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## Separation and Recycling Nitric Acid – Metals Waste Waters from a Brass Pickling by Diffusion Dialysis – Selective Physico - Chemical Methods

Afaf LALMI<sup>1</sup>, Chahrazad AMRANE<sup>2</sup>, Kamel-Eddine BOUHIDEL<sup>3</sup>

<sup>1</sup> Laboratory of Chemistry & Environmental Chemistry LCCE, Team: Water Chemistry, Environment & Desalination. Faculty of Sciences, University Hadj Lakhdar Batna1

<sup>2</sup>, Laboratory of Chemistry & Environmental Chemistry LCCE, Team: Water Chemistry, Environment & Desalination. Faculty of Sciences, University Hadj Lakhdar Batna1

<sup>3</sup>, Laboratory of Chemistry & Environmental Chemistry LCCE, Team: Water Chemistry, Environment & Desalination. Faculty of Sciences, University Hadj Lakhdar Batna1 <sup>1</sup>afaf-eau@hotmail.com

## Abstract—

This research work applies a clean technology to treat the concentrated waste waters of a brass pickling bath. This last contains high concentration mixture of nitric acid and dissolved metals ( $Cu^{++}$ ,  $Zn^{++}$  and  $Pb^{++}$ ).

Now the studied industrial plant neutralizes HNO<sub>3</sub> and precipitates the metals with these 3 major drawbacks:

- Loss of an expensive acid ( 50 % of the initial quantity ) Pollution of the treated and rejected effluents by nitrates
- Production of a problematic (filtration, storage, fate ...) metal hydroxides sludge;

To solve these problems we apply a clean hybrid technology:

- The diffusion dialysis with anion exchange membranes AMX; the result is excellent. We separate and purify HNO<sub>3</sub> with a high efficiency.
- The remaining metals mixture contains Cu<sup>++</sup>, Zn<sup>++</sup> and Pb<sup>++</sup> which are processed by chemical ( selective precipitation ) and physico – chemical ( cementation ,...) methods .

**Key** – **words:** diffusionnel dialysis brass pickling bath recovery of metals cementation.

#### I. INTRODUCTION

Surface treatments are intended to confer new properties to the metal through electrolytic or chemical deposition [1].

Brass is one of the most used alloys in the surface treatment industry. There is the brass operation (recovering metal parts with brass: copper, zinc and lead). There is also the stripping operation before or after manufacture of brass parts. There is also the operation where the piece of brass is covered by another metal (nickel, copper ...), a practice very common in this industry.

The enormous quantities of metals generated by this industry pose major problems in the management of this waste. In Algeria, in the absence of controlled landfills and recycling, sludge is either stored in factories or released into the environment.

To reduce or eliminate sludge production, it is necessary to apply clean technologies to the source of the pollution [2]. These technologies are based on the concepts of "waste minimization", "zero discharge", "pollution prevention", ... We will present a model example of the BCR / of Ain Kebira (Setif). The pickling of the brass takes place in concentrated baths of nitric acid. The attack of the oxidized surface solubilizes Cu<sup>++</sup>, Zn ++ and Pb<sup>++</sup>. The spent bath will therefore be neutralized by lime with simultaneous precipitation of the three metal hydroxides [3]. The major drawbacks of this conventional practice, widely applied in Algeria, are environmental (sludge and liquid discharges loaded with nitrates, pollution of water resources, soil, etc.) and economic (reagents, energy, basins, management, sludge and environmental penalties, ...). As for leaching, this also offers acid dialysis to recover and completely recycle excess nitric acid, isolation and selective separation of the three metals. A publication dedicated to this theme confirms the originality and relevance of this idea.



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## II. EXPERIMENTAL

- Nitric acid analysis and dialysis control : pH and titration ;
- Metal analysis : Atomic Absorption and Visible Spectroscopy ;
- Dialysis : A two compartment cell separated by an AMX anion exchange membrane ;
- Selective precipitation by different chemicals: phosphate, NaOH...

## III. RESULTS AND DISCUSSION

Diffusion Dialysis of nitric acid of the brass pickling used bath gives an excellent output of recuperation (approximately 50%). Separation by selective separation and cementation made it possible to recover the three metals of brass pickling bath. Results represented in tableau 1.

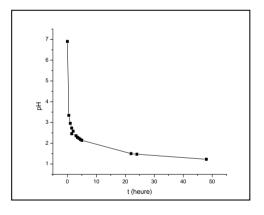


Figure 1: Evolution of the pH of dialysate during the dialysis of a solution of  $HNO_3$  in the system  $HNO_3$  (brass pickling)//H<sub>2</sub>O distilled.

Tableau 1: percentage of recovery of metals in the brasspickling bath.

	pks	Initial mass (mg)	The mass remain (mg)	R%
Lead II	43.5	6.21	$\cong 0$	100%
Copper II	36.9	225.35	9.6625	91.25%
Zinc II	32	13.436	5.19	86.61%

## IV. CONCLUSION

The objective of our work is the study of possibility of valorization of bath brass pickling by selective precipitation, cementation and dialysis.

The experimental study that we carried out allowed us to note the following results:

- Diffusionnel dialysis gives a very important (47.86%) of nitric recovery of acid of brass pickling.
- Selective precipitation by phosphates leads of the excellent outputs of recovery: 100 % of Pb <sup>++</sup>, 91.275 % of Cu <sup>++</sup> and 86.61% of Zn <sup>++</sup>.
- The test of copper cementation by powder of the zinc excess showed that the reaction gives an excellent output (74.32%) of copper recovery.

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