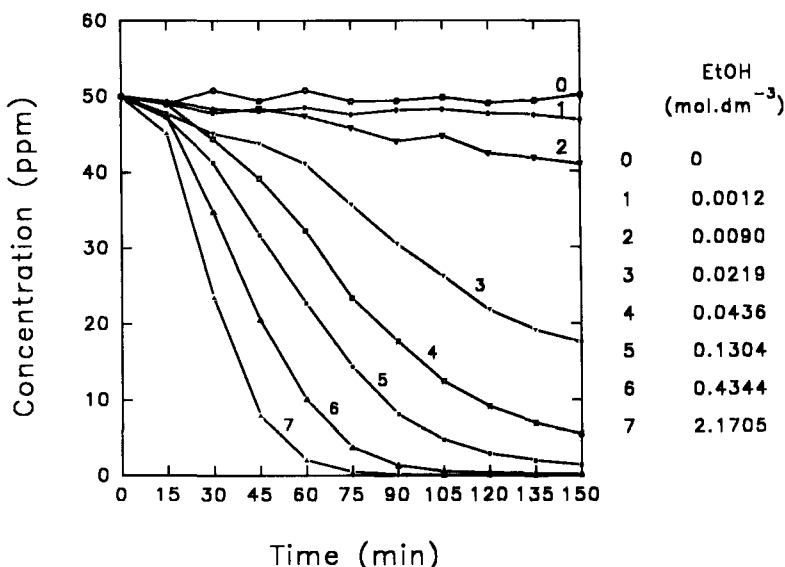


Figure 4—Rate of photocatalytic reduction of Rh(III) with variation of the ethanol concentration

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Rhodium(III)

The results of the reduction process are shown in Figure 5. It is seen that the rate of reduction reaches a maximum at pH value of 1.87 and then decreases with increasing pH value. At pH value 3.1 Rh(III) shows resistance to reduction and stays in solution. Upon further increase of the pH value, Rh(III) is still not reduced but there is a marked decrease in the Rh(III)-concentration in solution before the sample has been illuminated. This is due to either precipitation or adsorption of Rh(III) onto TiO₂. At a pH value of 11.75 Rh(III) is almost completely precipitated and/or adsorbed onto TiO₂ but on further increase in pH value it goes into solution again.

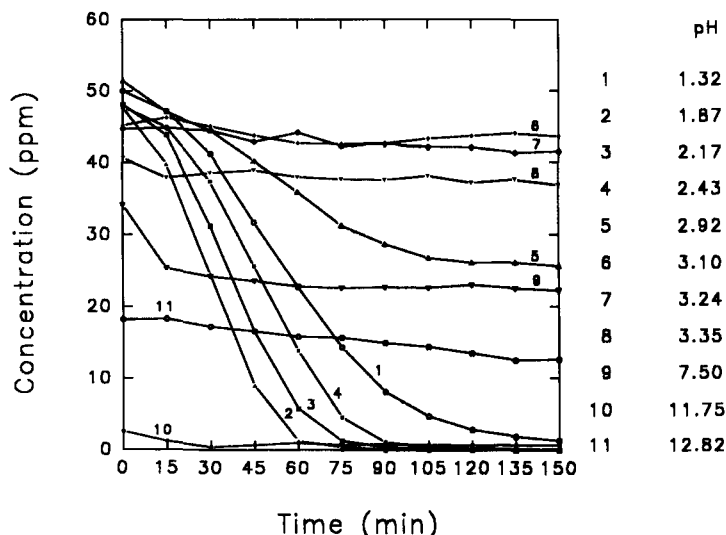


Figure 5—Rate of photocatalytic reduction of Rh(III) with variation of pH

From these results it follows that separation of Rh(III) might be obtained at two different pH values, namely (i) pH = 3.1, where Rh(III) stays in solution resisting reduction, and (ii) pH = 11.75, where Rh(III) is almost completely reduced and precipitated or adsorbed onto TiO₂ as a reduced species.

Palladium(II)

If the results of the reduction of Pd(II) shown in Figure 6 are compared with those of Rh(III) (Figure 5), it is seen that at a pH value of 3.1, where Rh(III) remains in solution, Pd(II) is entirely present in solution and upon illumination it is completely reduced to Pd metal. As in the case of Rh(III), all Pd(II) is adsorbed onto TiO₂ at a pH value of 11.8.

These results indicate that separation of Rh(III) and Pd(II) may be accomplished at a pH value of 3.1 by means of selective photocatalytic reduction in that Rh(III) is kept in solution while Pd(II) is reduced.

