

Effluent treatment: what role can modified waste/biomass play in the local platinum industry— a review

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The waste streams generated in the mining and metallurgical industry contain significant levels of heavy metals. In the platinum industry, the amount of platinum group metals (PGMs) contained in these waste waters could have a large economic benefit should the recovery be of high efficiency and cost-effectiveness. These waste waters contain a number of elements and it is essential that the removal of the PGMs be selective. Currently, the methods of removal that are employed for the treatment of waste waters are, among others, solvent extraction and ion exchange. Although these have been effective to some extent, incomplete removal, high reagent consumption and further production of waste are disadvantages. The need for a more effective and economical alternative to treat waste waters has led to research into biosorption—a method that uses the binding capacities of biological material for the adsorption of metals. Its major advantage is that it is usually very low cost, decreases metal concentrations considerably, and is especially useful in waste waters containing low concentrations of metal ions. The present review examines the possible application of biosorption techniques in the treatment of PGM waste water with particular reference to chemically modified waste paper. This particular adsorbent has been found to have high adsorption capacity and impressive selectivity for the metals of interest. For the platinum industry, such a method is envisaged to provide not only a simple effective metals' removal technique from solution but also high post metal recovery during elution. In this respect the process could prove to be an alternative to any current processes in use in alleviating environmental as well as economic concerns.

Keywords: Platinum group metals, biological material, biosorption, chemically modified, waste paper, ion-exchange, waste waters.

Introduction

During the refining of metals, the large volumes of aqueous by-products which are produced can contain substantial amounts of high-value metals. These precious metals are discarded, while the discharge of coexisting toxic metals leads to the contamination of the water systems¹. At present, precious metals such as gold, palladium and platinum are used extensively, not only in jewellery but also in a variety of well known advanced applications such as electrical and electronic devices, catalysts, and medical instruments. Because they exist in limited amounts in crustal rocks, there is a growing need that they be effectively and efficiently recovered from various wastes. The recovery process should exhibit high selectivity for the precious metals over the often coexisting base metals such as iron, zinc and copper, but this is usually limited due to economic constraints^{2,3}.

Industrial recycling techniques for the recovery of precious metals from wastes are currently hydrometallurgical or pyrometallurgical. Of the techniques employed, adsorption is the most cost-efficient and effective way of recycling precious metal ions^{4,5,6}. To date, a number of adsorption materials have been developed such as activated carbon and ion exchange resins. Ion-exchange resins made from petrochemical-dependent polymers and designed for varied complexes of metal ions are widely

employed in industry. The emission of carbon dioxide is a major global concern and as a result much attention has been focused on petrochemical independent products as an alternative^{5,6}.

In general, conventional methods applied for the removal of low concentrations of dissolved metal ions from waste waters have disadvantages. These include incomplete metal recovery, high capital costs, high reagent requirements and generation of toxic sludge or other waste products that require disposal⁴⁻¹⁴.

These drawbacks have given rise to the need for more economical and effective methods for the recovery of metals from waste waters. The search for new technologies involving the removal of precious metals from waste waters has now moved towards biosorption, a method that is based on the metal binding capacities of various modified and unmodified biological materials^{8,16-17}.

Biosorption

The biosorption process is a passive physical-chemical interaction between the charged surface groups of biological material and ions in solution, in which biological material can be used. The advantages of this process when compared to the conventional methods include low operating costs, minimization of the volume of chemical and/or biological sludge to be handled and high efficiency

in detoxifying effluents. They are also environmentally friendly and biodegradable¹⁷. This physical-chemical phenomenon of metal biosorption is relatively rapid and can be reversible. In the presence of such a mechanism, which fortunately is the most common, biomass has similar chemical characteristics to an ion exchange resin or activated carbon, implying many advantages in the industrial application of biosorption³.

