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FREE ACIDS AND CHEMICALS RECOVERY FROM STAINLESS STEEL PICKLING BATHS

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ABSTRACT

The conventional process used to treat waste acids from the stainless steel pickling baths is a direct neutralization with alkali. However, this process requires a high consumption of chemicals, produces a large amount of solid wastes, and furthermore, does not meet the recent European Waste Treatment Regulation because of the nitrogen contained in the final effluent. This paper presents an alternative treatment method based on a diffusion dialysis process for the free acid recovery and a neutralization process, followed by a salt splitting process, for the chemical recovery. This alternative eliminates waste disposal problems and, at the same time, it regenerates and recovers valuable by-products, which contributes to the

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economic feasibility of the process. Therefore, the work presented is focused on the technical and economical feasibility of electromembrane processes for the European spent pickling baths. Furthermore, bipolar electrodialysis and membrane electrolysis systems are compared for the salt splitting process. At the moment, there is a partial process in Washington Steel based on salt splitting; however, the recovery of free acid is not considered, and furthermore, the different concentration of the European pickling baths makes this process unsuitable for European stainless steel producers.

Key Words: Pickling; Stainless steel; Pickling liquors; Chemical recovery; Electrodialysis.

INTRODUCTION

During the pickling operation of stainless steel with nitric and hydrofluoric acids, metals are dissolved in the bath. When a critical metal concentration is reached, some kind of fluoride precipitation could appear, and therefore, the pickling bath is considered spent and has to be purged. This stream has traditionally been neutralized with alkali, producing sludges that represent serious environmental and economical problems because these wastes contain nitrates, fluorides, acids, and solid materials rich in heavy metals that have to be taken to special landfills. In addition, to consuming chemicals and producing wastes, this treatment does not meet the recent Waste Treatment Regulation because of the nitrogen contained in the final effluent. To avoid these severe environmental problems, new alternatives have been considered to establish a new process that would eliminate waste disposal problems and, at the same time, regenerate and recover valuable by-products to contribute to the economic feasibility of the process.

In this paper, an alternative for the treatment of nitric-hydrofluoric pickling liquors is presented. This alternative treatment is a combination of novel and existing technologies, selected in order to maintain the water balance in the system and to recover all of the valuable chemicals and sub-products. It includes three steps: free acid recovery, metal separation, and recovery of chemicals.

Figure 1 shows the flow diagram for the closed-loop recovery of spent stainless steel pickle acids, which is an improvement of the Aquatech System (9,10).

For free acid recovery, diffusion dialysis (DD) traditionally has been presented as one of the best and most energy efficient processes. A separation process is promoted by means of the concentration difference of two acid solutions separated by anion-exchange membranes. By arranging multiple anion-exchange membranes and circulating acid solution and water in counter current alterna-

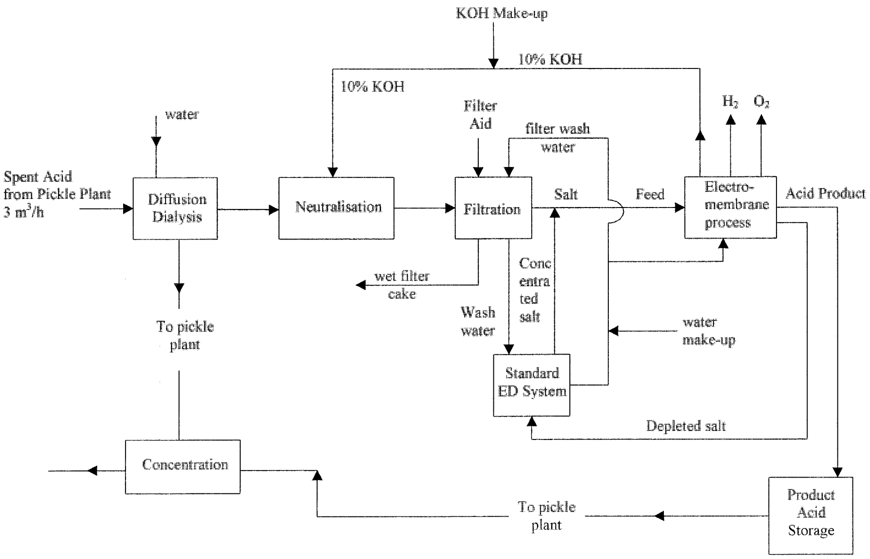

Figure 1. An alternative process for pickling liquor recovery.

