Small-scale recovery of noble metals from jewellery wastes

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Synopsis

This paper describes an investigation to separate and selectively recover silver, gold and platinum from jewellery and laboratory wastes, and produce technical grade material suitable for training purposes. Nitric acid was employed to dissolve and separate silver and other impurities from gold and platinum, which were then dissolved in aqua regia and precipitated selectively. The current work quantified the effects of temperature, stirring rate, leaching agent concentration and solid:liquid ratio on the recovery of the various noble metals and in summary yielded the optimum treatment conditions in each case. It was found that increased leaching reagent concentration (in the case of Ag), temperatures and stirring rates enhanced the recovery kinetics and optimum yield of the three noble metals. An increased liquid to solid ratio also enhanced the recovery of the silver. Recovery yields 97.1%, 99.9% and 99.4% for silver, gold and platinum, respectively, were obtained. The recovered silver, gold and platinum had purities of, respectively, 98.3%, 99.1% and 99.0%. A cost saving of approximately R10 000 per kilogram of scrap refined could be achieved by the in-house treatment of the waste, not to mention the time-saving benefit.

Introduction

Apart from all their other applications and uses, gold and platinum, together with silver, form the bulk of the metallic elements used on their own or in the form of various alloys in jewellery manufacture. It is therefore not surprising that mining and recovery of these metals have dominated world history and human endeavour from the earliest times. Gold mining in South Africa has been conducted on a commercial scale for nearly a century and a half. The production of platinum is also well established and takes place at a number of mines in various parts of the country. South Africa has been the major gold supplier in the world in the first half of the twentieth century, and continues to be an important role player in this market. The platinum price has not only surpassed that of gold, but so has its output, and it has once again placed South Africa in the forefront as the leading supplier in the world of a much sought-after noble metal.

The growing demand for high purity precious metals to be used in high-tech applications, the need for efficient and clean process technologies and the increasing amount of wastes containing low grade/secondary noble metals, all contribute to a greater emphasis on recovery, recycling and refining of precious metals[1]. Recovery of noble metals from wastes is very different from commercial mining operations because the chemical composition of the waste normally varies widely and dictates the approach to be followed and process to be used[2–4]. Recovery of noble metals from jewellery waste is not a new concept and has been described in the literature by various investigators[5–8] and patents[9–10].

Although cyanide leaching remains the overwhelming option for the recovery of gold because of its simplicity of operation and economic viability, it suffers from inherent drawbacks such as toxicity and slow leaching[11], both of which are significant disadvantages for a small-scale operation. Leaching with solutions other than cyanide as a means for the effective and less hazardous recovery of gold, silver and platinum therefore remains an attractive option[12]. A number of review papers describing developments and changes in gold processing have been published in the literature (see for example references cited by Prassad *et al*.[13]). Dube[14] recently described the use of various pyromet-

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Small-scale recovery of noble metals from jewellery wastes

allurgical and wet chemical refining procedures, such as the Miller process, Wohwill electrolysis, leaching with nitric acid, and the cupellation process (used in classical assaying of gold) for the recovery of gold from recycled materials. Chmielewsky and co-workers[15] developed a process for gold reclamation from jewellery wastes that consists of a combination of roasting and aqua regia leaching before final selective solvent extraction of the gold with diethyl malonate.

The present investigation was prompted by a need to find an 'in-house' solution to the recovery of noble metals from jewellery wastes collected during practical sessions of students being trained in jewellery design, in order to save time and financial costs (of sending the wastes to a commercial operation for recovery) and to supply a 'technical' grade purity of material for training purposes in introductory courses. A significant number of students following degrees and diplomas in Fine Arts: Jewellery Design at Tshwane University of Technology (TUT - previously Technikon Pretoria) generated a substantial amount of wastes during their training each year that was used as the starting material for this investigation. This paper will describe the work that has been done on this jewellery waste and the results that were obtained in the recovery of silver, gold and platinum from it.

Experimental procedure

Sample collection

During the training of jewellery design students, each bench is equipped with a leather collection bag and at the end of a training session the contents of each one is emptied into a clean glass jar. The contents of this jar, as well as some silver residues collected during the practical sessions of analytical chemistry students, were used as starting materials in this investigation.

Materials and equipment

Before commencing any work, a magnet sandwiched inside a plastic sheet /bag was used to remove all iron filings and traces of magnetite and hematite. All unwanted pieces of wood, plastic and paper were also removed. Thereafter a sieve analysis was performed on a 100 g sample of the jewellery waste, using a Model ES 200 Mark IV electronic sieve shaker. A representative portion of the jewellery waste was analysed on an ARL 9400 XP+ XRF spectrometer using a wide confidence limit program. Leaching tests were performed in Pyrex glass beakers, using magnetic stirrers equipped with heating control. Chemical analysis of leach solutions and final products was performed on a Spectro ICP-OES apparatus at the appropriate wavelengths, as shown in

Table I. All reagents used were of analytical grade and deionized water was obtained from a Millipore installation.