The immobilization by chemical modification of the biomass in solid structures creates a material with the right size, mechanical strength, rigidity and porosity necessary for use in columns. Immobilization can also yield beads or granules that can be stripped of metals, reactivated and re-used in a manner similar to ion exchange resins and activated carbon. Sorption processes using low cost and widely available biomass materials (living or dead) represent an interesting alternative as they have shown great promise for the treatment of pollutants in industrial effluents. From an environmental perspective, the use of biomass materials is more favourable because it is easier to recover the loaded metal ions either by elution or incineration, yielding concentrated solutions of the metal of interest. It is desirable that the least possible damage occurs to the adsorption properties of the biomass so as to allow the reuse of the biomass in subsequent adsorption-desorption cycles.

In biosorption based processes, the stationary phase is the biological material where the ions from solution are adsorbed onto the material by ion-exchange and other mechanisms. How effective this stationary phase is, depends on the following properties:

- Adsorption properties: a high affinity for the targeted metal is essential. Ideally, the adsorptive properties should be selective. The greater the selectivity, the greater the extent to which the metal values can be recovered.
- Desorption properties: no matter how good the adsorptive properties of the stationary phase are, it will not be effective for metal recovery if the metal values cannot be stripped from the material. The desorption chemistry can be similar to the adsorption chemistry, but this is not always the case.
- Separability: for the refining/concentration operation to be feasible, it is important that an efficient and inexpensive means is available for the separation of the stationary phase from the aqueous solution.

- Reusability: After the stripping operation, the stationary phase must be restored to its original condition so that it can be reused and recycled in a subsequent loading–stripping operation with minimal losses of adsorptive properties.
- Durability: due to the intended extensive recycling, it is important that its physical and chemical properties do not deteriorate excessively from cycle to cycle. This is very important in processes where metals recovery is carried out directly from leach pulps without prior solid-liquid separation.

This present review examines chemically modified waste papers that have been extensively studied for their high adsorption capacities. To determine whether a biosorption based process could be an alternative to the currently employed technology, the performance of chemically modified waste paper is investigated by looking into the following: (i) analysis of chemically modified waste paper; (ii) precious and base metals adsorption from varying concentration of hydrochloric (HCl) acid solutions; (iii) adsorption kinetics; (iv) adsorption capacities; (v) adsorption and elution cycles; (vi) adsorption from hard waters; (vii) precious and base metals adsorption from real industrial solution; and (viii) application opportunities in the South African precious metals industry.

Analysis of chemically modified waste paper

In the preparation of the adsorbents, certain selected constituents are extracted from the waste paper containing cellulose, then those constituents are sometimes modified (immobilized) by an amination reaction to enhance the efficiency of adsorption. Examples of these adsorbents include primary amine-immobilized waste paper gel (WP-NH₂), ethylenediamine-immobilized waste paper gel (WP-en), dimethylamine paper gel (DMA-paper gel) and p-aminobenzoic acid paper gel (PAB-paper gel). The preparation of these gels is illustrated in Figures 1 and 2^{1,18-19}.

The main components of paper produced from wood are cellulose and hemicellulose and the portion of amorphous (unstructured) and crystalline domain of cellulose depends on the kind or the nature of the plants used. Wood cellulose has the highest portion of amorphous domain among a variety of plant cellulose; consequently, paper produced from wood contains a high proportion of amorphous