tively, mainly the acid permeates through the membrane. Other electrolytes cannot permeate through the membrane because cations, except hydrogen ions due to their extremely small size, are repelled by the positive charge of the membrane. To prevent membrane fouling, concentration in suspended solids must be lower than 2–3 ppm and for that reason a double stage filter system is usually employed.

Diffusion dialysis has already been applied to the recovery of sulfuric acid, hydrochloric acid, nitric acid, and hydrofluoric acid and it has been adopted for the recovery of free acids from the pickling treatment of stainless steel plates. This process is simple and easy to operate and maintain, which allows an unattended and continuous operation over long hours. In fact, several such installations have been built and are working at this moment, as has been described in the existing research literature (1–7). Therefore, no further experimental study is necessary for the integration of this process in the new global treatment system and only the most relevant data from the literature, those that are relevant for the global process, are reviewed in this paper.

The acid recovery rate depends on the conditions of dialysis, especially when dialysis is carried out in the presence of three species: nitric acid, hydrofluoric acid, and ferric ion. Data obtained by Tokuyama Soda Co. showed a recovery rate of 97% and 50% of nitric and hydrofluoric respectively, with a recovered acid stream of 60 g/l HNO_3 and 6.4 g/l HF (1).

The concentration of the recovered acids is adjusted with fresh acids before this stream is recycled to the pickling bath. Removal of the free acids, is first step of the process, reduces not only the consumption of neutralizer agent and sludge production, but also the flow of the final effluent and its nitrogen concentration.

After removing the free acids, the waste acid solution, which still contains part of the free acids (10% HNO_3, 50% HF), the complexed acids, and the dissolved metals, is neutralized by a precipitation-controlled process. During this process, metal hydroxides and oxyhydroxides are generated and precipitated. The metal sludge is separated, washed (in order to avoid anion losses), and drained with a filter press in order to recover the metals, which can be reused after treatment as raw material for the steel process.

If KOH is used for the neutralization, the potassium salts (potassium nitrate and fluoride) that are formed during neutralization remain in the solution due to their high solubility. The salts of this filtrate are split into their corresponding acid and base in an electromembrane process, which can have either a bipolar membrane electrodialysis configuration or a membrane electrolysis configuration. Then the recovered acid is recycled to the pickling bath and the base to the precipitation process. The depleted salt solution is sent to a standard electrodialyzer with monopolar membranes to upgrade its concentration. The electrodialysis dilute water is used as rinsing water in the filtration process of the metal hydroxides to wash the cake, and the concentrated salt is returned to the electromembrane unit for its splitting, to further recover the acid and base content (8–10).

For closed-loop operation, it is necessary to maintain an appropriate water balance around the integrated pickling line and the acid recovery operations. Depending on the industry needs, this can be achieved by the reuse of the spent acid stream from DD as input in the acid compartment of the salt splitting unit and by the reuse of the dilute stream from the standard electrodialysis unit as the water stream for DD.

Proper operation of the integrated global system relies on the ability of the acid recovery system to adjust the concentration of spent pickle acid. Water is removed by the electrodialysis unit into temporary storage when the spent acid concentration delivered to it is lower than desired, and added from temporary storage when the spent acid concentration is higher than desired.

Due to the limited data available regarding the application of water splitting technology and in order to integrate the full system for the treatment of European pickling liquors, this paper focuses on the technical and economical feasibility of the electrochemical process. The alternative proposed in this paper, as an integrated process, improves the existing process used in the United States in several ways because it enables: the recovery of free acids, the reduction of base consumption, the selective precipitation of metals, and the reduction of wastes.

