Electrolysis of Gold from Filtration Waste by Means of Mechanical Activation

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The intensification of the gold thiourea leaching from a filtration waste (Košice, Slovakia) using mechanical activation as the pretreatment step has been studied. The leaching of "as-received" sample in an acid thiourea solution resulted in 65 % Au dissolution. However, after mechanical activation in a planetary mill 99 % of the gold was leached. The optimum redox potential for electrolysis is in the range 500-523 mV for the gold extraction 99.79 % from the mechanically activated sample. The mechanical activation resulted in an increase of the specific surface area of the waste from 0.7 m^2g^{-1} to a maximum value of 13.5 m^2g^{-1}. The physico-chemical changes in the filtration waste as a consequence of mechanical activation had a pronounced influence on the subsequent gold extraction.

Key words: *waste, gold, leaching, mechanical activation, electrolysis*

Introduction

The filtration waste is characterized by significant heterogenity and relatively high complexity. The chemical, biological and physical pretreatments are applied as intervention steps directed to the solid phase the goal of which is to change the composition and particle size of the gold-bearing substances and thus to facilitate the subsequent leaching. The process is called mechanical activation and samples are subjected to intensive milling. This milling results in particle disintegration and chemical or physico-chemical transformations which significantly affect the subsequent input material processing operations (Baláž et al., 1996; Baláž et al., 2000; Baláž, 2000; Baláž, 2008; Ficeriová et al., 2005a; Tkáčová, 1989).

Electrolysis of gold from Au-waste represents one of various processing possibilities, aimed at obtaining gold in metallic form. In regard to the expected recovery yields and method of further utilisation, a suitable solution appears to be the use of electrolyser with a porous cathode made from carbon felt (Ficeriová et al., 2005; Ficeriová et al., 2008). The use of carbon felt as a three-dimensional electrode appears to be very promising for the recovery of heavy metals, and toxic compounds removal from dilute solutions, considering its favourable physico-chemical properties: high specific surface area, good fluid permeability and compressibility, chemical inertness and good electrical conductivity. Application of the electrolytic method of gold-bearing waste processing represents practical, economic, as well as ecological importance for the whole society. Recovery of gold from waste is of extraordinary importance under conditions of inflationary development, when this precious metal is a stable source of covering the needs of society.

The aim of this work was to examine the possibility of recovering gold from the filtration waste by using thiourea leaching. This work presents the contribution of the carbon felt as electrode in thiourea advance of obtaining the gold - as a cathode for the electrowinning of gold solution obtained after the elution of loaded carbon. A mechanical activation was applied in order to determine its effect on the recovery of gold.

Experimental

Material

The filtration wastes (Košice, Slovakia) were selected as an input material for testing the leaching of gold. The chemical composition of the filtration waste was as follows: 0.035 % Au, 7.84 % Cu, 3.28 % Zn, 5.36 % Pb and 24.63 % Fe.

Mechanical activation

Mechanical activation was performed in a planetary ball mill. Planetary ball mill of type Pulverisette 6 (Fritsch, Germany) was applied under the following conditions: volume of milling chamber 250 ml, weight of sample 20 g, steel balls (205.5 g of 10 mm diameter), milling time 30 min; revolutions of the milling shaft 500 min^{-1}; ambient temperature.

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Physico-chemical characterization

The specific surface area S_A was determined by the low temperature nitrogen BET adsorption method using a Gemini 2360 sorption apparatus (Micromeritics, USA).

The particle size distribution of the milled filtration waste was measured by a laser beam scattering in a Helos and Rodos granulometer (Sympatec GmbH, Germany). The mean particle diameter was calculated as the first moment of the volume size distribution function.

Thiourea leaching

The leaching was investigated in a 500 ml glass reactor into which 400 ml of leaching solution having 10 gl^{-1} $CS(NH_2)_2$, 5 gl^{-1} $Fe_2(SO_4)_3.9H_2O$, 10 gl^{-1} H_2SO_4 and 20 g of milled filtration waste were added. The leaching was performed at pH = 1 during 60 min at 293 K using a stirring rate of 8.33 s^{-1}. Aliquots (5 ml) of the solution were withdrawn at appropriate time intervals for determination of the content of dissolved gold by AAS method.