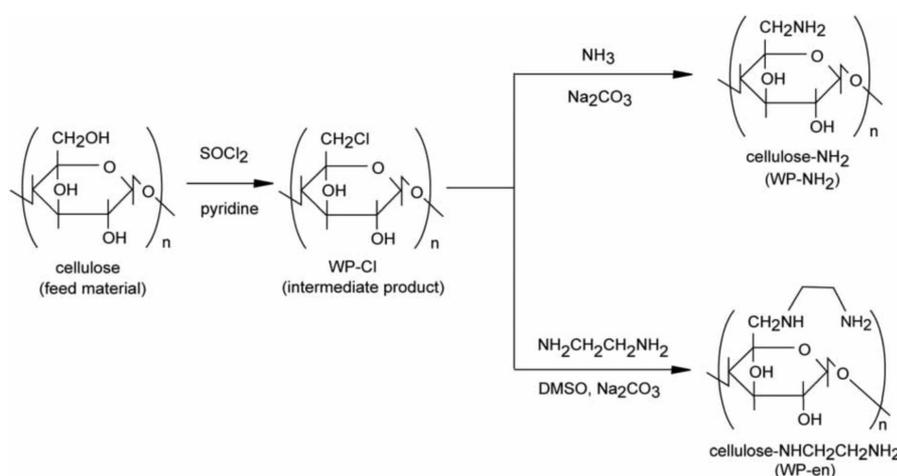


Figure 1. Preparation of WP-NH₂ and WP-en paper gels¹⁸

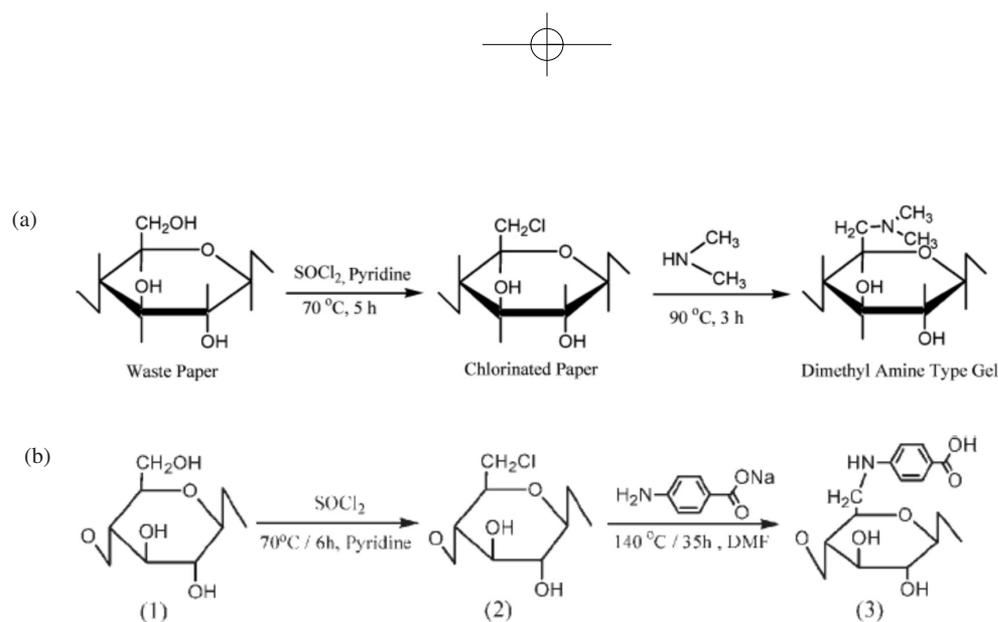


Figure 2. Preparation of (a) DMA-paper gel¹⁹ and (b) PAB-paper gel¹

domain. During the recycling process, waste paper is chemically or mechanically cleaved to a certain degree; hence, the ratio of the crystalline to the amorphous domain originally contained in the cellulose, is lowered. Because of the high portion of the amorphous domain in waste paper, the reagents for chemically modifying functional groups for metal adsorption can easily diffuse into the matrices of waste paper¹⁸.

Also, the fibres left over from the process of paper production, which contain over 70% of pure cellulose, are usually discarded in amounts of several thousands tonnes a year, thus presenting a significant opportunity for use of this material in the production and use of chemically modified biomass. Consequently, from the perspectives of both environment and economy, efforts are being made to reclaim value from this cheap biomass in the form of energy and advanced functional materials by the effective use of its unique characteristics¹.

The fundamental process of modification of waste paper materials aims at generating metal ions specific adsorption properties. This is linked to the nature of the expected the ions in solution. For example, precious metals will mostly exist as anionic complexes form in chloride systems as compared to the associated base metal ions which usually exist as cationic complexes. In this respect, anion exchange type of adsorbents can be developed. The work of Parajuli *et al.*²⁰, focuses on the chemical modification of the cross-linked lignophenol gel (CLP) so as to incorporate the recovery of palladium (Pd) and platinum (Pt) in addition to gold (Au) from chloride based systems.