EXPERIMENTAL

The characteristics of the stainless steel pickling liquors treated in this work are given in Table 1.

Table 1. Characteristics of Pickling Liquors

Free HNO_3	125 g/l
Free HF	30 g/l
(NO_3^-)total	137 g/l
$(F^-)_{total}$	80 g/l
Fe_{total}	40 g/l

The initial step in the chemical recovery system, after DD, is the neutralization of the waste acid solution by a controlled precipitation process with potassium hydroxide. Most of the fluoride present in the spent pickle acid is in the form of complexed metal fluorides rather than as free acid. Conversion of metal fluorides to form the associated potassium salts and metal hydroxides is represented by the following equations:

$$FeF_3 + 3KOH \rightarrow 3KF + Fe(OH)_3 \qquad (1)$$

$$CrF_3 + 3KOH \rightarrow 3KF + Cr(OH)_3 \qquad (2)$$

$$NiF_2 + 2KOH \rightarrow 2KF + Ni(OH)_2 \qquad (3)$$

These reactions are important because they enable the recovery of as much acid as possible in the global process. The obtained salt solution of potassium nitrate and potassium fluoride is then processed in the electromembrane water splitting unit. Two alternative systems have been studied: bipolar membrane electrodialysis and membrane electrolysis.

Bipolar Membrane Electrodialysis (BM)

In the bipolar membrane system, the ion–exchange membranes, as well as the cationic, anionic, and bipolar membranes placed alternatively are arranged in a 3-compartment cell stack configuration. The bipolar configuration system operates with three primary circulation loops (acid, base, and salt) that are continuously recycled through the cell stack.

When an ionic feed solution is pumped through the cell pairs and a direct current is applied, the positively charged ions (K^+) move across the cation-selective membrane migrating toward the cathode into the base compartment. At the same time, $(NO_3)^-$and F^- ions move across the anion selective membrane toward the anode into the acid compartment. The anions cannot pass the negatively charged membranes and the cations cannot pass the positively charged membranes.

The key element in this electrodialysis process is the bipolar membrane, so-called because it is composed of two different layers that are selective to ions of opposite charge (12). Water molecules diffuse from the acids and base circulation loops into the bipolar membrane interface, where they are dissociated to H^+ and OH^-. These electrically charged species selectively pass across the outer boundary of the bipolar membrane toward either the anode or cathode. The H^+ ions move into the acid compartment and associate with the NO_3^- and F^- ions from the salt splitting, thereby forming the mixed acid product that will be recycled to the pickle lines. The OH^- ions move into the base compartment and associate with the K^+ ions from the salt splitting, thereby forming the base product that is reused in the neutralization process.

Electrolysis occurs at the electrodes, with hydrogen (H_2) and hydroxyl ions (OH^-) being produced at the cathode and oxygen (O_2) and hydrogen ions (H^+) being produced at the anode. Thus, an additional electrode rinse circulation loop is required in the system to remove the small amount of gas that is formed at the electrodes of the cell stack.

To study the current efficiency of the ion-exchange membranes and the influence of the different variables on the electrodialysis process, the following initial operation conditions were chosen (see Table 2).

The experiments were carried out in a lab-scale reactor with the bipolar configuration system shown in Fig. 2. An extra cation-exchange membrane is used in the cathode side to avoid the migration of the OH^- ions, which are produced on

Table 2. Operation Conditions of the Electrodialysis Process

Cell components	
Electrodes:	Ti/IrO_x
Frames:	2 mm thickness
Turbulence promoters:	~0.9 mm. Type Tholen
Ion-exchange membranes of 10 by 10 cm^2:	Cationic: CMX Anionic: AHA–1 or AHA–2 Bipolar: BP–1
Operation conditions	
Initial volumes:	0.25 l
Pressure:	0.25 atm
Temperature:	20–25°C
Flow velocity:	300 ml/min
Current density:	100 mA/cm^2
Feed solutions:	Pure salt: KNO_3 1.5N Mixed salt: KNO_3/KF with low nitrate content KNO_3/HF with high nitrate content Acid: HNO_3 lN Base: KOH 1N