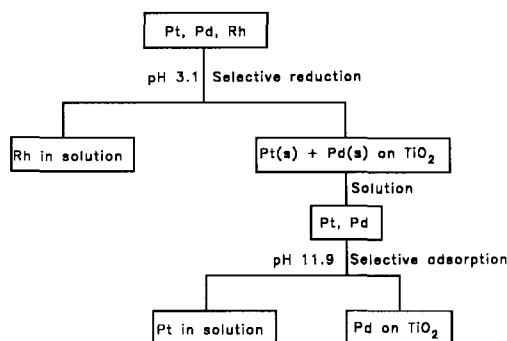
Platinum(IV)

From the results pertaining to zero illumination time (Figure 7) it is seen that Pt(IV) remains in solution over the whole pH range since no adsorption and/or precipitation occurs. Separation of Pt(IV) from Pd(II) and Rh(III) may therefore be obtained at a pH value of 11.8 by means of selective adsorption onto TiO₂. Pt(IV) stays in solution while Rh(III) and Pd(II) are completely adsorbed onto TiO₂.

Possible schemes for separation

From the previous experiments it is evident that separation of Pt(IV), Pd(II), and Rh(III) can be attempted via two different routes. Both routes make use of selective adsorption and selective photocatalytic reduction and differ only in the order in which the two processes should be carried out.

Scheme 1



Separation and recovery of some platinum group metals (PGMs)

