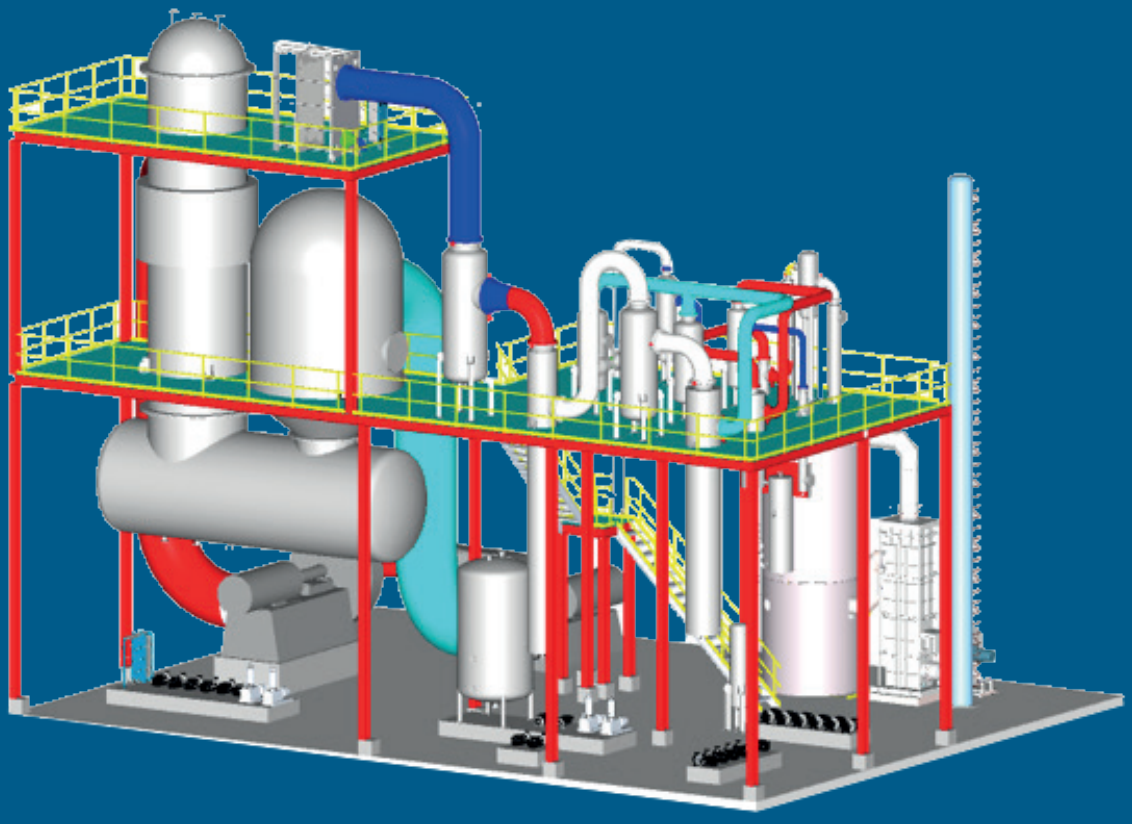
WILK-GRAPHITE
EQUIPMENT FOR THE CPI

ExtrAcid®

SULFURIC ACID CONCENTRATION/ DEWATERING PROCESS SYSTEMS



WILK GRAPHITE

Wilk-Graphite focuses on equipment and processes related to corrosive applications in relevant industries like Chemistry, Metallurgy, Pharma, Mining and Batteries. For more than 30 years we have been gaining experience by using materials like Graphite, Silicone Carbide (SIC) and PTFE and installing many thousand pieces of equipment. We developed process technology which have been acknowledged by the market as innovative and cost saving.

Wilk-Graphite is located in Lörrach (Germany) with offices and workshops in Germany, France, USA, Korea and China.

PROCESS TECHNOLOGY

- ExtrAcid® is the first system that recycles waste sulfuric acid under pressure using an innovative patented loop system producing high concentrated acid and process steam at the same time
- ExtrAkali® reduces Capex and Opex for Caustic Soda concentration. Saving 25 to 35% energy in 4 steps evaporation and using NoMoCorr® SIC block heat exchanger for 98% steps with long live time and sharply reduced maintenance cost. Shared development with Chem Eng.
- HCL Synthesis units and systems from Nantong Sunshine
- HCL purification systems from Nantong Sunshine

ASKR AND RDR

- Stirrer free crystallizer and reactor that-produces up to 3 times larger crystals. Limited residence time distribution provides homogenous products.
- Batch or continuous operation

MATERIALS AGAINST CORROSION

For more than 30 years the Team works successfully with many thousands references

- Carbex® Graphite
- NoMoCorr® Silicone Carbide (SSIC)
- PTFE

For universal chemical resistance in severe surroundings up to 450°C and 40 barg.