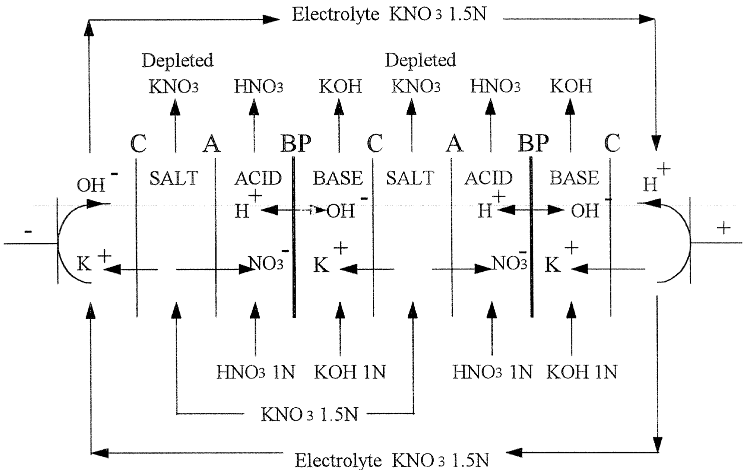

Figure 2. Salt splitting of the pure salt in a 3-compartment bipolar cell with 2 cell pairs.

the cathode, toward the other compartments and to keep the electrode solution neutral.

Membrane Electrolysis (ME)

The production of HNO_3/HF and KOH from KNO_3/KF has also been studied in a membrane electrolysis system (Fig. 3) with the same operation conditions as found in Table 2.

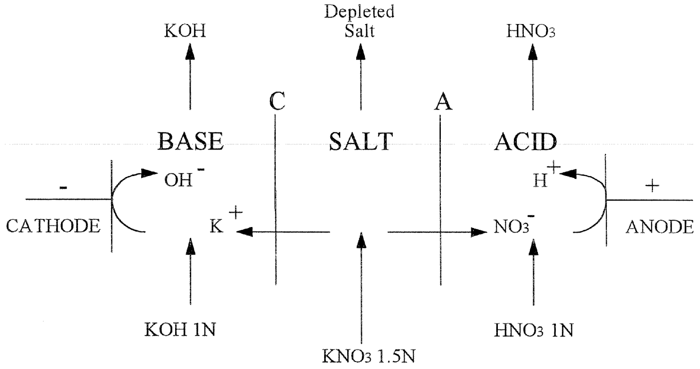

Figure 3. Salt splitting of the pure salt in a 3-compartment membrane electrolysis system.

Bipolar Electrodialysis and Membrane Electrolysis Experiments

The first set of experiments were carried out in order to optimize the cell and the operational conditions. After optimization, the production of mixed acid from high concentrated nitrate salts (to simulate the global process without the recovery of free acid) and low concentrated nitrate salts (to simulate the process with the free acid recovery step) was studied in order to determine the economic evaluation of the different electrochemical possibilities for the global chemical recovery task. Bipolar electrodialysis and membrane electrolysis were compared in order to select the best electromembrane process for the global treatment system.

RESULTS AND DISCUSSION

To test the possibilities of bipolar electrodialysis and membrane electrolysis for the salt splitting after neutralization, experiments were conducted with synthetic KNO_3 and with mixtures of KNO_3 and KF.

The production of nitric acid from pure KNO_3 salt solution has a relative low current efficiency for both the bipolar technology and the membrane electrolysis. For the bipolar configuration, the current efficiency for the production of 1–2 mol/l nitric acid from the acid compartment averaged 50%. In some of the previously conducted research (4), a somewhat higher value (up to 70%) is given.

Figure 4 shows membrane electrolysis experiments that produce high concentrations of nitric acid from pure KNO_3 solutions. This figure clearly shows that the current efficiency decreases as the nitric acid product concentration increases. At a high nitric acid concentration, the current efficiency becomes low because of the increasing proton leakage through the anion membrane.