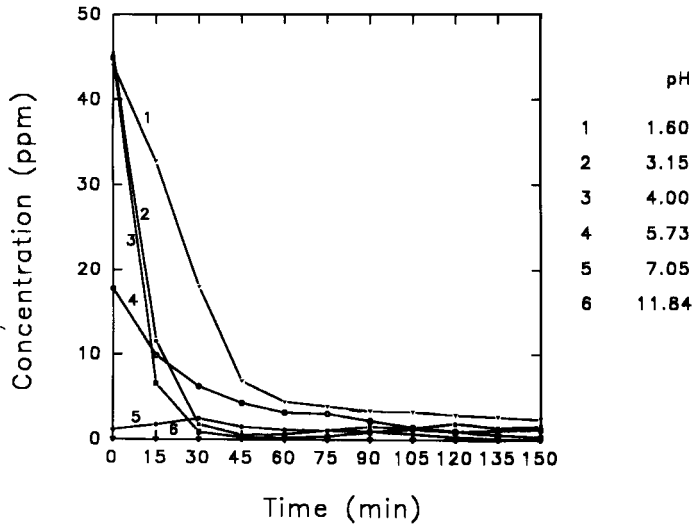


Figure 6—Rate of photocatalytic reduction of Pd(II) with variation of pH

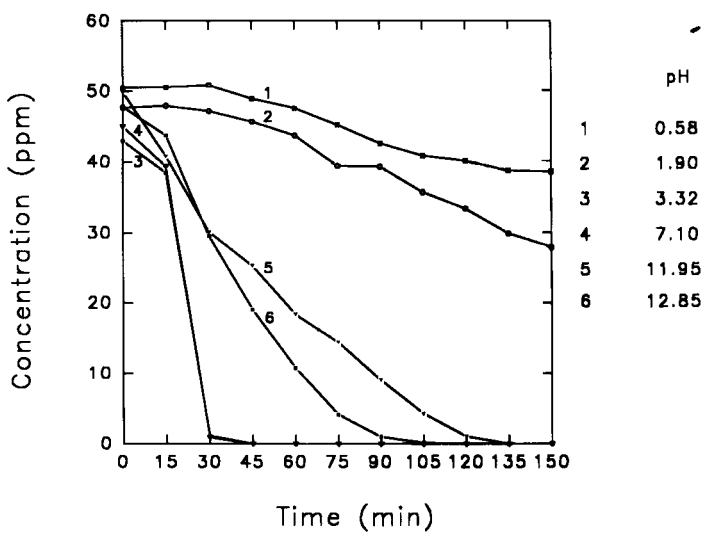


Figure 7—Rate of photocatalytic reduction of Pt(IV) with variation of pH

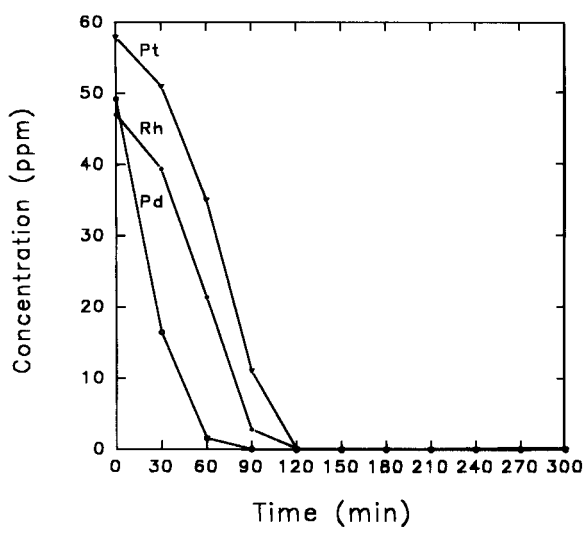
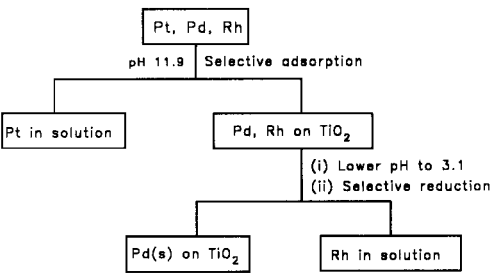


Figure 8—Rate of photocatalytic reduction of a mixture of Rh(III), Pd(II) and Pt(IV) at pH = 3.9

According to this method Rh(III) is separated from Pt(IV) and Pd(II) by means of selective photocatalytic reduction at a pH value of 3.1 followed by separation of Pt(IV) and Pd(II) by means of selective adsorption at a pH value of 11.9.

However, actual separation of Rh(III) from Pt(IV) and Pd(II) by means of selective photocatalytic reduction at a pH value of 3.1 did not succeed because Rh(III) did not resist reduction as was inferred from previous experiments (Figure 8).

Scheme 2



According to this method selective adsorption of Pd(II) and Rh(III) at a pH value of 11.9 is carried out as the first step, thereby separating these ions from Pt(IV). Pd(II) and Rh(III) are then separated at a pH value of 3.1 by means of selective photocatalytic reduction. Application of the principle shows that selective adsorption of Pd(II) and Rh(III) at a pH value of 11.9 should be successful as can be seen from the results of an experiment where a 500 ml solution containing 50 ppm of each of Pt(IV), Pd(II) and Rh(III) was stirred for five hours in the presence of 1g TiO₂. Pt(IV) remains in solution while Pd(II) and Rh(III) are completely precipitated and/or adsorbed onto TiO₂ (Figure 9).

The selective reduction of Pd(II) in the presence of Rh(III) at a pH value of 3.1 also proved to be successful (Figure 10). The pH value was 3.3 when illumination was started and decreased to 2.8 at the end of the experiment. It has been shown in previous experiments that the reduction of Rh(III) occurs at a pH value lower than 3.1. The slight decrease in the Rh(III)-concentration during the reduction of Pd(II) can thus be attributed to a fall in pH below the critical value of 3.1.

Separation and recovery of some platinum group metals (PGMs)