An approach of selective dissolution and recovery was followed to separate the various components from the waste mixture. The acids used were in their concentrated solutions 32% m/v HCl, 63% m/v HNO_3 and 98% m/v H_2SO_4.

Silver recovery

A cleaned sample of 50 g of the jewellery waste was dissolved in 52.8 ml of a 1:1 diluted nitric acid solution (~ 32% m/v) by slowly adding it to the filings[17]. When no more visible brown fumes (of NO_2) evolved from the solution, it was left to cool and then diluted with distilled water in the proportion 1:3 to a concentration of approximately 8% m/v nitric acid. The resultant solution was filtered through Whatman no. 40 paper to separate the undissolved gold and platinum particles from the dissolved silver. This residue was set aside for further work.

A 1:1 diluted solution of hydrochloric acid (~ 16% m/v) was added dropwise to the silver nitrate filtrate to precipitate silver chloride as a curdy, white preciptate. Addition was stopped when no more new precipitate was formed. The solution was allowed to stand for 24 h and then filtered. The residue was combined with others collected from silver waste solutions originating from the analytical chemistry undergraduate training laboratory.

The combined silver chloride residue was dried at 110°C for an hour in a laboratory oven before being mixed with potassium carbonate (K_2CO_3) in a ratio of 4:3[18]. The K_2CO_3 was added as a flux to facilitate melting and prevent oxygen absorption by the silver, which would make it brittle and unusable in jewellery manufacturing. The mixture was placed in a clay crucible and roasted in a furnace at 1000°C. One has to be very careful during the melting step of silver because toxic $COCl_2$ (phosgene gas) can be formed and therefore it is advisable to ensure that the furnace is hooded and proper extraction of the fumes occurs. After melting, the crucible was removed from the furnace, the slag discarded and the molten silver poured into another clay crucible where it was allowed to solidify and cool down. The purity of the final material was assessed by ICP analysis.

During the initial dissolution stage of the procedure, the effects of temperature, stirring speed, solid:liquid ratio and acid concentration on the amount of silver leached from the waste, were investigated.

Gold recovery

The dried residue from the silver-leaching step was weighed into a beaker and dissolved in 102 ml of aqua regia. The solution was filtered and washed 5 times with 20 ml portions of distilled water and the residue set aside. The filtrate was boiled on a hot plate until it had a syrupy consistency. A few drops of concentrated hydrochloric acid (~ 32% m/v) were added to it, and the solution diluted with 50 ml of distilled water before being boiled again to a syrupy state. This process of evaporation, dilution and re-evaporation with the addition of HCl was found to be a crucial step in the recovery process and was repeated at least 3 times. When all the excess nitric acid was removed, the syrup-like solution was cooled down before being diluted to four times its original bulk volume with hot water (final volume should be approxi-

Table I

Wavelengths used for the analysis of various chemical elements by ICP[16]

Element	Wavelength (nm)
Gold	208.209/267.595
Platinum	177.709/191.170
Silver	328.100

Small-scale recovery of noble metals from jewellery wastes

mately 800–1000 ml). The solution was covered with a watch glass and left to stand for 48 h. A separate solution was prepared by mixing 120 g of iron(II) sulphate ($FeSO_4.7H_2O$) in 300 ml of water with 40 ml of concentrated HCl (~ 32% m/v) acid. This was slowly added into 400 ml of the gold-containing solution while stirring. The gold that precipitated was filtered off, washed with hot water and dried at 110°C for 1 h. The filtrate was put aside for Pt recovery. The dried brown gold powder was mixed with an equal mass of lithium tetraborate flux and placed in a clay crucible. This was melted at 1100°C in a muffle furnace. After melting, the flux was separated from the gold. The molten gold was poured into a second clay crucible and allowed to solidify. The purity of the final material was assessed by ICP analysis.

During the recovery stage of the gold, the effects of temperature and stirring speed were investigated.