EQUIPMENTS

Equipment	Graphite	Silicone Carbide	PTFE
Heat Exchanger	X	X	
Columns	X	X	X
Internals	X	X	X
Compensator		X	X
Pipe systems		X	X
Reactors	X	X	X

SCANVEX SAFETY SHIELDS

- Safety Tape
- Safety Shield
- Safety Ring

ExtrAcid®

SULFURIC ACID CONCENTRATION/ DEWATERING PROCESS SYSTEMS

In one Sentence:
ExtrAcid® is the first system that recycles waste sulfuric acid under pressure using innovative patented loop systems producing high concentrated acid and process steam at the same time.

INTRODUCTION

Waste sulfuric acid from various chemical processes has to be recycled by removing water and other impurities from the acid. By dewatering the concentration rides from less than 15% to higher values of typically 98%.

Typical Acid concentration systems (SAC or SAS or dewatering systems) are separated in to lower concentration of up to 70%, medium and high concentration systems.

BENEFITS AT A GLANCE

- Energy recovery by steam production
- Innovative patented 2 loops design
- Flexible inlet concentration
- From 5 to 98%
- Handles impurities
- Small footprint
- Optimized capex and opex

APPLICATIONS FOR ExtrAcid®

- Titanium Dioxide
- Gas drying/purification
- Nitration processes
- Mining
- High purity acids

PATENTED DESIGN

The design of the ExtrAcid® system is patented covering the process including the loops for steam production and the usage of SIC material in the required pressure and temperature ranges.

SYSTEM WORKS FOR ALKALINE, HF OF HCL AS WELL

TURN ACID WATER INTO PROCESS STEAM

Well-known is that recycling by vacuum evaporation of water consumes high energy resources. Although the energy consumption has been optimized in recent years valuable energy cannot be recovered due to low temperature levels.

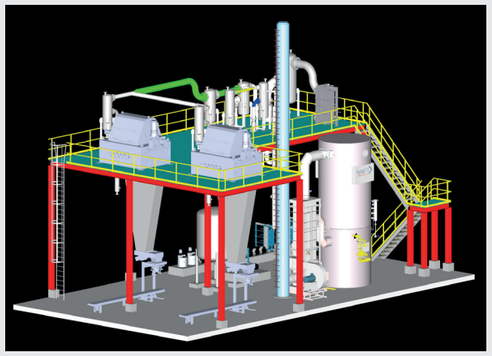
The innovative ExtrAcid® process system is the first that can significantly reduce the energy consumption by producing valuable process steam.

IMPURITIES

As waste sulfuric acid has many different sources it often contains organic or inorganic impurities other than water which have to be removed. Dependent on the nature of the process typical examples are HF, H_2O_2 or Fe.

It is important to know the kind and amount of the impurities before the design of the ExtrAcid® system. Some organics like H_2O_2 are decomposed into water and O_2 if the temperature is increased sufficiently. Others like HF stay in the vapor phase and can be separated this way. Some need special treatment like purification by crystallization.

ExtrAcid® can offer many solutions to this wide range of challenges. Due to the universal chemical resistance of our main material, it is suitable for HF applications, due to high temperatures it burns many organics into its elements or separates by evaporation.



PURIFICATION BY CRYSTALLISATION

A usual task for the treatment of waste acid coming from the production of Titanium Dioxide is the Fe load in the waste. It can be reduced by forming Ferrous sulphate Monohydrate. These kind of processes can easily be added to the ExtrAcid® system.

NoMoCorr® – INNOVATIVE SILICONE CARBIDE MATERIAL

Extracid is the first system with extensive usage of SSIC at high temperatures and pressures. Without SSIC block heat exchangers conventional systems have to use available materials such as Tantalum, graphite or glasslining. The limitation is the reason why the multistage systems operate under vacuum. Vacuum reduces the temperatures, however the equipment gets large and expensive.

