

The AX Process

Recovery of Acids from Stainless Steel Pickling Baths



Acid Extraction by Pulsed Columns

A stainless steel pickling bath originally contains 2.2 M HNO_3 and 1.6 M HF . The bath is used until the iron concentration has reached 40-80 g/l. At this stage, the bath contains about 50 % unused acids, in addition to the dissolved metals, mainly iron, nickel, chromium and molybdenum.

Undissociated acids like nitric and hydrofluoric acids form adduct complexes with organic compounds, containing oxygen donor atoms. As the adducts formed are soluble in organic solvents like kerosene, the acids can be extracted from an aqueous solution by solvent extraction.

In the used pickling bath, however, most of the acids are bound to the metals as nitrate and fluoride complexes. By adding sulphuric acid to the pickling bath, sulphate will replace some of the nitrate and fluoride in the metal complexes, leading to the formation of extractable, undissociated acids. The general procedure to remove monovalent acids from an aqueous solution by adding sulphuric acid and then extract the released acids with an organic donor molecule, dissolved in an organic solvent, is designated Acid eXchange or the AX process.

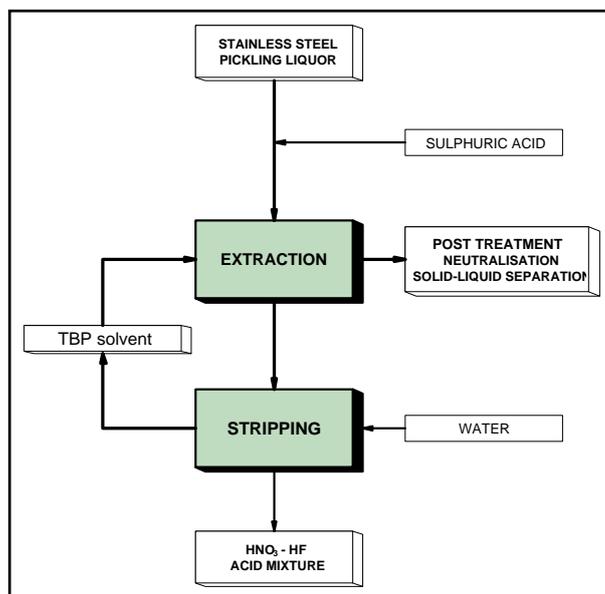
The main steps in a plant for treating spent stainless steel pickling liquid are:

MEAB

- Addition of H_2SO_4 and extraction of HNO_3 , HF and Mo with an organic solution, containing 75 % TBP in kerosene, in a pulsed column operation
- Precipitation of the metals (Cr, Fe and Ni) left in the aqueous raffinate
- Stripping HNO_3 and HF from the organic solution with water in a second pulsed column operation and recycle the acids back to the pickling bath
- Washing out Mo from the organic solution with a sodium hydroxide solution in a mixer-settler stage and recycle the organic solution to extraction.

A plant for treating 600 l/h spent pickling liquid (corresponding to an annual production of 25,000 ton stainless steel) was in

operation for some years in Sweden. The recovery operation was successful, however, the steel work is now closed.



References.

Recovery of Metals and Acides from Stainless Steel Pickling Baths,

Proc Int Symp Hydrometallurgy,- Chicago 1973.