Platinum recovery

The filtrate, set aside after the gold filtration step, was treated

Table II

Chemical analysis (%m/m) of the jewellery waste

Composition	Content (%)
Si	1.43
Ti	0.63
Al	0.50
Fe	0.35
Mn	0.16
Mg	0.17
Ca	1.20
Na	1.14
K	0.50
P	0.34
S	2.13
Cl	1.20
Cr	590 ppm
Ni	590 ppm
Cu	13.90
Zn	1.09
Ag	63.50
Pt	2.46
Au	9.11

Table III

Screen size analysis of jewellery waste

Sieve size range (m)	Weight % retained	Size distribution (%)
+212	14.59	85.41
-212 +150	18.23	67.18
-150 +106	18.35	48.83
-106 +75	26.52	22.31
-75	22.31	0
Total	100	

with 24 mg/l of ammonium chloride solution, while stirring at a temperature of 80–90°C[5,9,19]. This operation should be performed in a fume hood as corrosive NOCl can be formed during the process. The yellow-orange precipitate of ammonium hexachloroplatinate ((NH_4)$_2$PtCl_6) that formed, was filtered off and dried at 105°C for 2 h. Then it was dissolved in water at 80°C and reduced with a 98% solution of hydrazine to yield a black Pt powder. The powder was filtered from the solution, dried at 105°C for 2 h and melted with a blowtorch using an oxidizing flame in a silica-alumina crucible to produce a shiny ingot. The purity of the final material was assessed by ICP analysis.

During the recovery stage of the platinum, the effects of temperature and stirring speed were investigated.

Results and discussion

The chemical composition of the cleaned jewellery waste is given in Table II. The results indicate a gold content of 9.11%, with 63.50% of silver and 2.46% of platinum.

The results obtained with the screen analysis are given in Table III. It was found that 85.4% of the sample had a particle size smaller than 212 μm. The large number of particles with a small size is a favourable feature for precious metal leaching from it[1,20].

During the recovery process of silver from waste, it is firstly solubilized to silver nitrate according to the following chemical reaction:
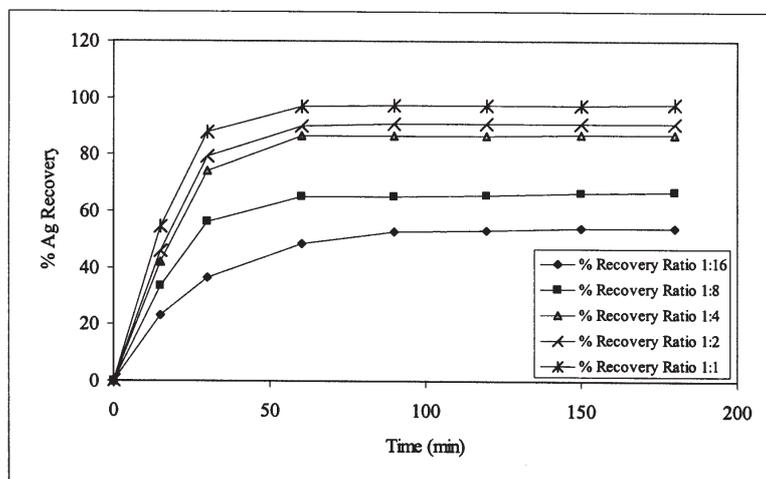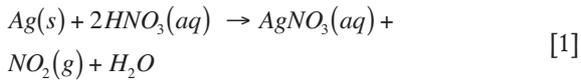

Figure 1—Effect of various acid concentrations on the dissolution of silver from jewellery waste (stirring speed 200 rpm; temperature 40°C; solid:liquid ratio 1:5)

Small-scale recovery of noble metals from jewellery wastes

$$Ag(s) + 2HNO_3(aq) \rightarrow AgNO_3(aq) +$$
$$NO_2(g) + H_2O \qquad [1]$$

In the next step the silver nitrate is converted to silver chloride by the addition of hydro-chloric acid to the filtrate:

$$AgNO_3(aq) + HCl(aq) \rightarrow AgCl(s) + HNO_3(aq) \qquad [2]$$
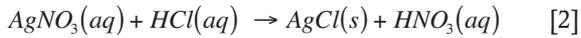
The final recovery of metallic silver in the refining step can be represented by the following chemical reaction:

$$2AgCl(s) + K_2CO_3(s) \rightarrow$$
$$2Ag(s) + 2KCl(s) + O_2(g) + CO(g) \qquad [3]$$
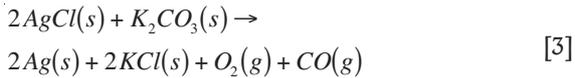
The residue after the silver-leaching step comprised 17 g, which means that 34% of the cleaned waste sample consists of Au and Pt, with the silver part making up the remaining 66%. According to literature[19], the optimum composition for separation is at a Ag:Au ratio of 75:25. During leaching of silver from the jewellery waste, the effects of a number of variables were investigated. The acid concentration can be expected to have a major influence on both the kinetics of the leaching, as well as the efficiency of the process. The effect of various acid concentrations on the dissolution of silver from the waste is depicted in Figure 1. It can be seen from the different curves that the amount of silver leached from the waste increases as the acid concentration increases, and that a leachant with a 1:1 ratio of HNO_3 (~ 16% m/v) and water gives the most complete recovery of silver in a convenient time frame at a temperature of 40°C. A total recovery of 97.1% was achieved after 1 h.

