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Separation of PCE-containing exhaust air by VOC concentrator and activated carbon

A practical example of large-scale industrialization

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Separation of PCE-containing exhaust air by VOC concentrator and activated carbon

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Outline

- Initial situation
- Characterization
- Process selection
- Plant concept
- Experience
- Conclusion

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Initial Situation

- Coating process using tetrachloroethene / perchloroethylene (PCE)
- Tightening of environmental regulations
- No substitution possible
- Design process data:
  - Raw gas volume flow: 8,000 Nm^3/h
  - Raw gas temperature: 20 to 35 °C
  - Humidity: 5.8 - 10.6 g_{H2O}/kg_{air}
  - Ø Concentration: ~ 4 ppm = 31 g_{PER}/Nm^3
  - Concentration peaks: ~ 20 ppm = 148 g_{PER}/Nm^3
  - Operating time: 8,500 h/a
- Legal requirements for exhaust air purification:
  - Classification TA Luft: Organic substances, Class 1 (5.2.5)
  - Mass flow: 100 g PER/h
  - Concentration: 20 mg PER/Nm^3

Characterization

- Properties / Substance characterization of PCE
  - CAS No.: 127-18-4
  - Molecular formula: C2Cl4
  - Substance group: halogenated hydrocarbon, aliphatic, unsaturated
  - Characterization: non-flammable, photosensitive, highly volatile
  - Solubility in water: sparingly soluble
  - Molar mass: 165 g/mol
  - Boiling point: 121°C
  - Ignition temperature: > 650°C / T1
  - Decomposition temperature: **≥ 140°C**
  - Decomposition products: Hydrogen chloride, chlorine, **phosgene**

Source: Gestis substance database, retrieved 8/4/21, https://gestis.dguv.de/data?name=013680

Process selection

- Process engineering preselection

Process	Suitability	Invest	Operating Costs	Space/Weight
Zeolite Concentrator	→	↑	↑	↑
Low temperature condensation	↓			
Activated carbon *(single stage)*	↑	→	↓	↓
Gas scubber *(opted out by c.)*	→	↑	↑	→
Thermal destruction	↓			

- Cleaning (adsorption) with activated carbon is state of the art, but causes high operating costs when implemented as single stage
- Preferred solution: Combination of Concentrator + Activated carbon
- Risk mitigation necessary with respect to **phosgene formation**

Process selection

- Literature search revealed no robust and even contradictory data
- Decision for experimental validation
- Objectives
  - Check of thermal stability of PCE
  - Temperature-dependency of PCE decomposition
  - Material compatibility of PCE with V4A (1.4571)
- Test setup and Equipment:
  - Samples: 4 g PCE/m^3 + 18 g water /m^3
  - Temperatures: 140 / 160 / 180 °C
  - Equipment: horizontal high-temperature furnace
    magnetic levitation scale
    thermogravimetric analysis (TGA)

Process selection

- Results of laboratory tests on thermal stability /material compatibility

Temperature	Chloride content [mg]	hypochloride content [mg]
Blank value	0,05	n.n.
140°C	0,03	n.n.
160°C	0,03	n.n.
180°C	0,04	n.n.

n.n. = not detectable (below detection limit)

  - Chloride contents in the same order of magnitude as 0.1 molar NaOH solution
  - **PCE does not age at contact times of approx. 30 sec!**
  - **Corrosive hydrogen chloride (HCl), chlorine gas (Cl_2) or phosgene ($COCl_2$) not formed!**
  - No optical changes to V4A material, no mass change detectable via TGA

Plant concept

- Two stage design: Zeolite concentrator followed by activated carbon plant
- 1st process stage: Concentrator
  - Raw gas containing PCE is purified by adsorption with zeolite rotor
  - Adsorber is regenerated with fresh air to produce the concentrate
  - Concentrate stream is fed to activated carbon plant
- 2nd process stage: Activated carbon plant
  - PCE-containing concentrate is purified by adsorption on activated carbon
  - Regeneration (desorption) of activated carbon with saturated steam
  - Separation of PCE/water mixture in two phase decanter

- Due to the concentrator, the concept leads to a compact plant design with reasonable operating costs at low risk!

