Extraction of Gold from Au(III) Ion Containing Solution by a Reactive Fiber

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SYNOPSIS

Metallic gold was extracted directly from a solution containing Au(III) ion by a reactive fiber with the amidoxime group. It is concluded that in the solution containing Au(III), Cu(II), Zn(II), and Cr(III) the reactive fiber presents high selectivity to Au(III). During the adsorption process, the adsorbed Au(III) is partially reduced into spongy metallic gold and the amidoxime group is finally oxidized into the carboxyl group. © 1993 John Wiley & Sons, Inc.

INTRODUCTION

The recovery of gold from waste water containing the Au(III) ion has occasioned extensive metallurgical interest. Since chelating resin or fiber presents highly selective adsorption capacity and is convenient to use, more and more attention has been paid to the extraction of gold by chelating resin or fiber.[1-9]

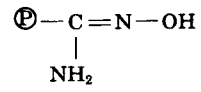
As far as the recovery process is concerned, the adsorption process should be followed by a reduction process because the gold adsorbed by chelating resin or fiber is usually ionic gold. Therefore, it will be more efficient if the Au(III) ion is adsorbed and directly reduced to metallic gold by a reactive fiber at same time.

In the present work, one kind of reactive fiber containing amidoxime groups was used in the recovery of gold. The adsorption behavior, valence of adsorbed gold, and the structural changes of the reactive fiber during the adsorption process were investigated.

EXPERIMENTAL

1. Preparation of Reactive Fiber Containing the Amidoxime Group

Hydroxylamine hydrochloride and sodium carbonate, with a weight ratio of 2 : 1.5, were put into a 250 mL flask, to which 40 mL of deionized water and 1 g of PAN fibers (staples) were added. After being treated at a constant temperature (from 60 to 75°C) for several minutes, the fibers were taken out, washed with deionized water, and then dried.[10] According to elemental analysis and IR, the structure of the functional group in the fiber is confirmed to be as follows:



The amidoxime group content was calculated as follows:

$$C = \frac{(W_2 - W_1)}{W_2 M} \quad (1)$$

where C is the amidoxime group content (mol/g); M, the molecular weight of hydroxylamine (33); and W_1 and W_2, the weights (g) of the fiber before and after reaction, respectively. The functional amidoxime group is referred to as "AO" group in the following discussion.

2. Adsorption Capacity

A few reactive fibers were dried in a vacuum to constant weight, then put into a conical flask and a solution containing Au^{3+} was transferred into the flask with a pipet. The flask was shaken at a steady temperature for several hours. The concentrations

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Journal of Applied Polymer Science, Vol. 49, 1635–1638 (1993)
 CCC 0021-8995/93/091635-04

of ions were detected by an ICAP-9000 inductive coupling plasma emission spectrometer (ICP).

The adsorption capacity was calculated as follows:

$$A = V(C_1 - C_2)/W \quad (2)$$

where A is adsorption capacity (mg/g); W, the weight (g) of reactive fiber; V, the volume (L) of the Au(III) containing solution; and C_1 and C_2, the concentrations (ppm) of ion before and after adsorption, respectively.

3. Structural Analyses

The IR spectrum of the fiber was obtained using a Nicolet 205 FTIR spectrometer with a KBr pellet. The WAXD spectrum was obtained using a Rigaku D/max 3A X-ray diffractrometer using Ni-filtered CuKα radiation. The morphology of the fiber was observed under a Hitachi-450 scanning electron microscope.

RESULTS AND DISCUSSION

1. Adsorption Behavior of the Reactive Fiber

Table I shows the adsorption capacities of the reactive fibers for Au(III). The adsorption capacity increases with increasing AO group content; when the AO group content is 7.6 mmol/g, the adsorption capacity is 626.7 mg/g. In addition, the amount of Au(III) ion adsorbed in per mol of the AO group rises with increase of the AO group content, which means that the adsorption efficiency of the functional group rises with increase of its content in the fiber. Table II shows the adsorption capacities of the reactive fiber to ions in an alloy solution containing Au(III), Cu(II), Zn(II), and Cr(III). It is clear that the reactive fiber presents very high selectivity to the Au(III) ion. Figure 1 shows the relationship between adsorption time and adsorption capacity. The adsorption capacity increases rapidly in the beginning and then increases slowly.

Table I Adsorption Capacity of Au(III) onto Reactive Fiber Containing the AO Group

	AO Group Content (mmol/g)		
	1.9	4.5	7.6
Adsorption capacity (mg/g)	107.5	338.3	626.7
Amount of Au(III) adsorbed per mol of AO group (10^4 mg/mol)	5.77	7.49	8.29

Initial concentration of Au^{3+}: 400 ppm; temperature: 30°C; pH: 2.3; time: 12 h.

