Recovery of polishing acid

K.-P.Martinek, T.Schulz

With its long-standing tradition dating back to 1834, the F. X. Nachtmann Company ranges among the leading manufacturers of lead crystal and crystal glass.

The situation

An important process in the lead-crystal manufacturing business is the chemical mirror finishing taking place after the glass has been diamond ground. The process uses a mixture of sulphuric acid (65 weight percent) and hydrofluoric acid (1.5 weight percent). The chemical machining not only leads to defined substrate surfaces but also has a positive influence on the corrosion resistance of the glass. Thus, chemically polished glasses tend to be much better suited to treatment in a rinsing machine.

Acid polishing is a multi-stage process, with a two-step pretreatment (cleaning, degreasing) followed by the actual polishing in a polishing bath. Subsequently, the glasses are washed in a three-stage rinsing cascade.

The task

The goal was to close chemical substance circuits as part of measures designed to optimize the plant. An important step here is the reprocessing of used acids and rinse waters with a view to recovering the sulphuric acid contained in them. However, the sulphuric acid can only be used for the regeneration of the polishing bath if a minimum concentration of 85 weight percent is achieved.

The benefits

Such a recovery allows wastewater treatment (neutralization) to be cut by up to 90 %. This clearly means substantially less environmental pollution and a corresponding reduction in costs, as less sulphuric acid – the raw material required for regenerating the process solution – needs to be purchased.

The solution

The reprocessing requirements can only be met by an evaporating plant that is both efficient and corrosion-resistant.

Schulz + Partner GmbH developed a steam-heated forced-circulation evaporator that satisfies these requirements.

Evaporation must take place under vacuum, as sulphuric acid at the required concentration would have a boiling point of over 230°C under normal atmospheric pressure, while the appropriate plant materials can only be used for a temperature of approx. 140°C at a maximum.

Description of the process:

The required vacuum is generated by a vacuum pump with a level-controlled cooling circuit and is controlled by a vacuum meter. The process solutions to be treated are sucked into the concentration stage by a vacuum of 30 mbar (A) generated by the vacuum system. A circulation pump then transfers the medium into the evaporator by way of an external heat exchanger. In the evaporator, the heated solution expands, with part of it evaporating at a boiling temperature of approximately 135°C (see Fig. 1). Subsequently, the steam is liquefied in a separate condensation stage and is then discharged from the evaporator by a condensate pump on a level-controlled basis.

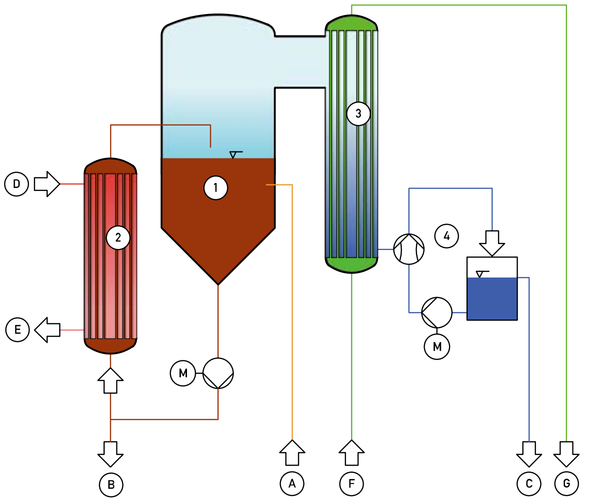

Fig. 1: A supply **B** concentrate **C** distillate
D/E steam/condensate **F/G** cooling water **1** flash evaporator
2 heater **3** condenser **4** vacuum system

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The required degree of concentration (85 weight percent) is monitored radio metrically. A pump discharges the concentrate on a discontinuous basis.
A gas-heated steam generator supplies the plant with the necessary heating energy. The cooling water required for condensation is generated by a cold-water aggregate delivered with the plant.

The plant runs automatically and is controlled via a stored-program controller (SPC) allowing process visualization. All operating data are stored and shown on a graphic display.
Owing to the high chemical aggressiveness of all of the solutions used in the plant, special materials had to be used in its construction. The entire heat exchanger is made of graphite, and all of the plant sections that come in contact with the product feature a PTFE/PFA coating. Selecting the appropriate process measuring and control technology was particularly difficult because such high temperatures are involved.
Figure 2 shows the upper part of the plant. The circulation pump, the concentrate pump and the radiometric measuring equipment are installed in the basement of the building (approx. 5 m lower).

Fig. 2

The following table shows the weight assessment of the evaporating plant:

	Unit	Feed	Concentrate	Distillate
Density	kg/l	1.32	1.78	1.00
H_2SO_4 concentration	weight %	41.5	84.5	max. 3
H_2SO_4 concentration	g/l	550	1500	max. 31
Volume flow	l/h	210	76	134

Summary

The plant has been in operation since early 2000. It ensures a closed circuit for most of the chemical substances used in the acid polishing process.
The high concentration of the product and especially the high corrosion requirements call for a customized technology posing special engineering demands.
The desired savings in wastewater treatment, together with a substantial reduction in the resulting quantity of waste (neutralization gypsum), ensure a very fast payback.