Plant concept

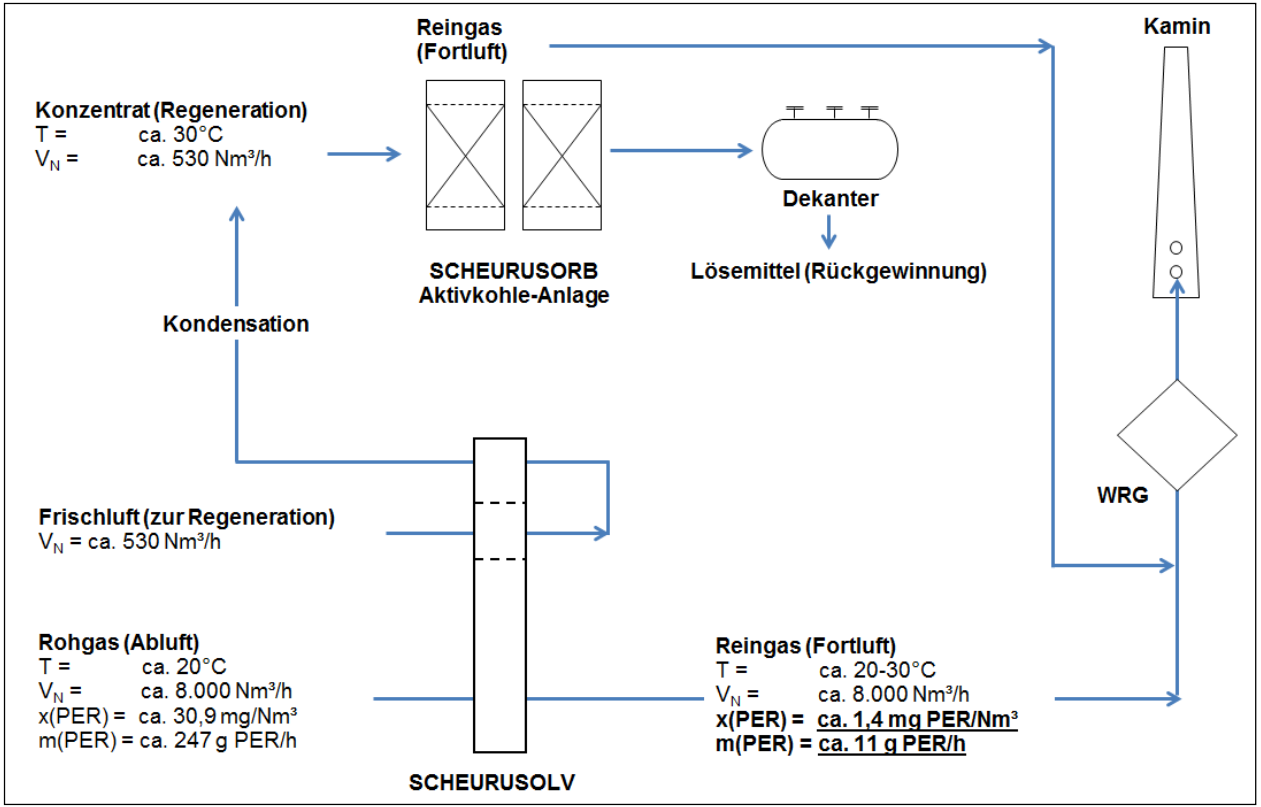
- Dimensioning of activated carbon plant / service life adsorbens

Parameter	Value	Unit
Activated carbon mass	460	kg
Loading capacity	30	%
PER adsorption mass	138	kg
PER mass flow	0,23	kg h^{-1}
Adsorption time	600	h