The effect of stirring speed on the leaching process is illustrated in Figure 2, and can be seen to increase drastically in efficiency from static conditions to cases where stirring has been applied. Provided that a minimum stirring speed is maintained, further increases in the stirring rate do not cause a higher degree of silver leaching from the waste. This is in agreement with results from other workers reported in the literature that agitation leaching is usually the most economic choice for high-grade ores or ores requiring fine grinding to liberate the desired metal(s)[21].
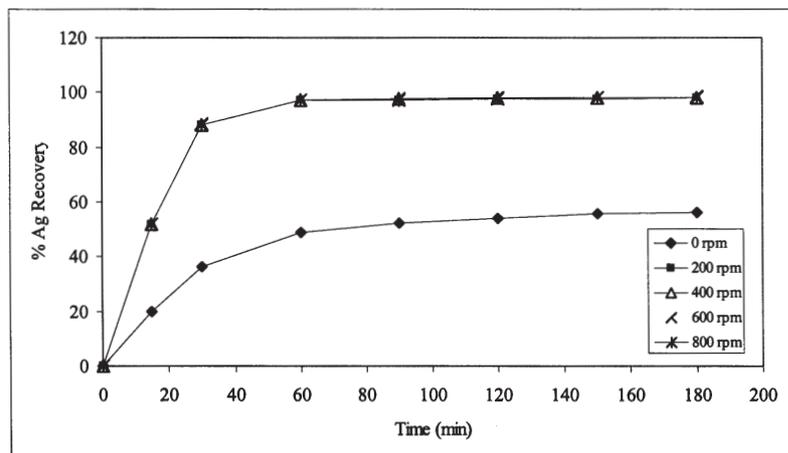

Figure 2—Effect of stirring speed on the dissolution of silver from jewellery waste (acid concentration 1:5 HNO_3 to water; temperature 40°C; solid/liquid ratio 1:5)
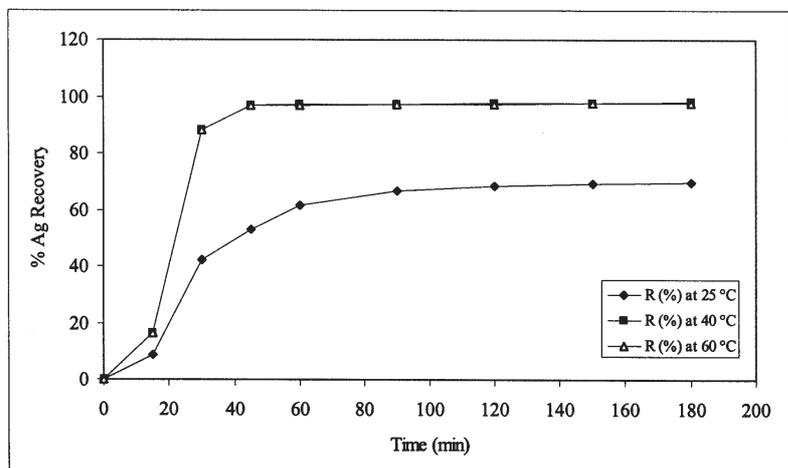

Figure 3—Effect of temperature on the dissolution of silver from jewellery waste (acid concentration 1:5 HNO_3 to water; stirring speed 200 rpm; solid:liquid ratio 1:5)

Small-scale recovery of noble metals from jewellery wastes

When increasing the leaching temperature up to 60°C, a significant increase in the percentage silver dissolved from the jewellery waste can be achieved in a given time. It was found that only 61.7% of the silver was extracted at room temperature (25°C), which increased to 97.1% at elevated temperatures (≥ 40°C). This is schematically shown by the curves in Figure 3.

The fact that a stirring speed above 200 rpm did not increase the recovery of the silver, justifies the conclusion that sufficient leachant is delivered to the surface of the waste particle undergoing dissolution and that the rest of the process is probably mainly diffusion controlled. The observation that the recovery increases with an increase in temperature from room temperature to 40°C, confirms this deduction and is in agreement with the work and opinions of other researchers reported in literature[21–24]. This combined dependency of the rate and amount of silver extracted from the waste on stirring speed and temperature, seem to indicate that the mechanism of dissolution is a combination of mass transport and diffusion control.

In plant operations the solid:liquid ratio can be an important factor that has a bearing on the amount of a desired metal that can be recovered from its ore. In technical terms it translates to the fact that the leachant should not be the limiting reagent in the process or reaction. The effect of various solid:liquid ratios on the amount of silver extracted from the jewellery waste is illustrated by the curves in Figure 4. It can be seen that once a solid:liquid ratio of 1:5 is exceeded, no further gain in either the leaching efficiency or rate was observed.