Precious and base metals adsorption from HCl solutions

Kawakita *et al.*¹⁸, attempted to use waste newspaper for the adsorption of metal ions by immobilizing functional groups with high affinity for specified metal ions. Primary amine and ethylenediamine groups exhibited very high selectivity towards precious metals such as gold, platinum, and palladium over base metals when adsorbed from hydrochloric acid solution. This selectivity proved to be better than that of available ion-exchange resins. For example, the dimethyl amine, polyethylene poly amine and iminodiacetic acid showed poor adsorption when compared to the modified waste newspaper gel.

The number of primary amine groups contained in the primary amine-immobilized waste paper gel (WP-NH₂) and the number of ethylenediamine groups contained in the

ethylenediamine-immobilized waste paper gel (WP-en) were calculated as 4.1 and 3.4 mol/kg-dry gel, respectively. The maximum adsorption capacities for palladium and platinum were found to be greater than 1 mol/kg-dry gel. The effect of pH and HCl concentration on the metal adsorption was also examined to explain the adsorption mechanism.

The precious metals, Au(III), Pt(IV), and Pd(II) were quantitatively adsorbed for both adsorbents (WP-NH₂ and WP-en) in the range from 0.25 to 6M HCl, whereas no base metals except for Cu(II) and Fe(III) were practically adsorbed. With increasing HCl concentration, the adsorption of Cu(II) decreased for both WP-NH₂ and WP-en. At HCl concentrations greater than 0.1 M, platinum and palladium were considered to be adsorbed on the ammonium form of the gels as the chloro-complexes, [PdCl₄]²⁻ and [PtCl₆]²⁻, respectively. The experimental data presented in the preceding discussion essentially demonstrates the feasibility of separating and recovering of these precious metals from chloride solutions using immobilized adsorbents.

On the other hand, Adhikari *et al.*^{1,19} also carried out studies on waste newsprint paper. In their first study, they modified the paper with dimethylamine (DMA) and p-aminobenzoic acid (PAB) to obtain amine type adsorption gels called DMA paper gel and PAB-paper gel, respectively (Figure 2). The gels exhibited selectivity only for precious metals with a remarkably high capacity for Au(III) for Pd(II) and Pt(IV) (Figure 3 (b) and (c)). Also, Au(III) was reduced to the elemental form during adsorption, forming aggregates.

The adsorption kinetics of Au(III), Pt(IV), and Pd(II) on the DMA-paper gel was studied at 30 °C with the equilibrium times reached within five hours (5 h) for Pd(II) and Pt(IV) (Figure 3(d)). The phenomenon involving gold was different in the sense that equilibrium was reached at times in excess of five hours, the equilibrium time defined for platinum and palladium. In this work, Parajuli *et al.*², explain the adsorption mechanism of gold to involve the formation of Au(I) ions in the system. This particular species undergoes elution due to the non-selectivity nature of the gel towards it. It is in this context that whereas palladium and platinum reach equilibrium faster, gold takes much longer to do so.

In contrast, the adsorption kinetics of the PAB-paper gel for Pt(IV) differed considerably (Figure 4) from that of Au(III) and Pd(II). In the case of Pt(IV), equilibrium was reached within 5 h, whereas it took up to 24 h for Au(III)