When mixed salt solutions with a high nitrate concentration (the case in which no free acid recovery is considered in the global system) are electrolyzed, the anions are removed in two parts. Figure 5 shows the result of an experiment in a membrane electrolysis cell. In the first part, the nitric acid content in the acid product increases and the fluoridic content stays constant. The nitrate is removed first because the fluoride is only partly dissociated in mixed acid solutions. The current efficiency in the first part of the experiment is comparable with the current efficiency for pure nitric acid production as shown in Fig. 4. In the second part of the experiment, when almost all the nitrate is removed, fluoridic acid is also produced. The cumulative current efficiency increases from 55% to above 70% in this part, indicating that the fluoride removal in the second part of the experiment is very effective.

As mentioned above, it is more effective to treat the spent acid with a combination of diffusion dialysis and salt splitting. In the first step, the free nitric acid is removed (90%) with a part of the free fluoridic acid (50%). After neutralization,

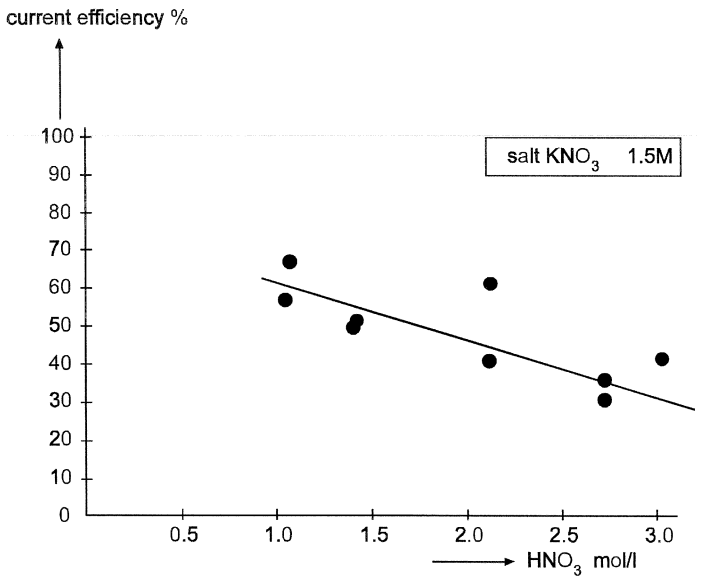

Figure 4. Production of nitric acid from pure 1.5 M KNO_3 solution with membrane electrolysis.

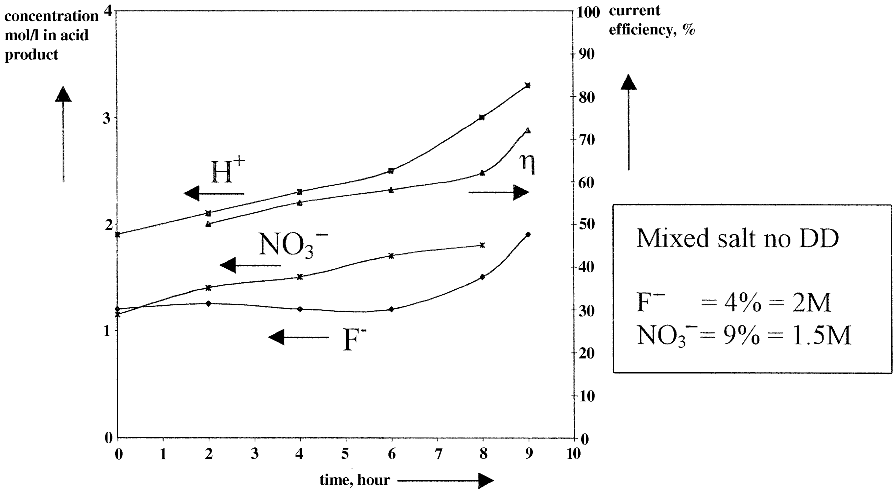

Figure 5. Production of mixed acid from salt solution with low ratio fluoride to nitrate (no free acid recovery).