Gold and platinum are both dissolved in aqua regia. In the case of gold, there is a series of reactions involved, but the overall reaction can be summarized as[25]:

$$Au(s) + 3/2\ Cl_2(g) + Cl^-(aq) \rightarrow AuCl_4^-(aq) \quad [4]$$
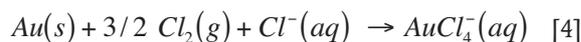
The precipitation of the dissolved gold from solution with iron sulphate can be described by the reaction:

$$3FeSO_4(aq) + AuCl_4^-(aq) \rightarrow$$
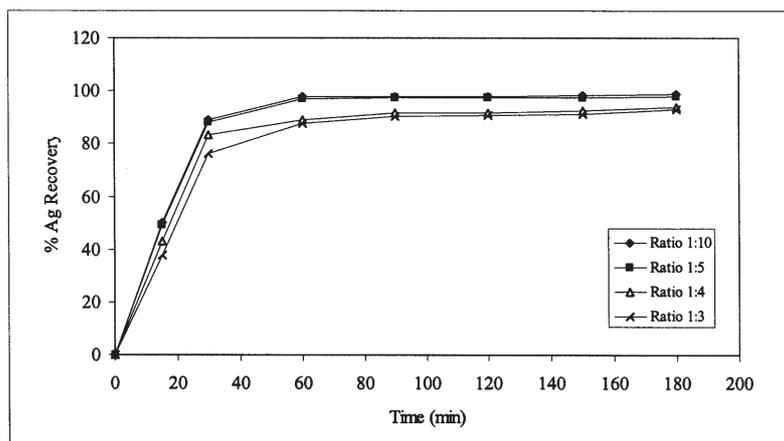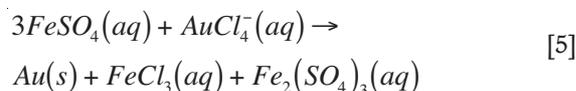$$Au(s) + FeCl_3(aq) + Fe_2(SO_4)_3(aq) \quad [5]$$

Figure 4—Effect of solid:liquid ratio on the dissolution of silver from jewellery waste (acid concentration 1:5 HNO₃ to water; stirring speed 200 rpm; temperature 40°C)
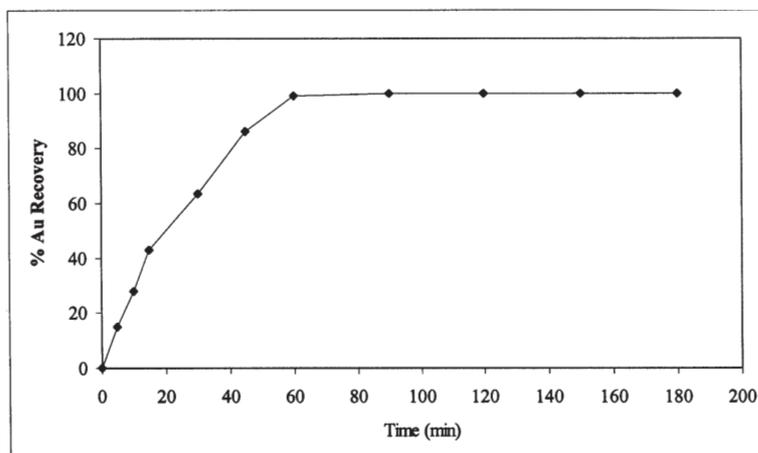
Figure 5—Gold recovery from a pregnant aqua regia solution as a function of time (stirring speed 200 rpm; temperature 40°C)

# Small-scale recovery of noble metals from jewellery wastes

The kinetics of gold precipitation from solution by the iron sulphate reagent is illustrated in Figure 5. Analysis of the barren solution after filtering off the precipitate indicated a total recovery of 99.9% of the gold.

The effect of temperature on the rate of gold precipitation from the pregnant solution is represented by the curves in Figure 6. Once again one observes an increase in the amount precipitated and an acceleration of the reaction with an increase in temperature up to 40°C. This is in agreement with what has been found in the case of silver.

Figure 7 graphically shows the effect of stirring rate on the kinetics of gold precipitation from the mother liquor. There is a huge difference in the amount of gold precipitating from solution when it is static, compared to when it is stirred, with a total removal of 59.5% in the case of the former condition as compared to 99.9% in the latter. An increase in the stirring speed beyond 200 rpm results in only a marginal increase in the rate of gold precipitation up to an hour. Thereafter, no further differences in either the rate or the amount of gold recovered is observed.