Silicon carbide solves the problem and allows high temperatures reaching the boiling point of H_2SO_4 even under pressure. It is fully resistant against all concentrations and temperatures and in addition even to impurities like HF.

SIC is used as a material of choice for the following components for high temperatures. Most of them are innovatively designed in detail or generally by Wilk-Graphite for the use in severe applications.

- Heat exchanger up to 450 °C and 40 barg
- Separators
- Pipes
- Compensator
- Thermowells
- Columns

SIC – THE UNIVERSAL ANTI CORROSION MATERIAL

- Universal Chemical resistance
- Highest thermal conductivity
- No abrasion
- No contamination
- Extremely hard ceramic
- Low wall thickness
- Low fouling rates
- High velocities
- High thermal shock resistance
- Many designs possible
- High temperature resistance
- Low surface roughness
- No aging / no fatigue

KEY PROPERTIES

- Density > 3 120 kg/m³
- Thermal conductivity: 130 W/(m.K)
- Vickers hardness: 19,2 GPa (500g load) > Tensile Strength: 210 MPa
- Young's modulus: 420 GPa

SIC BLOCK HEAT EXCHANGER

Block Heat exchanger with blocks made of NoMoCorr® Silicone carbide have been developed matching the market demand for a universally resistant, robust heat exchanger. It has no limitations coming with other materials or designs.

The block technology for heat exchanger is well-known since decades by units made of graphite blocks. It is a simple, easy to build and maintain structure which now has been adopted for blocks made of SIC. NoMoCorr SIC blocks are made of one very hard piece with various different drilling sizes following the latest studies on SIC design.

NoMoCorr SIC block heat exchanger combine the benefits of the material for chemical resistance bursting the limitations known before in temperatures and pressures, lifetime and economics.

Our heat exchanger are made in France under stricked quality control and have been used since the beginning in those applications where other materials and constructions fail. At the same time they are competitive in Capex and Opex to most of the systems known saving money and keeping away troubles.

APPLICATIONS

- HF in all temperatures and concentrations
- H_2SO_4 all concentrations up to the boiling point and above
- Stainless steel pickling lines
- Multi purpose plants
- Acid recovery
- Caustic Soda

SIC (SILICONE CARBIDE) BLOCK HEAT EXCHANGER

- temperature and pressures (40 barg 450 °C)
- very high thermal conductivity
- no contamination
- no abrasion
- low fouling
- innovative baffle system
- universal chemical resistance
- Modular design
- SIC heads
- Low number of flat gaskets
- no aging / no fatigue

LIMITATIONS

- Block Ø < 300 mm
- Block height < 320 mm
- Area up to 60 m²
- Design pressure < 40 barg
- Temperature < 450 °C
- Hole diameter 8 / 12 mm

PROCESS DESCRIPTION

INNOVATIVE LOOPS

The ExtrAcid® system has 2 innovative patented loops. Function of these loops is to remove the water from the system even when is partly loaded with gaseous acid contents.

Based on fundamental physical properties the amount of H_2SO_4 in the vapor after the evaporation increases with higher concentrations. In the loops the H_2SO_4 loaded vapor is separated by partial condensation into the process steam (water) and acid. While the acid is getting back into the process steam.

By using the loops system the process is much easier to handle as conventional columns are avoided and replaced by condensation and mixer. The system is getting smaller and requires much less equipment.

TECHNICAL DATA

- Flow rates from 2 up to 200 MPTD SA97
- Size of 200 MTPD: 2,4 m x 2,7 x 12 m height skid mounted
- Inlet concentration: 5 to 50 %
- Outlet concentration: 50 to 98%
- Organics: mostly decomposed at high evaporation temperatures