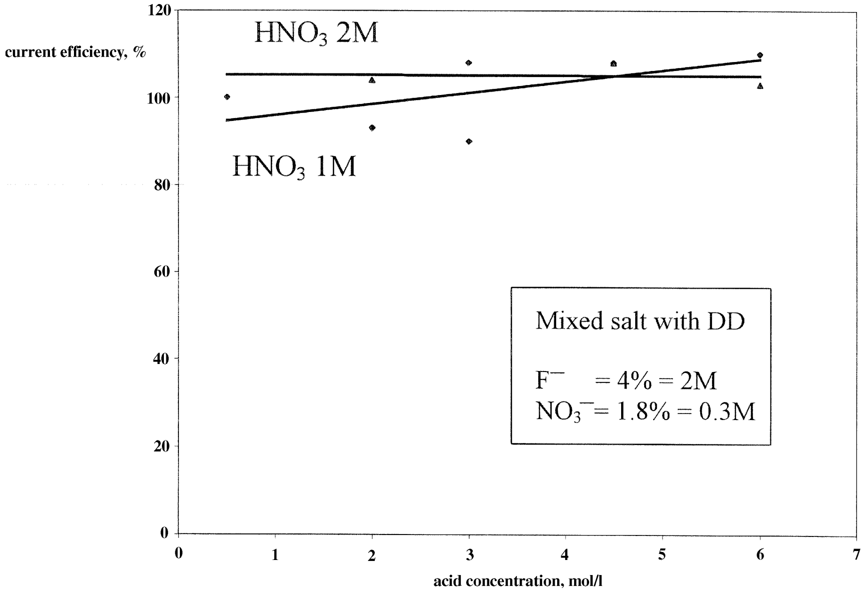

Figure 6. Production of mixed acid from salt solution with a high fluoride to nitrate ratio (after free acid recovery).

a mixed salt solution with a high fluoride to nitrate ratio is produced. In the following salt splitting step, the fluoride can be removed very efficiently. Figure 6 shows the results for a membrane electrolysis of a mixed salt solution with a high fluoride to nitrate ratio.

The current efficiency in this case was 100%, indicating a very efficient production of mixed acid, even in concentrations up to 6 mol/l. In these experiments, the starting acid solution contained 1 or 2 mol/l HNO_3. Normally, if strong acid is present in the acid compartment, one should expect proton leakage, resulting in a low current efficiency. In this case, however, the current efficiency was high, indicating that the protons that leaked to the salt compartment were transported back to the acid compartment as bifluoride (HF_2^-).

Practical Aspects to Produce Highly Concentrated Mixed Acid

To produce highly concentrated mixed acid, it is necessary to make a coupling from the output of the salt splitter to the input of the free acid recovery. In theory, a bipolar unit or a membrane electrolyzer can be used. However, under practical conditions, membrane fouling or corrosion problems can occur.

When a bipolar unit is used, it can be expected that due to the co-transport of potassium through the bipolar membrane (approximately 2–3 mol%) (13), the acid product will contain a relatively high concentration of potassium. This potassium-rich acid product can cause severe fouling problems in the free acid recovery unit if the spent acid contains silica. Silica can form insoluble K_2SiF_6 salt, which can foul the membranes. In the practical spent acid used for this research project, silica was present in a concentration of 3 g/l. The silica was dissolved in the bath from the stainless steel, which contained a low percentage of silica. The experiments showed membrane fouling and, therefore, the process in Washington Steel cannot be used for the treatment of the European liquors that contain silica.

When a membrane electrolyzer is used, there is no transport of potassium toward the acid compartment. However, the corrosion stability of the anode can be a critical point. To get a feasible process, the electrode lifetime should be at least 4000 hours or more. Most electrodes do not have this lifetime in fluoridic media.