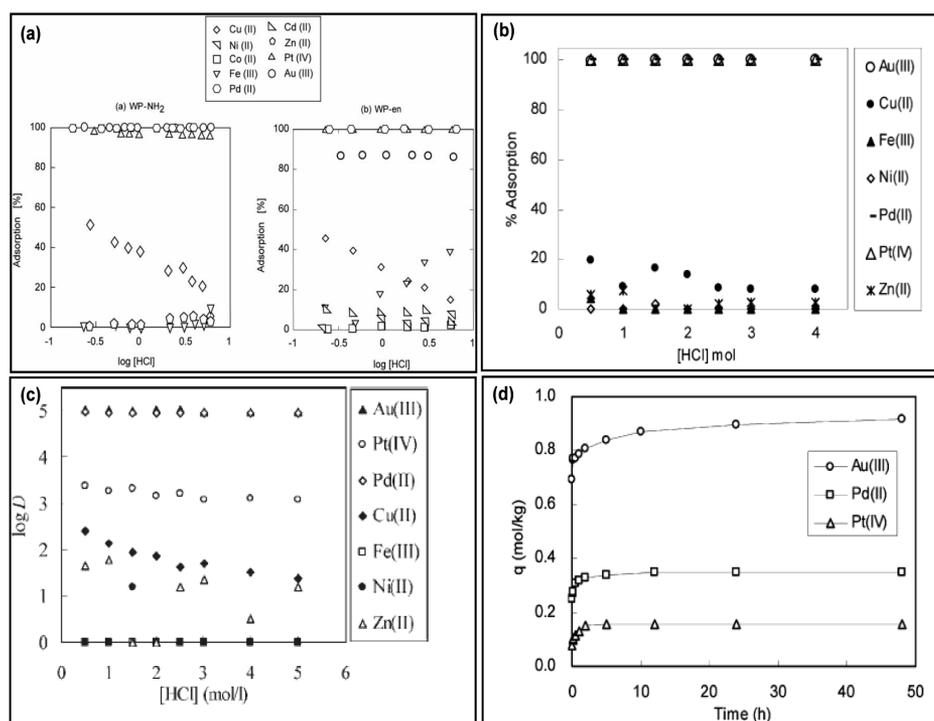


Figure 3. Effect of HCl concentration on % adsorption of metal ions (a) primary amino and ethylenediamine immobilized waste paper gel¹⁸; (b) DMA-paper gel, initial concentration of metal ions = 0.2 mM, wt. of gel = 20 mg, shaking time = 24 h, temperature = 30°C¹⁹; (c) PAB-paper gel [D = (mol Mn⁺/kg of dry gel)/mol Mn⁺/l of solution]¹ and (d) kinetics of adsorption of metal ions on DMA-paper gel. Initial concentration of metal ions = 0.5 mM, [HCl] = 1 M, wt of gel = 20 mg, temperature = 30°C¹⁹

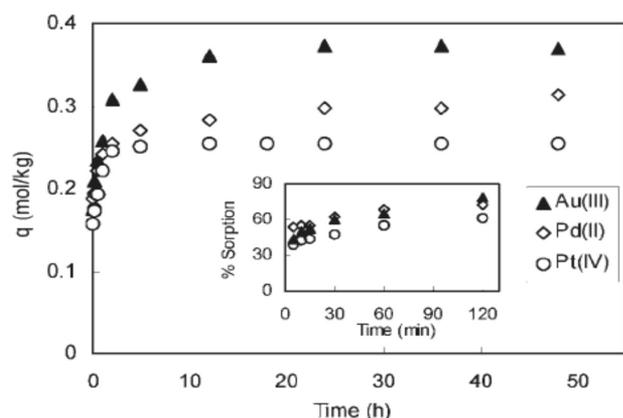


Figure 4: Sorption kinetics of metal ions using the PAB paper gel. Initial concentration of metal ions = 0.5 mM, [HCl] = 1 M, wt. of gel = 20 mg, temperature = 30°C¹⁹

and Pd(II). There is an inherent benefit for the PAB-paper gel system in that almost 80% of the Au (III) and Pd (II) ions present in the solution are adsorbed within 2 h. In this regard, although the total equilibrium time is longer compared to ion exchange resins of dimethyl amine, polyethylene poly amine and iminodiacetic acid, the sorption rate at the beginning is rapid¹ and the same observation was made for Pt (IV), where 60% adsorption was observed within the same period of two hours.

Loading capacities of biomass

The fundamental studies on the loading capacities of various gels and known ion exchange resins follow the use of adsorption isotherms. The Langmuir Equation [1] allows

for the determination of maximum loading capacity using experimental data.

$$q_e \frac{1}{q_e} = \frac{q_{max} K_L C_e}{1 + K_L C_e} \quad [1]$$

where q_e is the equilibrium amount of metal loaded (mol/kg) on the gel, C_e is the concentration of metal remained in the solution (mol/L) after adsorption, q_{max} is the maximum loading capacity of gel (mol/kg) and K_L is the Langmuir constant related to the energy of adsorption (L/mol). The work of Adhikari *et al.*¹ showed that adsorption of Au(III), Pd(II), and Pt(IV) increased with increasing metal concentration and approached constant values suggesting typical Langmuir type adsorption.