Table II Selective Adsorption for the Ions in an Alloy Solution by a Reactive Fiber with AO Group Content of 7.6 mmol/g

	Ion			
	Au(III)	Cu(II)	Cr(III)	Zn(II)
Initial ionic concentration (ppm)	2075	4250	14.5	875
Adsorption capacity (mg/g)	1274	0	14	118

Temperature: 45°C; time: 8 h; pH: 2.0.

2. The Valence of Gold Element Adsorbed by the Reactive Fiber

The SEM photographs of the reactive fibers before and after adsorption are shown in Figure 2(a) and (b). There is much precipitation on the surface of the fiber after adsorption. Figure 3 shows the WAXD spectrum of the fiber after adsorption. Obviously, there are four sharp diffraction peaks in the spec-

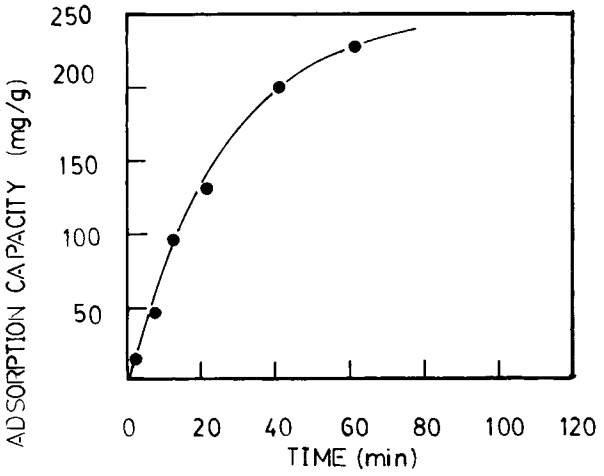

Figure 1 Relationship between adsorption time and adsorption capacity. Temperature. 48°C; pH: 2.8; initial concentration of Au(III): 400 ppm.

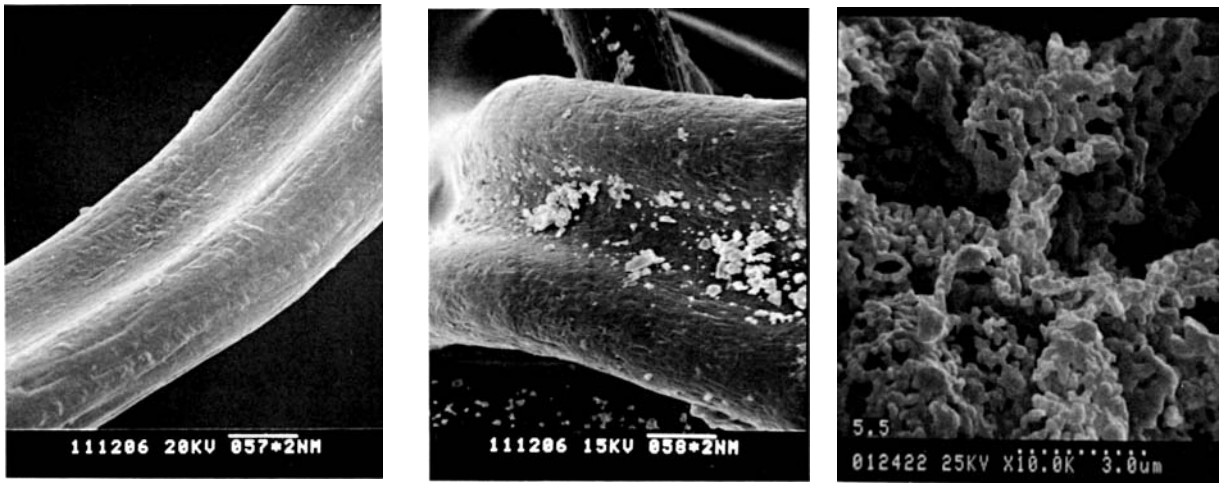

Figure 2 SEM photographs of the reactive fibers: (a) the reactive fiber before adsorption (AO group content is 7.6 mmol/g); (b) the reactive fiber after adsorption (adsorption capacity is 338 mg/g); (c) the remaining gold.

trum. Since the diffraction peak of the crystallite in the reactive fiber is located only at 16°,[10] those peaks may correspond to crystallite in the precipitate. As shown in Table III, the values of the interplanar spacings relating to the diffraction peaks of the precipitate are, respectively, similar to those relating to the planes in the crystallite of metallic gold[11]; therefore, the precipitate is metallic gold.

After the reactive fiber with gold precipitate was heated in an electric oven in the presence of oxygen for several days, until the fiber was entirely burnt out, the golden remainder was washed with 10% HCl aqueous solution and was confirmed to be metallic gold. According to the weight of the remaining gold, and the total amount of Au(III) ion adsorbed in the fiber, the reduction percentage of the reactive fiber to the Au(III) ion can be calculated. For the reactive fiber with an AO group content of 7.6 mmol/g, the reduction percentage is 76%.

Figure 2(c) shows the morphology of the remaining gold. It is clear from the picture that the gold obtained is spongy.