PRECONCENTRATION

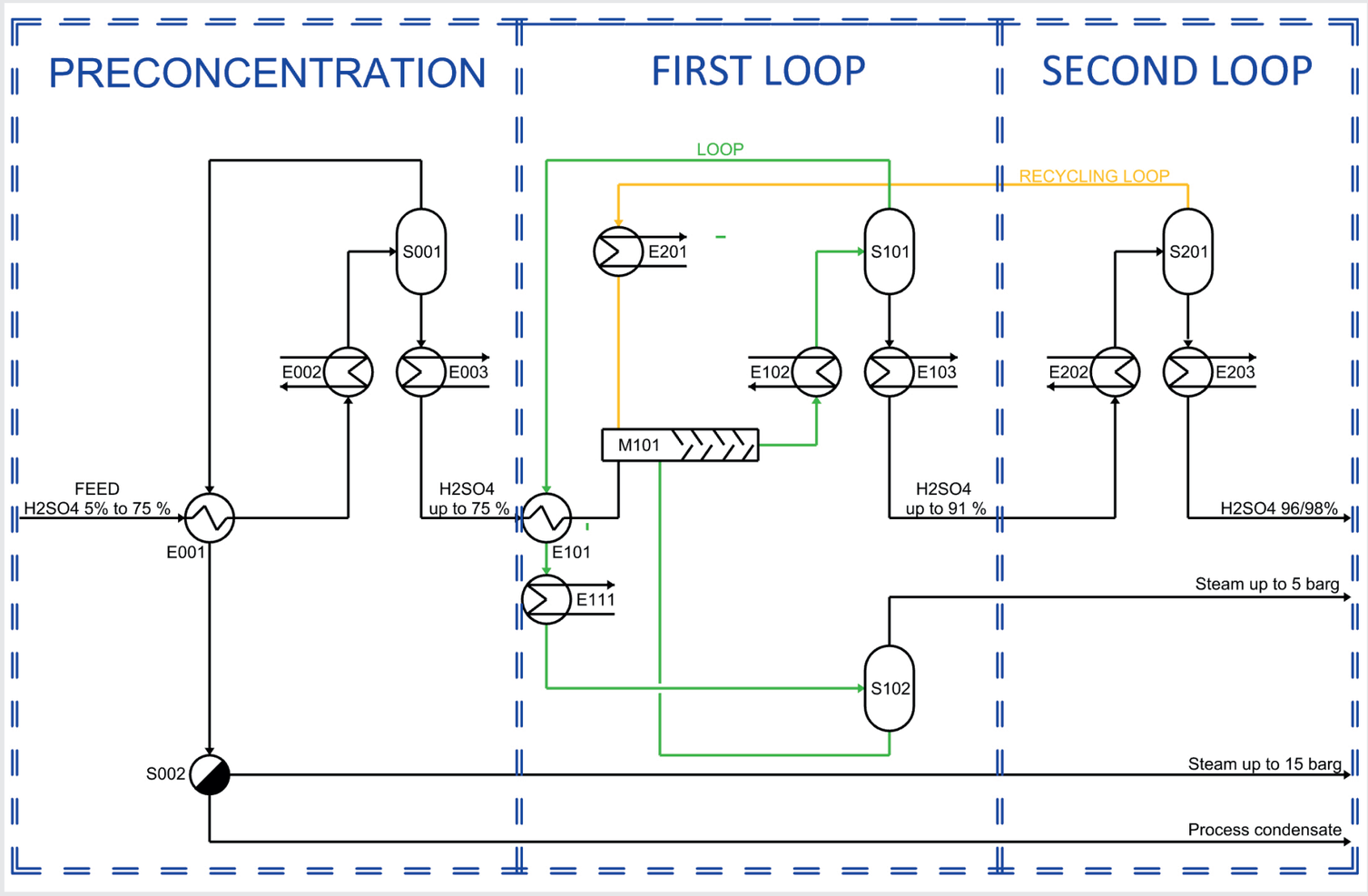
The feed of H2SO4 is preheated in an interchanger (E001) that recovers the energy from the vapor generated in the vaporizer (E002). On the PFD, it is for 1 effect, but we can add up to 5 stages that would look similar to recover the energy directly inside the ExtrAcid®. In this case the steam from the 1st loop and the 2nd is reused in the preconcentration.

FIRST LOOP

The acid goes into a preheater (E101) that also condense the acid from the loop to then reinject it as liquid in the mixer (M101). The steam is under pressure and can be reused. The liquid from the mixer goes to a vaporizer (E102) to be concentrated and separated in the gas / liquid separator (S101). The concentrated acid up to SA91% is then cooled to avoid flashing when reducing the pressure to atmospheric pressure.

SECOND LOOP

The acid from the first loop is directly vaporized in the vaporizer (E202) and again separated (S201). The obtained liquid is the final product while the vapor is fully condensed (E201) and recirculated in the first loop to be treated.