- Theoretical service life approx. 600 h per column
- Approx. 15 desorption cycles per year estimated

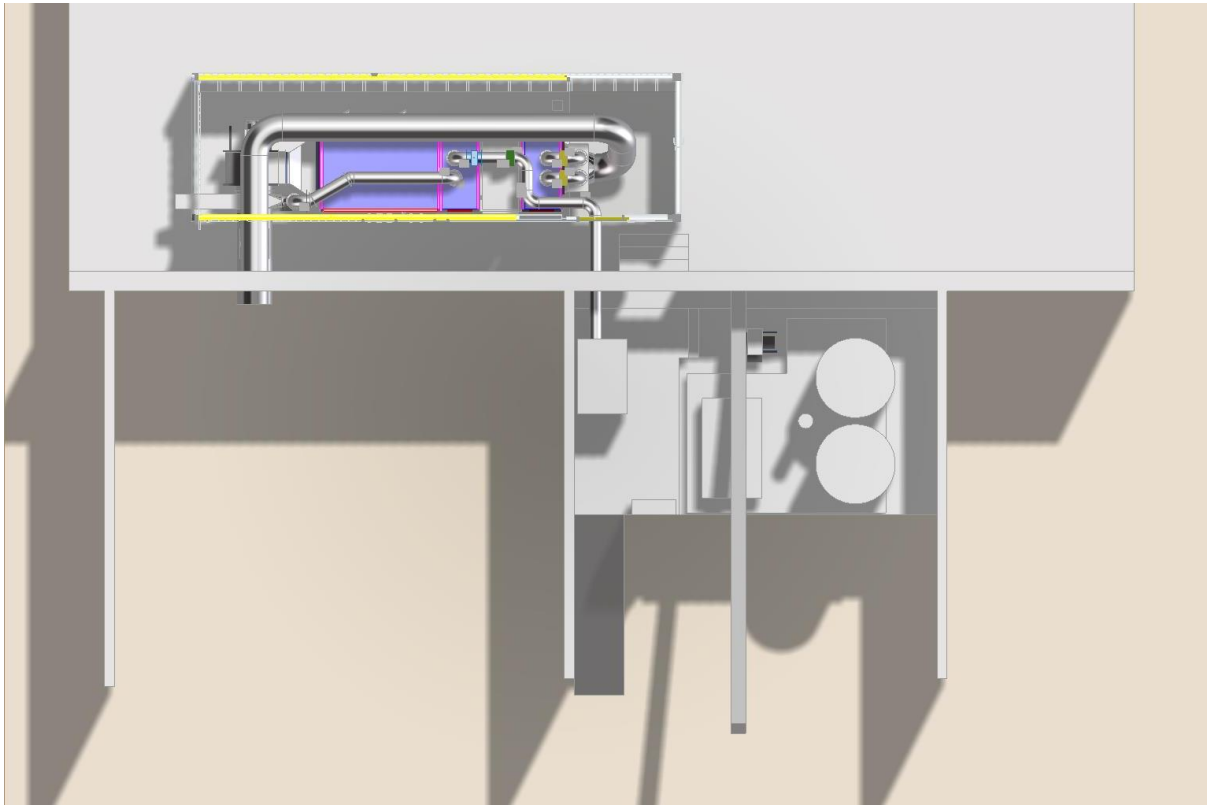