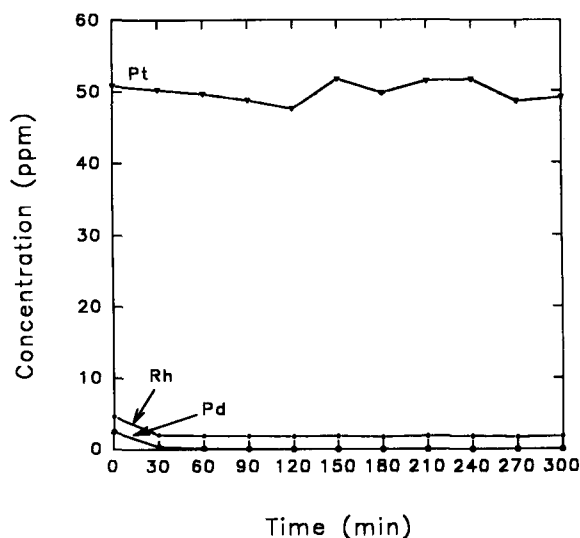


Figure 9—Selective adsorption of Pd(II) and Rh(III) onto TiO_2 at pH = 11.9

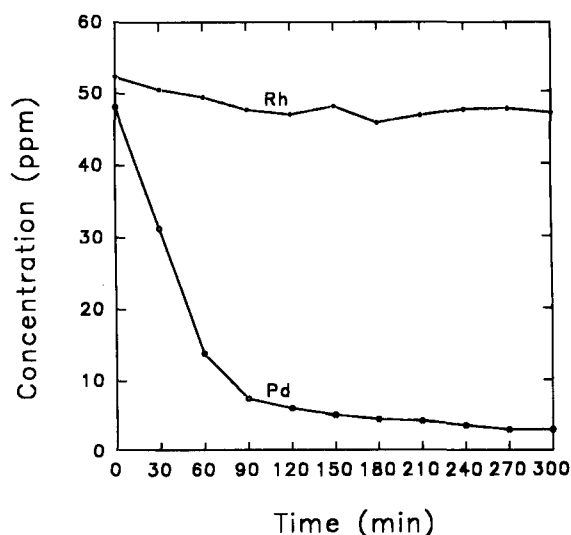


Figure 10—Selective photocatalytic reduction of Pd(II) in the presence of Rh(III) at pH = 3.3

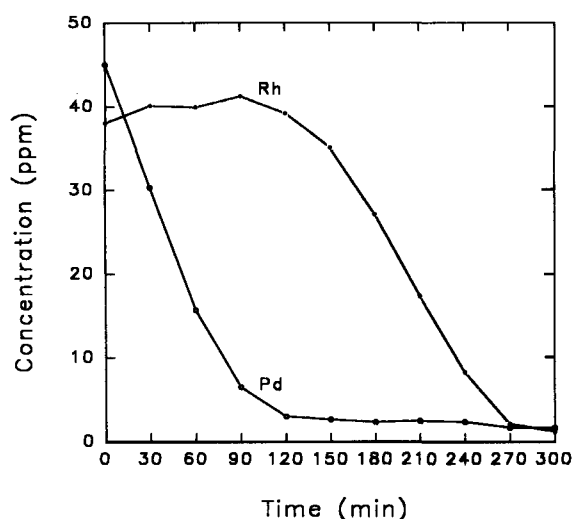


Figure 11—Photocatalytic reduction of Pd(II) and Rh(III) at pH = 3.3 after adsorption at pH = 11.9

The two experiments described previously, i.e., the selective adsorption of Pd(II) and Rh(III) and the selective reduction of Pd(II) have been carried out independently. Subsequently selective adsorption and selective photocatalytic reduction were combined in that a solution of Pd(II) and Rh(III) was first stirred for two hours in the presence of 1g TiO_2 at a pH value of 11.9 simulating the conditions for the selective adsorption of Pd(II) and Rh(III) before the pH value was lowered to 3.3 for the selective reduction of Pd(II) by illumination in the presence of 0.13 mol. dm^{-3} ethanol.

The Pd(II) and Rh(III), however, did not separate as expected. Reduction of Rh(III) started almost as soon as all the Pd(II) was deposited on the TiO_2 . Again there was a drop in pH value from 3.3 to 2.7 (Figure 11).

This dramatic change in the reduction pattern for Rh(III), shown in Figure 11, can be ascribed to the presence of other Rh(III)-species formed at high pH values.

- At the outset, at low pH values, the Rh(III) solution has a red-pink colour due to complexes of the type $[\text{Rh}(\text{H}_2\text{O})_6\text{Cl}_n]^{3-n}$, e.g. $[\text{Rh}(\text{H}_2\text{O})_4\text{Cl}_2]^+$.
- Raising the pH value to 11.9, in order to perform selective adsorption, gives rise to a yellow solution, indicating the formation of a different complex ion, most likely the $[\text{Rh}(\text{H}_2\text{O})_5\text{OH}]^{2+}$ complex ion¹³.
- When the pH value is lowered again, to 3.1, to carry out selective reduction, the solution keeps its yellow colour. The fact that the Rh(III) solution does not return to its original colour shows the kinetic stability of the complex formed at high pH values.

In order to separate a mixture of Pd(II) and Rh(III) successfully the reduction of Rh(III) must be prevented. The pH value of the solution, as a first step, was kept constant between 3.1 and 3.4. This had a dramatic effect in preventing the total reduction of Rh(III) (Figure 12). It can be seen that Rh(III) was reduced only after Pd(II) had been almost fully deposited onto TiO_2 .

As a second measure, to prevent reduction of Rh(III), the photocatalytic activity of the TiO_2 can be reduced by lowering the concentration of the SRA (ethanol) and thereby reducing the availability of the excited electrons.