Platinum dissolution when the jewellery waste is treated by aqua regia can be represented by the following chemical reaction:

$$8HCl(aq) + 2HNO_3(aq) + Pt(s) \rightarrow$$
$$H_2PtCl_6(aq) + 4H_2O(aq) + 2NOCl(g) \tag{6}$$
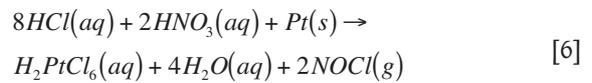
When this solution comes into contact with ammonium chloride, the following reaction takes place:

$$H_2PtCl_6(aq) + 2NH_4Cl(aq) \rightarrow$$
$$(NH_4)_2PtCl_6(aq) + 2HCl(aq) \tag{7}$$
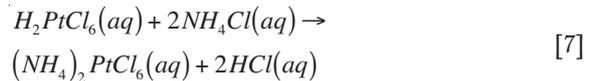
The use of hydrazine reduces the ammonium hexachloroplatinate complex to metallic platinum according to the reaction

$$(NH_4)_2PtCl_6(aq) + N_2H_4(1) + 6NaOH(aq) \rightarrow$$
$$Pt(s) + 6NaCl(aq) + 2NH_3(aq) + N_2(g) + 6H_2O(aq) \tag{8}$$
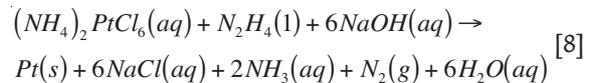
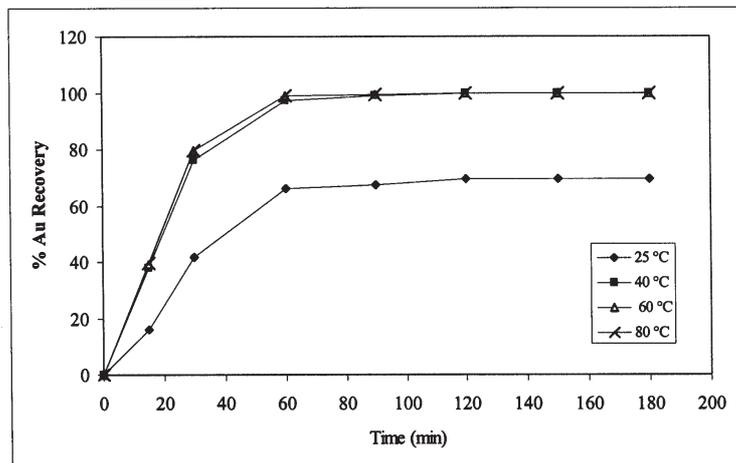Just as in the case of the gold, the precipitation of

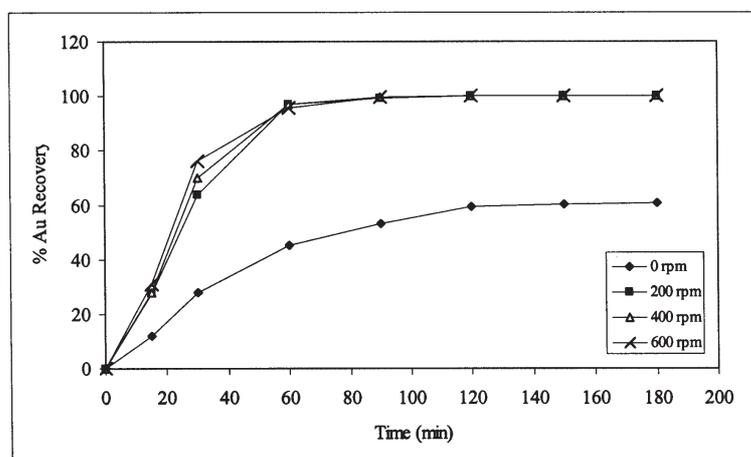**Figure 6—Gold recovery from a pregnant aqua regia solution as a function of temperature (stirring speed 200 rpm)**