Electrolysis of gold from lixivium at thiourea leaching

The electrolysis was performed in a laboratory electrolyzer with carbon cathode and Pt-anode (Ficeriová et al., 2005b). The following experimental conditions have been selected: total time of electrolysis t_E = 60 min; catholyte – thiourea lixivium after leaching of filtration waste, catholyte volume – 800 ml; anolyte 10 gl^{-1} H_2SO_4, anolyte volume – 500 ml; electric current I = 0.5 A; volume of velocity flow of catholyte and anolyte V_V = 1600 $ml.min^{-1}$; temperature T = 25 °C; pH = 1. The redox potential in optimum leaching conditions was 500-523 mV (versus S.H.E.). Electrochemical redox potential (Pt with fiber with respect to saturated argentochloride electrode) was measured using a VDM-1 milivoltmeter (MESIT Company, Czech republic).

Results and discussion

Mechanical activation of waste

The mechanical activation of filtration waste is characterized by an increase in specific surface area of the treated material. The effect milling is shown in Fig. 1. The original value of surface area 0.7 m^2g^{-1} increased rapidly to a maximum value of 13.5 m^2g^{-1} with increase milling time.

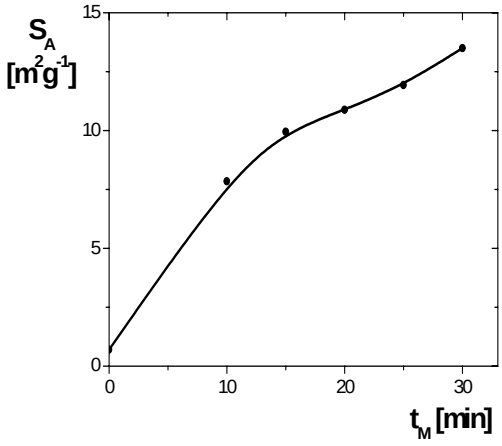

Fig. 1. The specific surface area, SA as a function of milling time, tM of the mechanically activated sample: Planetary ball mill, milling time, tM = 30 min.

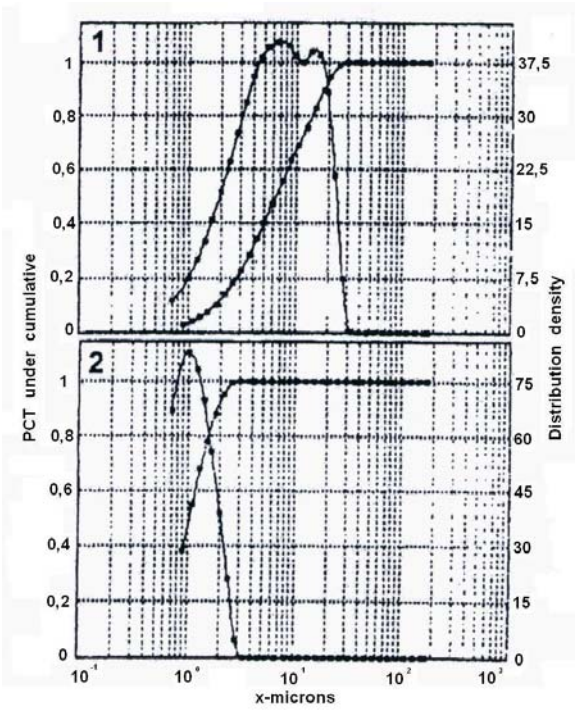

Fig. 2. Particle size analysis of the mechanically activated sample: 1 - "as-received" sample, 2 - planetary ball mill, milling time, tM = 30 min.

Increases in the fraction of fine particles and specific surface area are changes which are frequently observed as the consequence of intensive milling (Baláž, 2000; Baláž, 2008). This manifold new surface area

formation correlates with the particle size analysis of the milled samples (Fig. 2). The "as-received" sample has a 100 % abundance of particles with a diameter under 30 μm (1) but only 10 % of particles with diameter less than 10 μm. For the mechanically activated sample (2) the particles are smaller than those of the "as-received" sample.

In Tab. 1 the percentage of undersized particles is given for this sample.

Tab. 1. The percentage of undersized particles for filtration waste.

Sample No.	**Milling time, t_M** [min]	**Undersize** [%]			
		- 5 [μm]	**- 10** [μm]	**- 40** [μm]	**- 60** [μm]
1	-	23	68	96	100
2	30	98	100	100	100

1 – "as-received" sample, 2 - sample after mechanical activation in planetary ball mill.

Senna (1989) analysed the effect of surface area and the structural disordering on the leachability of mechanically activated samples. In order to solve the problem – whether surface area or structural disorder are predominant for the reactivity - the rate constant of leaching is divided by the proper surface area and plot against the applied energy by activation (or milling time).