Plant concept

- Mass balance



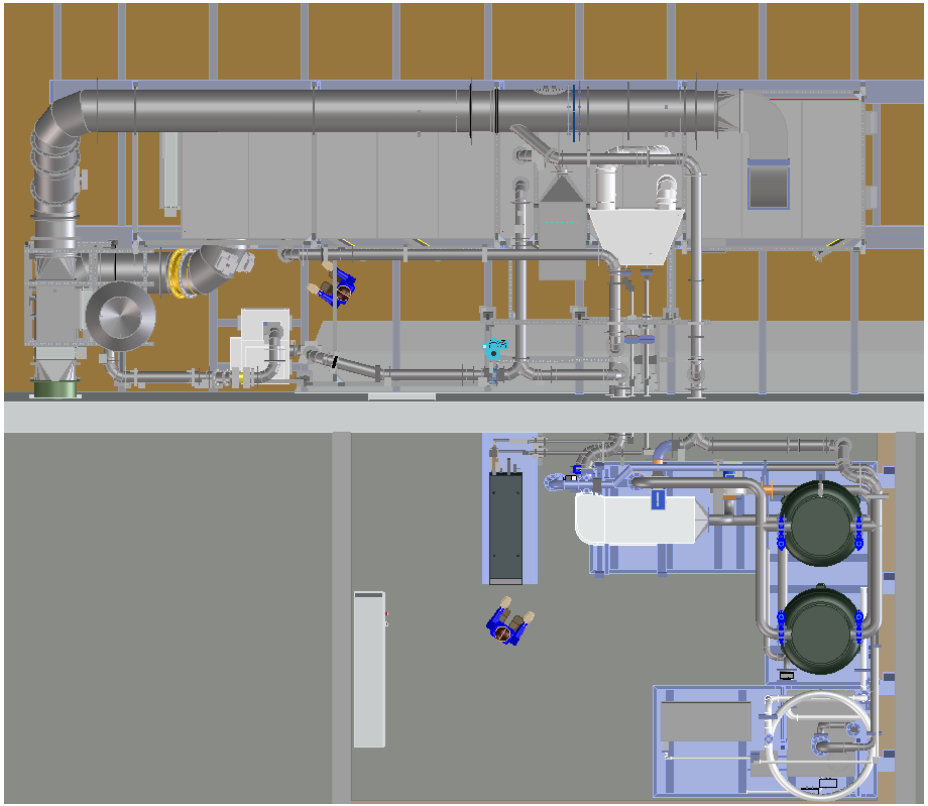
Plant concept

- Layout