Platinum has a long lifetime, but it is too expensive to be used on practical scale. Lead-alloy and carbonaceous materials are cheaper, but in general, they are not stable enough. Special electrodes like ebonex have a relatively good stability in fluoridic media. If this type of electrode is used in combination with an extra compartment, the corrosion problems probably can be minimized. No research on corrosion stability has been conducted in this study.

Economical Evaluation

Table 3 shows the cost information regarding the electrochemical unit operations. The bipolar salt splitting is relatively expensive due to high investment costs and high costs for membrane replacement.

Table 3. Comparison of the Electrochemical Treatment Methods

	Bipolar Unit	Membrane Electrolyse	Membrane Electrolyse
Type of solution	High nitrate salt	High nitrate salt	Low nitrate salt
Capital costs (euro/keq. acid)	26.8	9.3	6.7
Energy costs (euro/keq. acid)	7.0	14.9	10.7
Membrane costs (euro/keq. acid)	16.8	7.8	2.2
Anode costs (euro/keq. acid)	0	3.1	5.6
Total (euro/keq. acid)	50.6	34	25.2

Table 4. Basic Data for Economical Calculations

Spent acid flow (m^3/year)	24.000
Rinse water flow (m^3/year)	100.000
Composition spent acid	See Table 1
Composition rinse water	spent acid 80 times diluted
Costs chemicals (100 weight %)	
HNO_3 (euro/ton)	1160
HF (euro/ton)	1000
$Ca(OH)_2$ (euro/ton)	70
Sludge disposal (euro/ton)	12.5
Overall acid recovery (%)	95
Overall base recovery (%)	95

A cost estimation has been made for the pickling process with mixed HNO_3/HF acid on a scale of 24,000 m^3/year of spent acid production. The total process also involves the treatment of the rinse water (100,000 m^3/year) by reversed osmosis. Table 4 gives the basic information for these calculations.

Table 5 shows the results of the economical evaluation for the total process. For the salt splitting step, membrane electrolysis was evaluated because this is the most promising option for silica containing mixed acid from a technical and economical point of view. Two options were considered with and without electrowinning of the metals. In this evaluation, the credits for the recovered chemicals and the decreased sludge disposal were also considered.

The payback time for each option is approximately 2.2–2.4 years. During the next part of the Brite Euram project (pilot plant experiments) more detailed economical information will be gathered.

Table 5. Cost Estimation of the Treatment of Mixed Acid with Membrane Electrolysis

	HNO_3/HF K Euro	HNO_3/HF with Electrowinning K Euro
Operating costs	6738	6048
Total Installation costs	7082	10165
Credit and savings	9641	10714
Yearly net operation costs	2902	4668
Payback time	2,4	2,2
Sludge disposal costs	125 euro	

CONCLUSIONS

This paper has presented a process to treat HNO_3/HF spent acid from stainless steel production in Europe. The process combines free acid recovery and electrochemical salt splitting.

In the first free acid recovery step, more than 90% of the free nitric acid can be removed as well as approximately 50% of the free fluoridic. After neutralization of the remaining stream, a salt solution with a high fluoride to nitrate ratio can be split very efficiently into acid and base. The acid product of the salt splitting unit can be used as input for the free acid recovery unit.

When bipolar membranes are used, the acid product will contain potassium. This potassium can cause membrane fouling of K_2SiF_6 in the free acid recovery unit if the spent acid also contains silica, as in the case of the European liquors. When a membrane electrolysis system is used this problem will not occur; therefore, this alternative is more promising. However, with membrane electrolysis corrosion, stability of the anode in this situation can be a problem. The use of better fluoride resistant electrodes will have to be tested on their performance.

The most relevant advantages of the proposed process are undoubtedly related to environmental pollution. This new proposed process would represent the possibility of having a closed-loop of water and no solid or very low solid waste production in stainless steel factories. Furthermore, the recovery of valuable metals and chemicals would contribute to the economical viability of the process. The payback time of the proposed process is less than 2.5 years.

Local regulations (sludge disposal, effluent emissions) regarding waste disposal can be very different. Therefore, the PIBARE project can be adapted to the local needs of individual stainless steel producers.

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