RETURN ON INVESTMENT

Today the recycling of waste acid is to be regarded as a investment in clean environment. The energy for the dewatering has to be paid for and it is not possible to compensate for this amount by the value of the saved acid and potential cost for waste disposal. Today a positive ROI is also dependent on the fairly high cost for the vacuum system, which require much more equipment space and naturally high investment cost.

By creating steam and acid from cheap energy as power or gas the situation changes fundamentally. The system itself is small in size and in numbers of equipment. An easy tool provided by Wilk-Graphite just needs the customers individual cost and it is easy to calculate the TCO. The ROI can be less than a year dependent on the individual cost. After that the system generates money for the customer.

With each design we deliver an excel sheet which allows our customer to fill their individual cost for items like energy or waste acid treatment. These data are then used to calculate the opex no capex of the plant offered. Resulting is a ROI number as well as annual operational cost, which can be regarded as a guideline.

OPEX AND CAPEX

Like all systems which evaporate water from acids energy has to be added to the system. While all other systems cannot use the energy after the usage, ExtraAcid® provides steam.

Taken from a project to concentrate H_2SO_4 from 25% to 85% the opex shows signifcantly positive numbers: Based on the data on the opposite site significant saving can be realized.

STEAM SELF USUAGE

In some application customers may not be able to use the steam which is provided by the system. In such a case the steam can be used in a 4 stages precentration system, which recovers 75% of the steam directly in the system.

Based on the data of the opposite page 1650 KW of the steam of 2210 KW can be reused and the overall cosumption is 1.495 KW. Still 20% less than todays systems.

COMPARISON TO VACCUM PROCESSES

Vacuum systems for the concentration of acid use multistage evaporation under vacuum. They need vacuum to be able to handle the required temperature of boiling sulfuric acid with conventional materials like graphite, quartz or tantalum.

The evaporation usually uses steam and does so efficiently by multistage evaporation. Industrial prices for steam compared to natural gas or electric power show significant difference resulting in higher operating cost.

As up to 80% of the energy used by the system can be regained in form of steam the Opex may even get positive with an input of electricity or natural gas and an output of concentrated acid and steam.

	ExtraAcid®	Vacuum
Pressure	up to 10 bar	Vacuum
Heating by	Thermooil	Steam
Footprint	small	large
Number of Equipment	small	high
Maintenance	low	high due to choice of material
Opex	low	high due to steam
Capex	low	high due to much equipment

COMPARISON OF A SYSTEM FOR 5.500 KG/H OF ACID CONCENTRATION FROM 30% TO 85%

SIC SYSTEM SCU – SA85

- Inputs (3400 kW)
  - > 5500 kg/h of H_2SO_4 30% at 30 °C
  - > 400000 kg/h of thermal oil at 300 °C **(3150 kW)**
  - > 34500 kg/h Cooling water at 32 °C
  - > System composition
  - > 3 SIC heat exchangers – Total = 50 m²
  - > 1 SIC Gas / Liquid separator
  - > 1 PTFE Gas / Liquid separator
  - > 20 m of piping
  - > Few valves and instruments
  - > 2 PFA Pumps for H_2SO_4 30-35%
- Outputs
  - > 1941 kg/h of H_2SO_4 85% at 50 °C
  - > 3600 kg/h of Steam at 5 bar **(2210 kW)**
- **Energy recovery ratio: 70%**
- **Net Energy consumption (without Cooling water)**
  - > 940,00 KW
- Total water consumption
  - > 34.500 kg/h

MULTISTAGE SYSTEM

- Inputs (1.850 kW)
  - > 5500 kg/h of H2SO4 30% at 30 °C
  - > 3250 kg/h Steam 10 bar **(1850 kW)**
  - > 235000 kg/h Cooling water at 32 °C
  - > System composition
  - > 3 Graphite heat exchangers – Total = 860 m²
  - > 5 SIC Heat exchangers – Total = 100 m²
  - > 10 Process vessels and tanks
  - > 2 Utilities tanks
  - > 2 Hydraulic vacuum unit
  - > Over 100 m of piping
  - > 2 Pumps for the acid
  - > Countless valves and instruments
- Outputs
  - > 1941 kg/h of H_2SO_4 85% at 50 °C
  - > 85 m³/h of exhaust duty
- **Energy recovery ratio: 0%**
- **Savings**
  - > 1.850,00 KW 49%
  - > 235.000 kg/h 85%

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