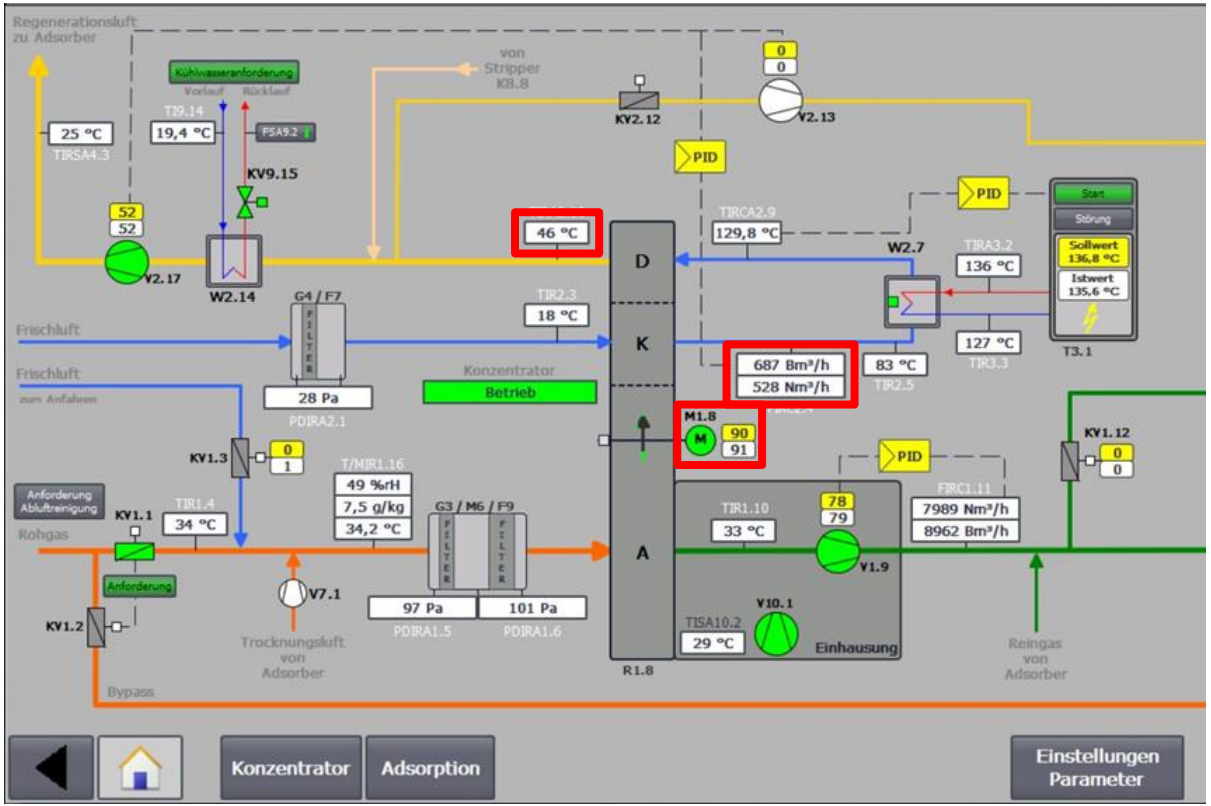
Plant concept

- Layout



Experience

- Plant settings - concentrator



Experience