Figure 7—Gold recovery from a pregnant aqua regia solution as a function of stirring speed (temperature 40°C)
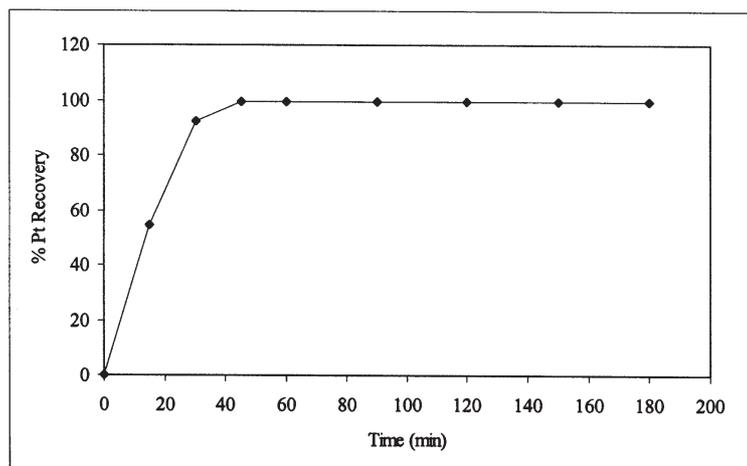
Figure 8—Platinum recovery from a pregnant aqua regia solution as a function of time (stirring speed 200 rpm; temperature 80°C)
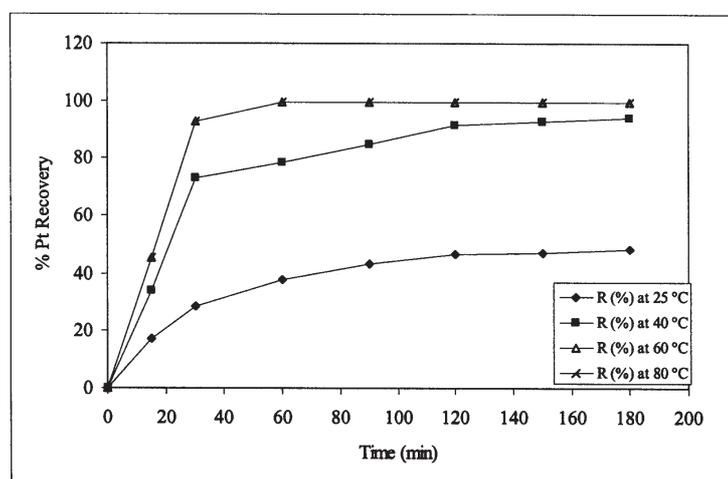

Figure 9—Platinum recovery from a pregnant aqua regia solution as a function of temperature (stirring speed 200 rpm)

platinum from solution was found to be completed within 45 minutes and yielded a recovery of 99.4%. According to Harris and Stanley[26], selective recovery of platinum from chloride liquors originating from the treatment of various primary and secondary materials, constitute a complex and challenging problem in precious metals refining. This work has proven that with the correct choice of a suitable reducing reagent, a very good recovery can be achieved within an hour. The results of this investigation are shown graphically in Figure 8.

The effect of temperature on the precipitation of platinum

from solution is illustrated by the curves shown in Figure 9. Contrary to the case for gold, more pronounced differences were observed for Pt regarding the precipitation rate and amount recovered from solution with variations in the temperature of the solution. It seems that one requires at least a temperature of 80°C to obtain a reasonably fast and good recovery of Pt from the mother liquor. The effect of stirring speed on the precipitation of Pt from solution follows a similar pattern to that of gold, as can be seen from the results depicted in Figure 10.

The final purities and yields that can be obtained for each noble metal are summarized in Table IV. The extraction of silver, gold and platinum from the jewellery waste can be summarized by the flowchart in Figure 11.

Cost analysis

One of the goals of this investigation was also to find an economical way of treating the jewellery wastes. As a first attempt, an estimate was made of the running and operating costs of the proposed treatment process. The assumptions made to assist with these calculations were:

Table IV

Recovery yields and purity of the noble metals obtained from the treatment of jewellery waste

Noble metal	Recovery yield (%)	Purity (%)
Silver	97.1	98.3
Gold	99.9	99.1
Platinum	99.4	99.0

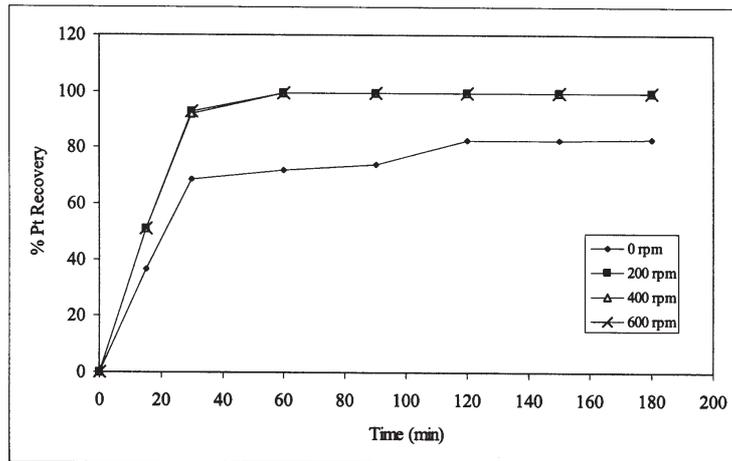# Small-scale recovery of noble metals from jewellery wastes