In the case, where k_{Au}/S_A increases with increasing milling time, as shown in Figure 3, the surface area S_A, may be the effective surface area, with an overlapping effect of the structural disorder, as a result of mechanical activation in dry mode. Gold is metal with great plasticity. During its milling, introduction of some type of deffects is expected (e.g. dislocations) which can be responsible for the observed phenomena of its structural disorder.

Thiourea leaching of gold from mechanically activated waste

The dependence of gold recovery on leaching time for mechanically activated sample is represented in Fig. 4. The recovery of only 65 % of Au was reached after 60 min of leaching for the "as-received" sample (curve 1). The results for the mechanically activated sample (curve 2) indicated that the physico-chemical changes of the filtration waste brought about an acceleration of the process of thiourea leaching. It was possible to achieve a gold recovery of 99 % after one hour of leaching for mechanically activated sample. In Tables 2-3 are shown redox potentials under different times of electrolysis. From Table 3 it is evident that maximum recovery of gold in 15 minutes was 99.79 % at the redox potential 500-523 mV. The results have shown in all cases that the deformation of elements brings about a shift in potential to more negative values (in comparison with non-deformed elements). The values of electrode potentials relax in the course of time. The process of relaxation of potential is dependent on the kind of electrolyte and extent of deformation of element surface as well as on the kind of element.

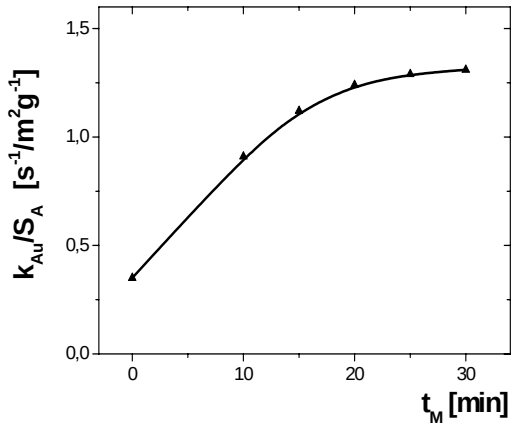

Fig. 3. Specific rate constant of gold leaching, kAu/SA vs. milling time, tM for the mechanically activated sample in a planetary ball mill.

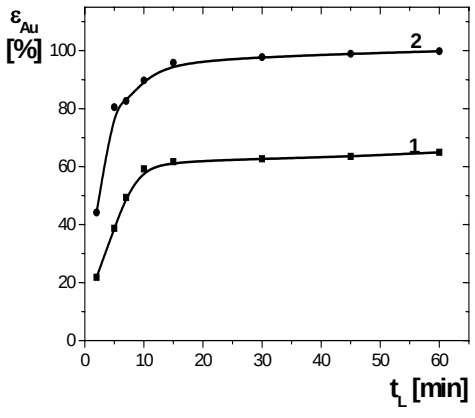

Fig. 4. Recovery of gold, Au vs. leaching time, tL for the mechanically activated sample.
1 - "as-received" sample, 2 - planetary ball mill, milling time, tM = 30 min.

Tab. 2. Redox potentials of the electrolytic system and recovery of Au for „as-received" sample, temperature = 25 °C, pH = 1.

Times of electrolysis [s]	**Range of redox potential** [mV versus S.H.E.]	**Recovery of Au for „as-received" sample** [%]
300	433-465	18.96
600	470-491	45.79
900	500-519	58.14
1800	581-603	46.37
2700	476-490	57.65
3600	475-487	49.26

Tab. 3. Redox potentials of the electrolytic system and recovery of Au for sample after, mechanical activation in planetary ball mill, temperature = 25 °C, pH = 1.

Times of electrolysis [s]	**Range of redox potential** [mV versus S.H.E.]	**Recovery of Au for sample after mechanical activation in planetary ball mill** [%]
300	430-464	26.54
600	475-490	83.29
900	500-523	99.79
1800	587-610	76.10
2700	480-492	79.44
3600	472-491	82.57

Conclusion

Mechanical activation of filtration waste (Košice, Slovakia) has a positive influence on gold leaching. An optimum Au recovery of 99 % was achieved from the mechanically pretreated sample 30 minutes in planetary mill already in 45 minutes of thiourea leaching. The leaching of "as-received" (non-treated) waste resulted in only 65 % Au dissolution. The consumption of milling time has an influence on the physico-chemical changes of filtration waste due to mechanical pretreatment as is evident in the thiourea leaching. Thiourea leaching is very advantageous kinetic process for gold solubilization in comparison with kinetics and noted disadvantage of the classical cyanide leaching. The redox potential connected with optimum leaching conditions is in the range 500-523 mV with maximal gold extraction during 15 minutes.

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