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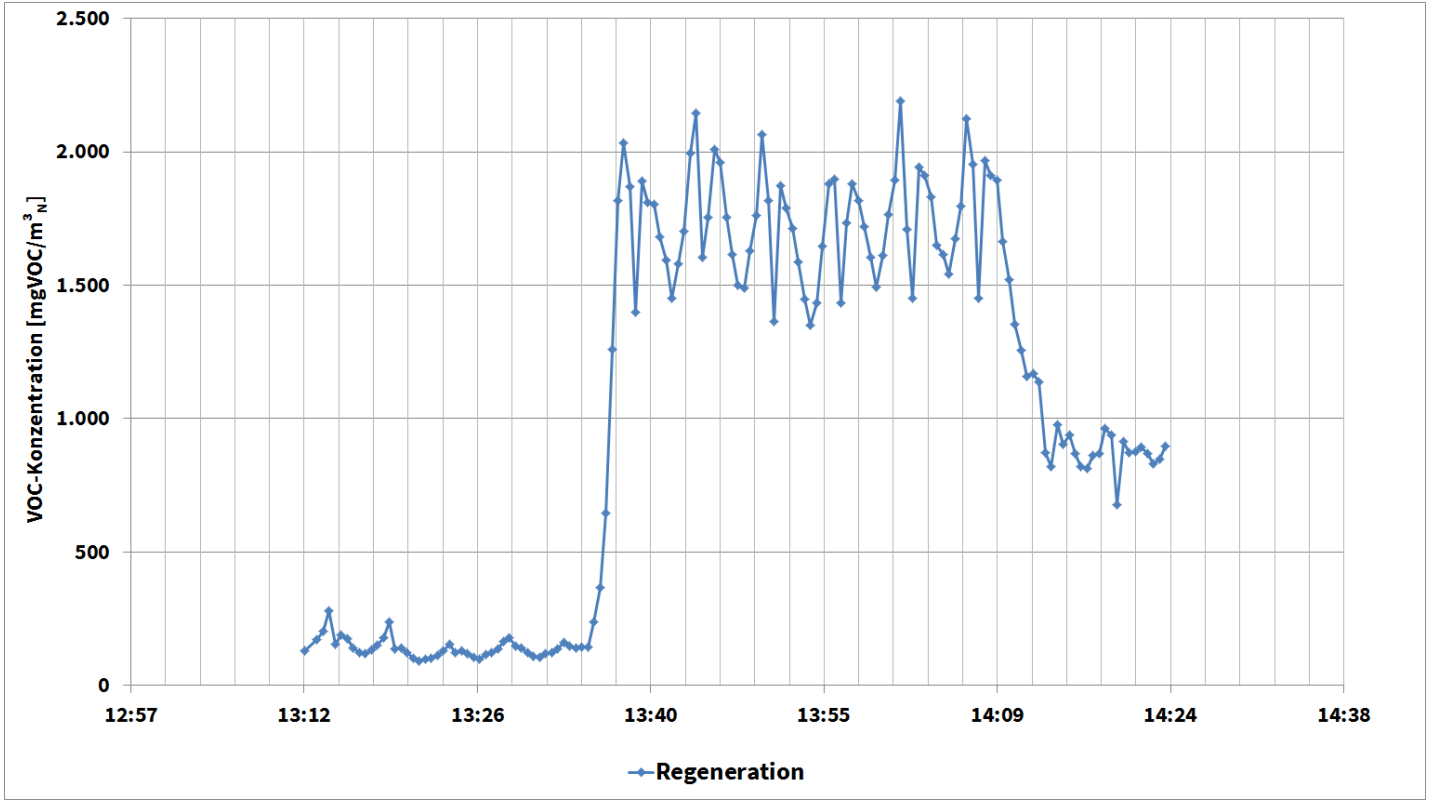
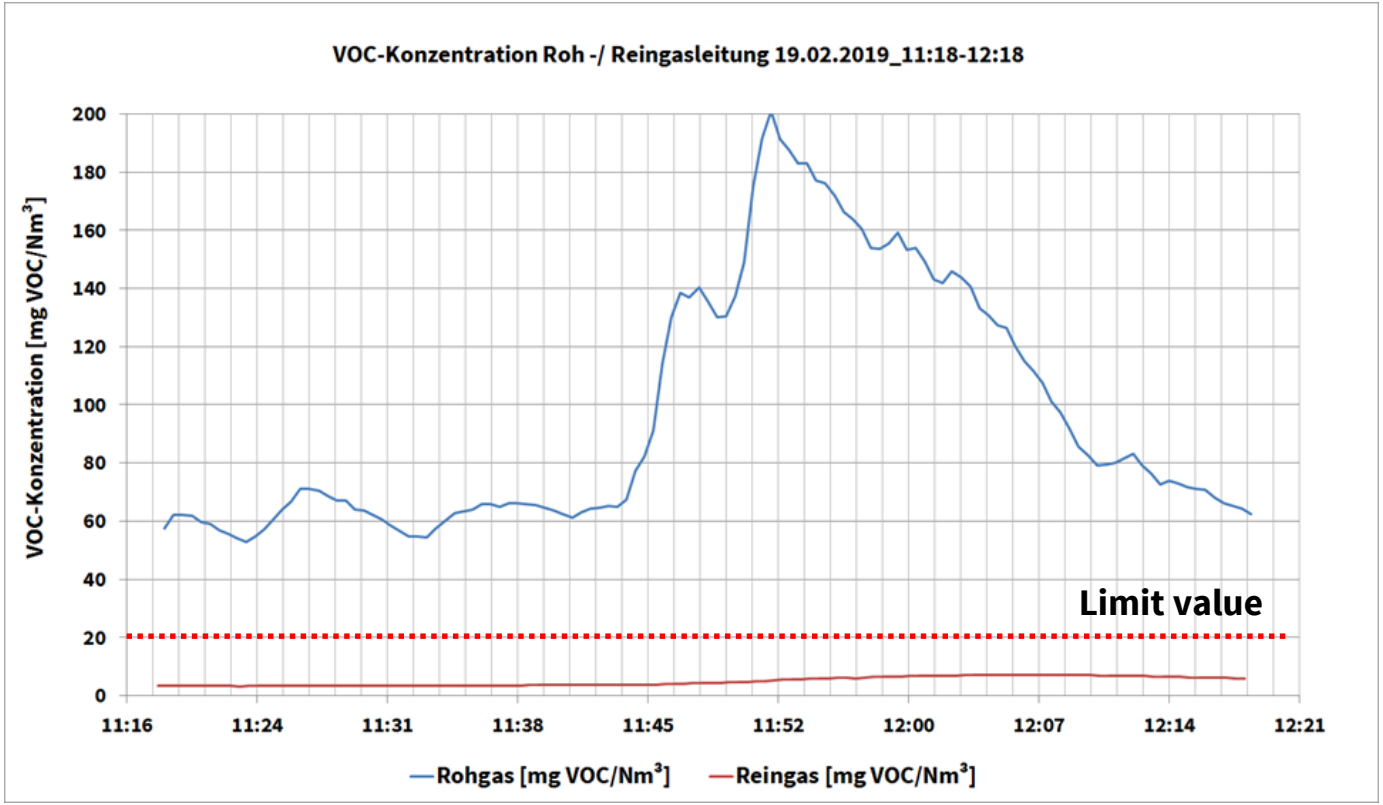