From the above, the reactive fiber with AO groups is able to reduce the adsorbed Au(III) into spongy gold, which has not previously reported. Obviously, such a characteristic of the AO group on the reactive fiber is very advantageous to the recovery of metallic gold directly from waste water containing Au(III) ion.

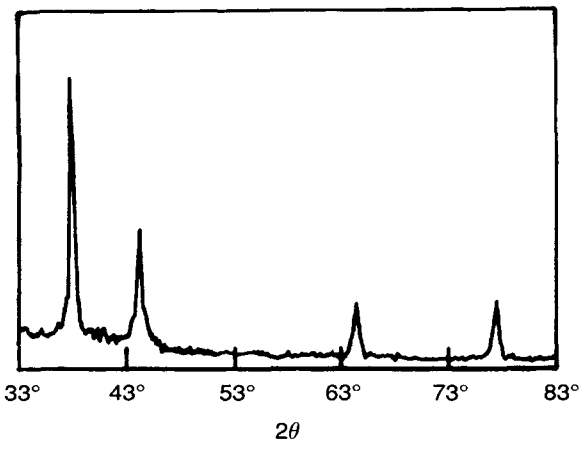

Figure 3 WAXD spectrum of the reactive fiber after adsorption. Adsorption capacity is 338 mg/g.

Table III The Interplanar Spacings Relating to the Diffraction Peaks and to the Crystallite of Metallic Gold

Sample	Interplanar Spacings			
Reactive fiber with precipitate	2.357	2.041	1.447	1.232
Metallic gold[a]	2.355	2.039	1.442	1.230
	(110)	(200)	(220)	(311)

[a] The data in the parentheses represent the Miller indices.

3. Analysis of the Reactive Fiber after Redox Reaction

Figure 4 presents the IR spectra of the reactive fibers before and after adsorption. After the adsorption process, the peak at 1600 cm^{-1} corresponding to the stretching vibration of C=N bond weakens and the peak at 925 cm^{-1} corresponding to the stretching vibration of N—O bond disappears. On the other hand, two new peaks at 1770 and 1220 cm^{-1}, corresponding to the stretching vibrations of the C=O bond and the C—O bond in the carboxyl group, respectively, appear. Consequently, the AO group is oxidized to carboxyl group during the redox process. To confirm this deduction, the IR spectrum of the reactive fiber oxidized by 0.05 *M* $KMnO_4$ solution at 25°C was compared with that of poly(acrylate acid). As shown in Figure 5, the two spectra are identical. Accordingly, it is certain that the AO group in the reactive fiber is finally oxidized into the carboxyl group by the Au(III) ion during the adsorption process.

CONCLUSION

1. The adsorption capacity of Au(III) onto the reactive fiber increases with the increase of AO group content. In the solution containing Au(III), Cu(II), Zn(II), and Cr(III), the reactive fiber presents high selectivity to Au(III).
2. During the adsorption process, the adsorbed Au(III) is partially reduced to spongy metallic gold and the AO group is finally oxidized to the carboxyl group.

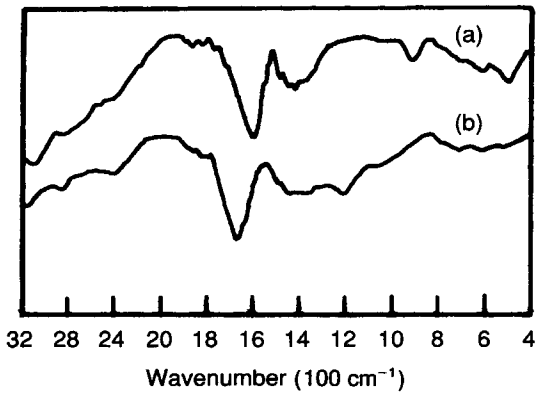

Figure 4 IR spectra of the reactive fibers: (a) before adsorption; (b) after adsorption.

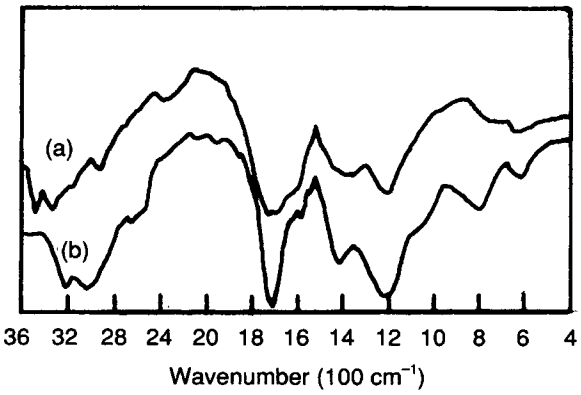

Figure 5 IR spectra for (a) reactive fiber oxidized by $KMnO_4$ and (b) poly(acrylate acid).

The authors gratefully acknowledge the support of the State Educational Commission (Grant No. 9255810) and the Zhongshan University Scientific Foundation (Grant No. 058A034).

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Received October 13, 1992
Accepted November 6, 1992