For both WP-NH₂ and WP-en, all plots cluster on straight lines. The intercepts (q_{max}) of these straight lines were found comparable to the loading capacity of the commercially available resin DIAION WA 30, indicating that the gels prepared from waste paper can be used as the adsorbents for precious metals. A similar approach was used in the determination of adsorption capacities for Au(III), Pd(II), and Pt(IV) of the DMA-paper gel and PAB-paper gel. Taking into account the selectivity over other metal ions the reported experimental data represent important application opportunities.

Loaded metals recovery

It is imperative that hydrometallurgical unit processes are not looked at in isolation, because they can affect either down or upstream process efficiencies. The same principle applies to biosorption processes, where loaded metal ions must either be eluted or recovered through other processes that can either be destructive or non-destructive to the

adsorbents. The use of ion exchange resins is often obviated by the problem that while metals ions can be strongly adsorbed, elution of the loaded metals is often very difficult. Consequently, these metals are recovered by incinerating the loaded resins. However, the incineration of plastic resins is not so easy. In many cases, it results in the formation of tar and coke, the post treatment of which is tedious and consumes a large amount of energy for incineration. On the other hand, it is much easier and consumes only a small amount of energy to incinerate the adsorption gel prepared from waste paper and fruit. This is the biggest advantage of the waste paper gel over commercially available ion exchange resins.

The non-destructive process of elution is the most desirable. Adhikari *et al.*^{1,19}, presents some detailed and elegant experimental data on the elution of precious metals from DMA-paper gel. To demonstrate the selectivity of the process they used Cu(II) as an impurity because it is a representative base metal coexisting in practically all waste leach solutions. Their findings are directly reproduced in Figure 5 where it is clear that copper can be selectively separated from the precious metals. The experimental results in Table I further confirm the effectiveness of these paper gels in the recovery of the precious metals provided enough time is allowed.

Modified waste paper gel application opportunities in the recovery of precious metals

The effectiveness of the recovery of precious metals from real industrial and effluent liquors is of paramount importance both from an economic and an environmental perspective. Both Parajuli *et al.*²⁰ and Adhikari *et al.*¹⁹ have demonstrated not only the capability of waste paper gels to recovery precious metals but their stability in harsh oxidizing leach conditions often encountered in aqua regia systems. In this respect, precious metals recovery from electronic waste presents an obvious opportunity because of the abundant availability of this type of waste in the modern age.

Published work^{17,21} clearly documents that electronic scrap can be not only a significant source of precious metals but is often an abundant source of copper. In this respect, either an extensive and laborious selective leach process needs to be followed. Alternatively, complete dissolution followed by selective recovery processes is required. There are definitely pros and cons to each process route. Total dissolution followed by selective metals recovery can only be attractive if there exists adsorbents or solvent extraction reagents that have the desired properties.

Table I
PAB-paper gel performance in consecutive adsorption—elution cycles¹⁹

(a) Recovery of Au(III) in the presence of an excess of Cu(II) ^a						
Cycle	1	2	3	4	5	
Adsorbed/mg	1.87	1.84	1.95	1.93	1.83	
Eluted/mg	1.73	1.74	1.77	1.75	1.69	
Recovery(%)	92.5	94.5	91	91	93	
(b) Competitive sorption of Pd(II) and Pt(IV) ^b						
Cycle	Pd(II)			Pt(IV)		
	Adsorbed (mg)	Eluted (mg)	Recovery (%)	Adsorbed (mg)	Eluted (mg)	Recovery (%)
1	4.89	4.16	85	3.79	3.45	91
2	2.64	2.27	86	4.44	3.75	84.5
3	3.59	2.98	83	5.33	4.63	87
4	2.74	2.34	85.5	4.51	4.03	89

a Feed concentration of metal ions: Au(III)/Cu(II) = 20/100 mg/l, feed time = 15 h. The sorption of Cu(II) was negligible.

b Feed concentration of metal ions: Pt(IV)/Pd(II) = 20/20 mg/l (cycle 1); Pt(IV)/Pd(II) = 100/20 mg/l (cycles 2–4), feed time = 24 h