Figure 10—Platinum recovery from a pregnant aqua regia solution as a function of stirring speed (temperature 80°C)
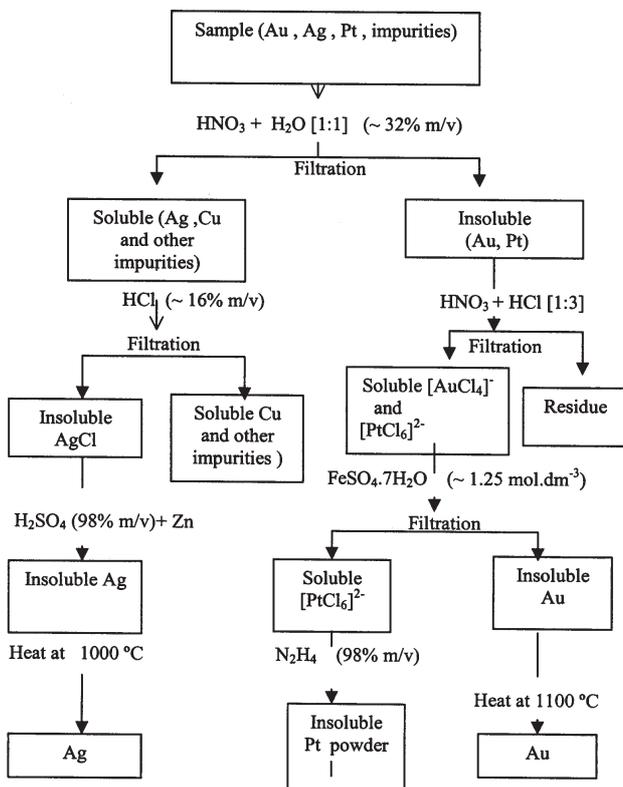
Flow Sheet of the process

Figure 11—Flowsheet of the separation process to recover noble metals from jewellery waste

Table V

Exchange rates and commodity prices used in calculations[29]

Exchange currency	Exchange rate	Commodity	Price (US$/oz)
Rand / dollar	6.97	Gold	383.1
Rand / pound	11.64	Platinum	756
Rand / euro	8.01	Silver	5.05

Oz = Troy ounce = 31.1 grams.

Table VI

Cost analysis of the proposed process

Income	R12 089.65
Cost of stirring	R159.20
Cost of refining	R690.00
Cost of reagents	R825.00
Profit (rand / kg)	R10 415.45

➤ A unit mass of 1 kg of waste requiring treatment was considered.

➤ A stirring rate of 200 rpm was employed.

➤ The maximum treatment time would be 1 h.

➤ The cost of energy consumption was based on an electrical unit cost of R0.85/kWh[27].

➤ Chemical prices of compounds required were supplied by Aldrich[28].

➤ Exchange rates and commodity prices as quoted in the press at the time and summarized in Table V were used[29].

➤ A silver content of 66%, and gold and platinum levels of 17% each were present in the waste.

➤ A final purity of 98.3% for Ag, 99.1% for Au and 99.0% for Pt, respectively.

An elementary and simple cost estimation summarized in Table VI gives an indication of the economic benefit of the proposed treatment process. The indications are that a worthwhile amount of money can be saved by employing the proposed process in-house, because commercial refining companies normally charge a substantial percentage of the refined metals' value to do this work. Furthermore, it has the advantage of being reasonably time efficient too, as it can be accomplished within a week at the most, even if a mass of one kilogram of the waste needs to be processed.

Conclusions

This investigation showed that it was possible to separate, selectively recover and refine silver, gold and platinum from jewellery and laboratory wastes, and produce technical grade

Small-scale recovery of noble metals from jewellery wastes

material suitable for training purposes. A simple procedure was used, in which nitric acid was employed to dissolve and separate silver and other impurities from gold and platinum, which were then dissolved in aqua regia and precipitated selectively. It should be stressed that the proposed process in its current form will be suitable for small-scale laboratory refining only, because it involves a number of evaporation, dilution and re-evaporation steps. The current work quantified the effects of temperature, stirring rate, leaching agent concentration and solid:liquid ratio on the recovery of the various noble metals and in summary yielded the optimum treatment conditions in each case. The proposed treatment procedure does not use toxic cyanide as is the case in conventional primary recovery of gold from ores, but some corrosive and toxic gases are nevertheless released during the procedure, which necessitates the use of fume hoods/cupboards to perform the procedure safely. A definite advantage is that all the reagents are easily obtainable and it has potential time saving and financial benefits. However, the final refining of the recovered metals needs to be investigated further, as well as noble metal losses to effluent streams and possible deportment to silver, none of which has been considered in the current investigation.

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