Such an experiment was carried out in which the ethanol concentration was lowered from 0.13 to 0.087 mol. dm^{-3} . This had the desired effect in that the reduction of Rh(III) was prevented and the separation of Pd(II) and Rh(III) was successfully accomplished (Figure 13).

Separation and recovery of some platinum group metals (PGMs)

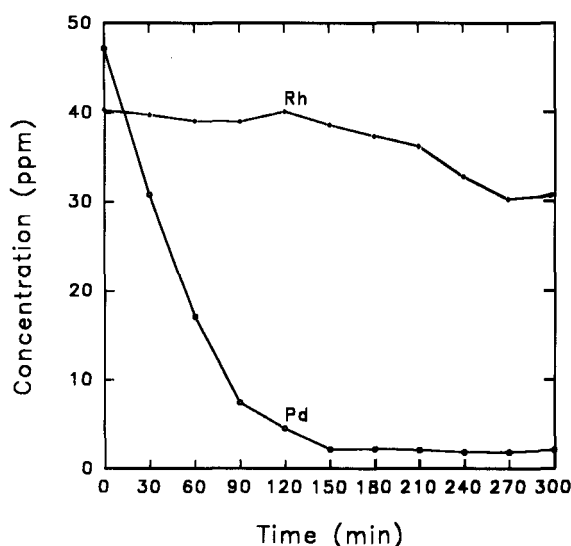


Figure 12—Photocatalytic reduction of Rh(III) and Pd(II) at 'constant' pH(3.1-3.4) preceded by adsorption

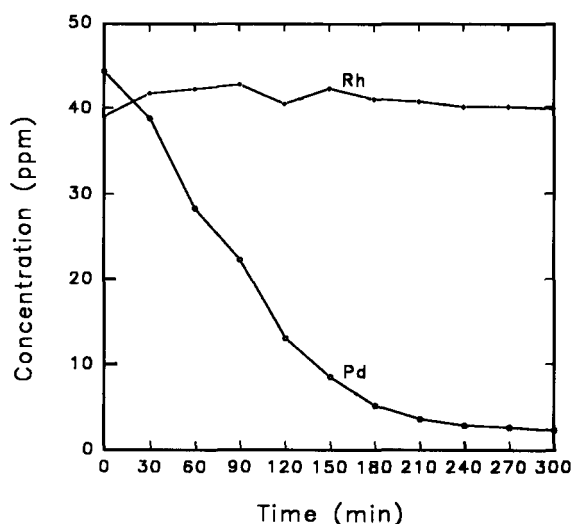


Figure 13—Photocatalytic reduction of Rh(III) and Pd(II) at 'constant' pH(3.1-3.4) with a decrease in the SRA-concentration preceded by adsorption

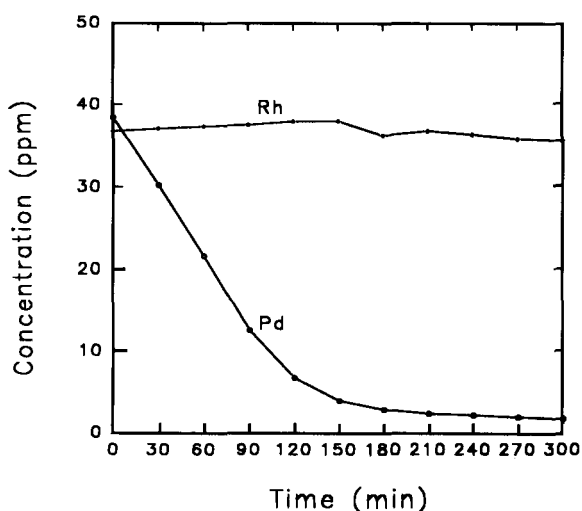


Figure 14—Selective photocatalytic reduction of Pd(II) and Rh(III) with table sugar as SRA(0.0006 M). The pH was kept between 3.1 and 3.35

Sucrose as SRA

Having demonstrated that Rh(III) and Pd(II) can be separated satisfactorily with ethanol as SRA, the use of another SRA with ethanol as SRA, the use of another SRA was investigated, namely sucrose (table sugar).

Sucrose can be expected to be a much stronger SRA than ethanol because it has more sites per molecule where oxidation can take place. This expectation was confirmed by the successful separation of Rh(III) and Pd(II) using sugar as a SRA at a concentration of $0.0006 \text{ mol.dm}^{-3}$ or 0.1 g/500 ml (Figure 14).