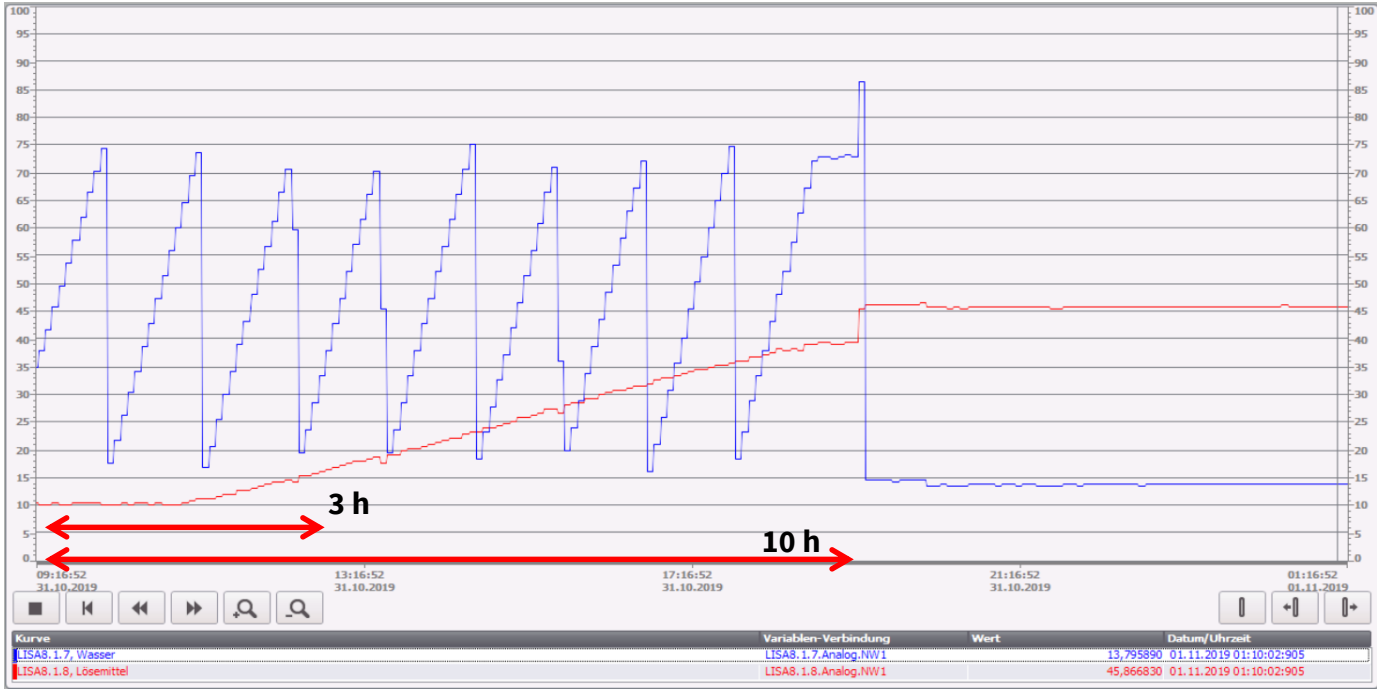
Experience

- Cleaning performance - FID measurements



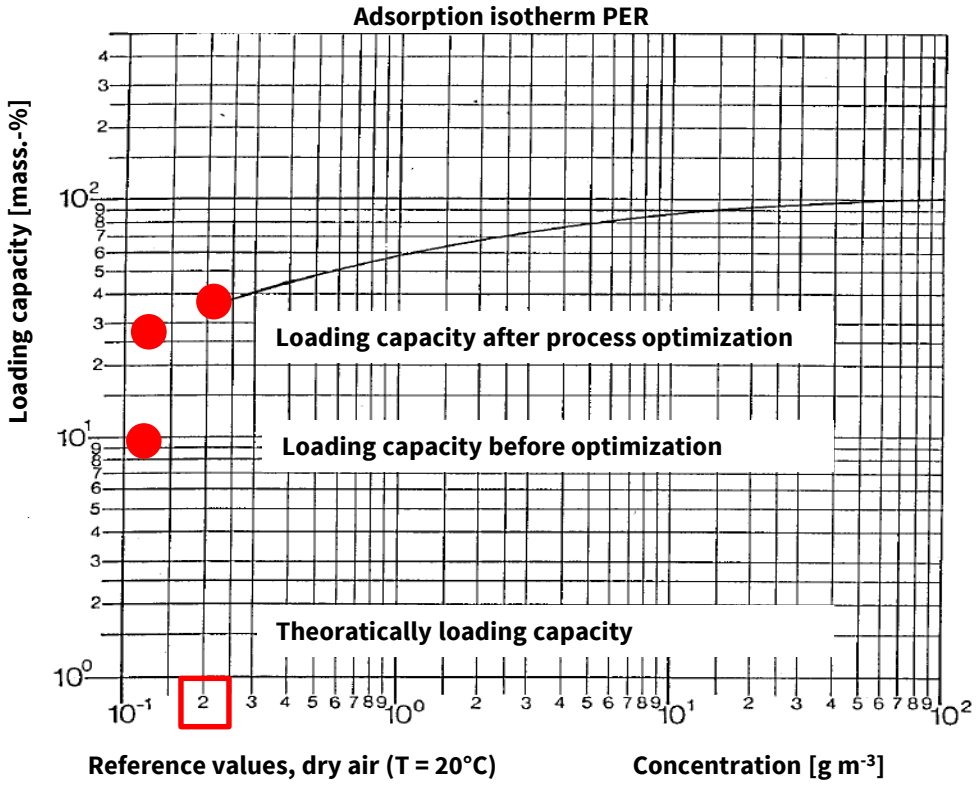
Experience

- Desorption of activated carbon unit – two phases decanter



Experience

- Adsorption isotherme for PCE and achieved loading



Experience

- Adsorption/desorption behaviour of concentrator extremely dependent on process parameters
  - **Rotation speed of zeolite rotor!**
  - **Concentrator desorption works with 140 °C**

- Adsorption/desorption behaviour of activated carbon plant much slower than expected
  - **Duration of desorption vs. loading capacity/cycle time per column (600 h)**

- Practical adsorption capacity fits perfect with expectations

Conclusion

- Design approach
  - First time to use zeolite concentrator with PCE
  - Total economic optimum achieved by innovative process combination

- Performance and stability
  - Trouble-free plant operation since 2019 only PCE flow meter needed upgrade
  - No corrosion problems detected so far
  - Perfect fit with all regulatory requirements

Many thanks for your attention

In case of further questions please don‘t hesitate to contact us

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