

A closed-loop electrogenerative recycling process for recovery of silver from a diluted cyanide solution

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Electronic supplementary information

1. Experimental

1.1 Chemicals and materials

Potassium silver cyanide, $\text{KAg}(\text{CN})_2$, sodium cyanide, NaCN , palladium chloride, PdCl_2 were purchased from Merck (Malaysia). Tin chloride, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, nitric acid, HNO_3 and hydrochloric acid, HCl were obtained from ChemAR (Malaysia). While pyridine-barbituric acid (Fischer Scientific, USA) was used as the reagent for cyanide determination. Oxygen, O_2 and nitrogen, N_2 gaseous were supplied by MOX (Malaysia). Electrodes used were reticulated vitreous carbon (RVC) 80 ppi, porous graphite (PG) (SG 132) and pure zinc (99.0% purity), which all were procured from The Electrosynthesis Company (USA). A saturated calomel electrode (SCE) (Hanna Instruments) with a Luggin capillary was used as a reference electrode.

1.2 Batch cell configuration

The batch cell used in this study is consisting of two electrolyte compartment of dimensions $11.0 \text{ cm} \times 5.5 \text{ cm} \times 8.0 \text{ cm}$, which was separated by a cation exchange membrane, Neosepta® CM-01 (Tokuyama Corp.). Parafilm (American National Can™) was placed between the compartments. The electrode materials used for silver recovery, RVC, PG and zinc have the dimensions of $5.0 \text{ cm} \times 2.5 \text{ cm} \times 0.5 \text{ cm}$. The electrodes were then attached to stainless steel strips where the strips acted as the current collectors. The attachment was made by PTFE tape in order to prevent the silver deposition onto the part of current collectors that in contact with electrolyte. The stainless steel was then connected by an external conducting wire to complete the circuit. The distance between the cationic membrane and each of the electrode was kept as close as possible but not touching the cationic membrane.

1.3 Optimization procedures

Various initial silver cyanide catholytes were prepared from $\text{KAg}(\text{CN})_2$, and NaCN , in which the concentrations of the silver ranged from 10 to 500 mg L^{-1} in 0.2% (w/v) NaCN solution. The used anolyte (0.5% (w/v)) was prepared from NaCN . All solutions were freshly prepared prior to each experiment by using $18 \text{ M}\Omega^{-1}$ distilled deionized water. In some experiments, the electrolytes were

deoxygenated with N₂ gas for 10 minutes with a flow of N₂ gas blanketed the electrolytes throughout the experiments. 100 mL of catholyte and 100 mL of anolyte were used in their respective compartment for each experiment. Both catholyte and anolyte were stirred throughout the experiments by using the magnetic stirrers with the speed of 300 rpm. Details of experimental conditions are shown in Table 1.

The cathodes were also activated using a Shipley–type solution [1]. The batch cell was short-circuited and a known amount of catholyte was withdrawn periodically in order to monitor the performance of the electrogenerative process and ultimately determine the silver concentration. The remaining silver concentration was determined by using an atomic absorption spectrometer (Perkin Elmer model 2100).

As for the reproducibility test, a particular experiment was performed three times. In order to verify the presence of silver deposited at the cathode surface after the experiment, the cathodes were visualized by using a scanning electron microscope, SEM-EDX (Leo Supra model 50 VP). All of the output voltage and current of the system were measured with a digital multimeter (Sanwa). All experiments were conducted at room temperature.

1.4 Determination of cyanide solution

After the silver recovery process is completed, the concentration of catholyte and anolyte were determined spectrophotometrically with pyridine-barbituric acid as the reagent, using a UV-Visible spectrophotometer (UV-2600 Shimadzu). The presence of cyanide was confirmed with the formation of a red-blue coloured complex and the absorbance was measured at 578 nm [2].

References:

[1] C.Y. Yap, N. Mohamed, Electrogenerative processes for environmental applications. CLEAN-Soil, Air, Water 36 (2008) 443–452.

[2] American Public Health Association (APHA); American Water Works Association (AWWA); Water Environment Federation (WEF), Standard methods for the examination of water and wastewater, 20th ed.; Washington, D.C. 1999.