Conclusion

We have shown that Pt(IV) can be separated from Pd(II) and Rh(III) by means of selective adsorption of the latter onto TiO_2 at a pH value of 11.9. Subsequent separation of Pd(II) and Rh(III) by means of selective photocatalytic reduction at a pH value of 3.1 can then be accomplished.

Both ethanol and sugar are suitable as sacrificial reducing agents, but various other compounds can be considered depending on the requirements of the system.

Although the blacklight fluorescent lamp proved to be a sufficient energy source, especially for a laboratory investigation, sunlight can be utilized in the recovery of the PGMs; separation of the PGMs by using sunlight warrants further investigation. For the system investigated, the process of photocatalytic reduction can be easily manipulated by either changing the pH value or the concentration of the SRA.

By using the photocatalytic reduction process described in the paper it is possible to recover *very low concentrations* of the metals, especially of Rh, which is the most valuable component. Only one stage has been used in the procedure as described. If implemented on an industrial scale more than one stage may be required. For example, if as a result of a rather high Rh:Pd ratio the reduced Pd would contain a small amount of Rh, the metals can be brought into solution again and separated by a second reduction.

The results obtained for the chosen model system, comprising Rh(III), Pd(II), and Pt(IV), illustrates the potential of photoreduction as a recovery and/or separation method for PGMs. ♦

OBITUARY



R.C.J. Goode: 1909–1995

An association of 59 years with The South African Institute of Mining and Metallurgy, ended on 26th February, 1995, with the death of Robert Charles Jeffrey Goode, known to his friends as Jeff.

Jeff was born in Hastings in England, spent his childhood in Zambia (then known as Northern Rhodesia), and returned to England for his schooling. After obtaining a B.A. degree at Cambridge and undergoing a short practical introduction to mining at the Camborne School of Mines, he obtained bachelor's and master's degrees in mining and metallurgical engineering at McGill University in Montreal, Canada. In 1936, he joined the staff of East Geduld Mine as a Junior Mining Engineer. During the same year, he joined the SAIMM.

He was involved for a few years in war service in Italy. After the war, he made rapid progress in the Union Corporation Group, being General Manager successively of Marievale Gold Mine Ltd and St Helena Gold Mine Ltd, after which he held the posts of Consulting Engineer and Chief Consulting Engineer. In 1967 he was appointed Mining Executive Director of Union Corporation Ltd, retiring from that position in 1973. During this latter period, he served on the Executive and Gold Producers' Committee, and was President of the Chamber of Mines in 1972.

Jeff was always an enthusiastic mining man. He never overlooked the theoretical or academic background of mining. He applied this knowledge practically, particularly to gold mining, but also took great interest in other precious-metals mining, as well as coal and base-metal mining, regardless of whether the mining processes were underground or surface-pit operations. He realized, however, that technical knowledge had to be supplemented with the management techniques of planning, organizing, motivating, and controlling those involved in the operations. Likewise, he never overlooked the fact that mining is a business and that entrepreneurial skills also need to be applied.

In addition to the aforementioned activities, he enjoyed playing golf, but his greatest interest lay in institutional and society matters. As previously mentioned, he joined the SAIMM as an Associate in 1936. Later, after becoming a member, he was elected to Council and served with distinction as Honorary Editor of the *SAIMM Journal*, was Chairman of the Programme Committee, and was elected President in 1968. His presidential address was entitled 'The Institute in retrospect and prospect'. In it he portrayed the important contribution the Institute had made to the mining industry, but stressed that circumstances were changing, and suggested ways and means of tackling the problems that lay ahead and the role that the Institute should play.

As past-president, Jeff served for many more years on the Council of the SAIMM. But these weren't his only activities. In 1972 he was elected President of the Associated Scientific and Technical Societies after having served on its Controlling Executive for some years. After he had retired to the Natal North Coast, the Natal Scientific and Technical Societies nominated him as Natal's representative on the AS&TS Controlling Executive, and he travelled to Johannesburg regularly to attend meetings in this capacity.

Jeff was also a Fellow of the Institute of Mining and Metallurgy, London, and he attended several Commonwealth congresses. As is well known, the programmes of these congresses consist of technical-paper presentations, technical discussions, and technical visits, most often followed by banquets in various areas. He was often asked to propose a vote of thanks to the hosts on these occasions.

In recent years, Jeff became very fond of reminiscing about his mining experiences. In 1994 he was called upon to propose the toast to South Africa at the SAIMM Centenary banquet, which was held on 24th March. This was to be his final Institute duty. ♦