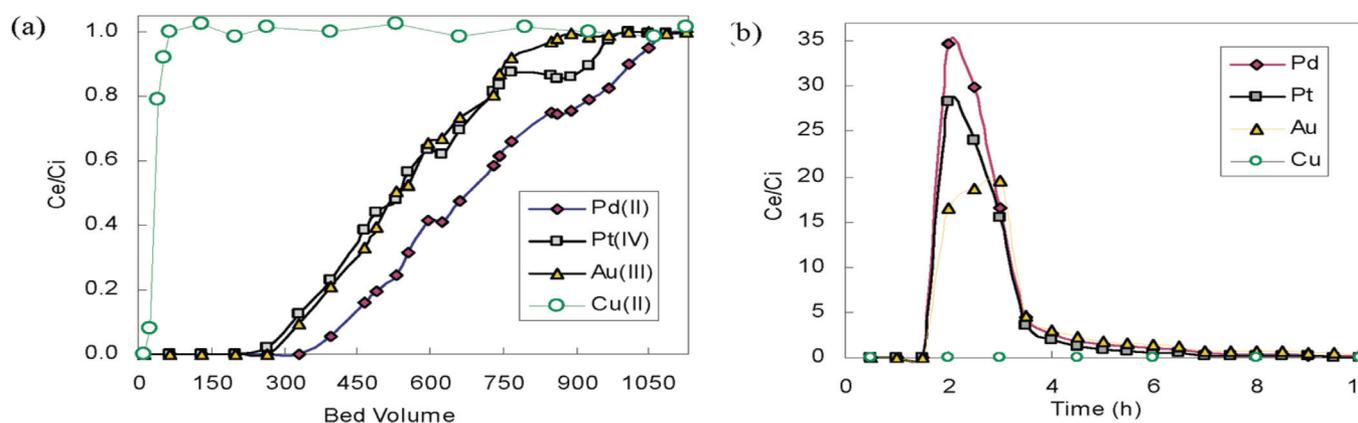


Figure 5. (a) DMA-Paper gel breakthrough profiles for Cu(II), Au(III), Pd(II), and Pt(IV). Feed concentration: Cu(II) 100 ppm, Au(III), Pd(II), Pt(IV) ~ 20 ppm, flow rate 6 mL/h. (b) Elution profiles for loaded Pd(II) and Pt(IV) with 0.1 M thiourea in 1 M HCl. Flow rate 6 mL/h¹⁹

The work of Adhikari *et al.*^{1,19} and that of Kawakita *et al.*¹⁸ clearly indicate the feasibility of separating and recovering precious metals by the use of waste paper gels obtained through chemical modification processes. The competitiveness and selectivity of this technique as compared to conventional processes of ion exchange and solvent extraction technologies, does not only present research opportunities in this area but also the possible development of process flowsheets that can enable precious metal ions recovery from various dilute and 'dirty' leach streams. It is, however, imperative that these gels are characterized not only in terms of their efficiencies but also the physical-chemical properties that define their cost-effectiveness.

The other area of interest in the application of these paper gels is the treatment of waste streams generated from refining operations. Because of the fact that these waste streams are of low concentration (both precious and base metal ions), ion exchange type recovery techniques based on biomass is envisaged to be of great use. For the platinum industry, such a method is envisaged to provide not only a simple and effective metals-removal technique from waste solutions but also high post metal recovery during elution. In this respect the process could prove to be an alternative to current processes and alleviate environmental and economic concerns.

Conclusion

The paper gels, prepared by chemical modification of waste paper, exhibited remarkable capacity and efficiency for the preconcentration and separation of gold, platinum and palladium from other co-existing metal ions at low to high concentration levels. Their major advantages are that they are usually of very low cost, decrease metal concentrations considerably and are especially useful in waste waters containing low concentrations of metal ions. Besides waste water streams, there is considerable potential application of these adsorbents in the recovery of precious metals from electronic waste leach solutions. These leach solutions often contain trace concentrations of precious metals in the presence of a wide variety of base metal ions. In the long term, not only does the process provide research opportunities, it also allows process flowsheet development with a wide application ranging from pure waste streams treatment for clean disposal to purely recovery processes.

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