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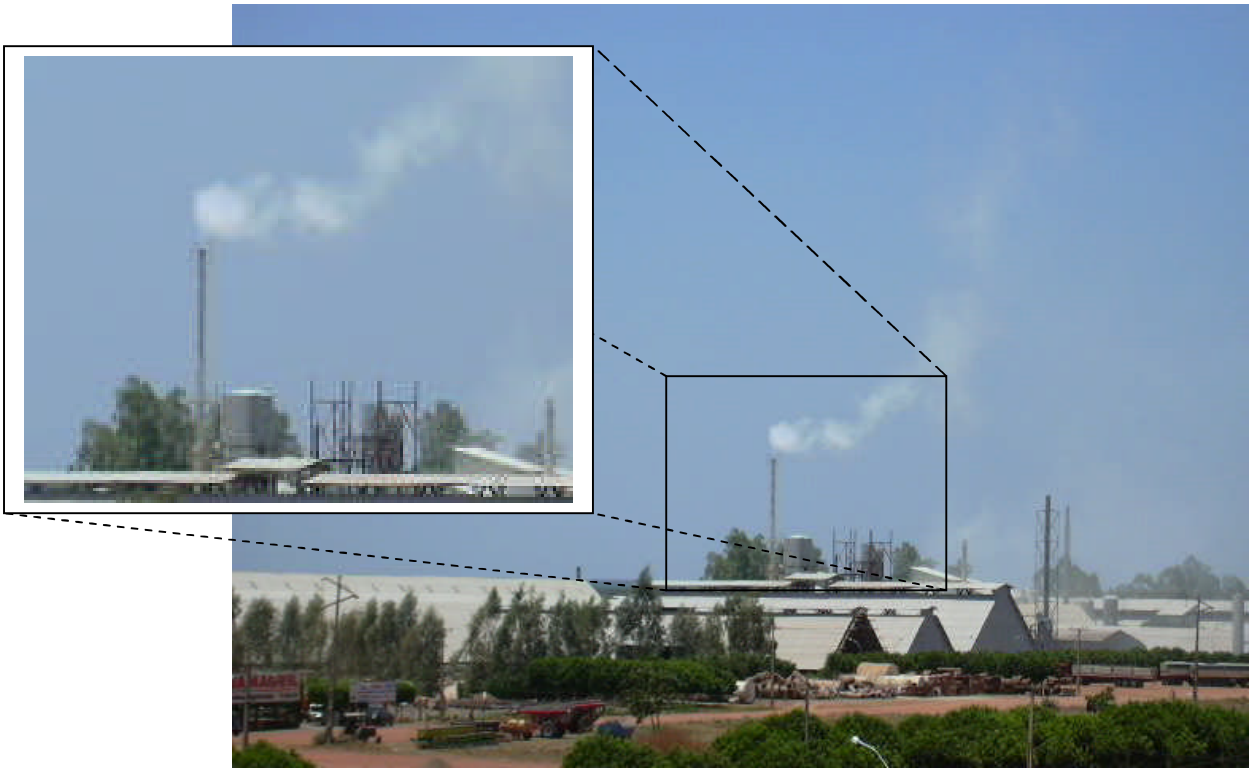

Mist Eliminator Troubleshooting – Sulphuric Acid Service

To establish the cause of operating problems with mist eliminators, there are ideally two things to do:

- Inspect the filters
- Review the operating data and visual evidence , i.e.
  - pressure loss,
  - liquid drainage from the filters,
  - liquid drainage after the filters or stack exit appearance,
  - evidence of product contamination downstream or process affected,
  - sight glass check,
  - operation change such as gas flow increase or decrease
  - operation change such as temperature or oleum production variation
  - upstream liquid distribution performance
  - stick test or sampling measurement (see Stick Test Guide)

Note : It is often as important to know the rate and timing of changes as to know the amount of change.

It is not unusual for a combination of factors to camouflage the real cause and so the more information available, the better.

Apparent emission problems or changes of efficiency are associated with filter failure in most operators minds, but in many cases it can actually be a process problem that is giving the filters an abnormal duty, not causing, or caused by, any actual failure.

The first questions we will ask are :

- Has the pressure loss increased, quickly or slowly
- Has the pressure loss decreased, quickly or slowly
- Has there been evidence of droplets (e.g. no visual problem, but spitting from the stack, or sight glass / stick test experience)
- Has there been evidence of mist (e.g. plume from stack, or sight glass experience)
- What process changes or problems have happened

With some or all the answers we can look to narrow down the possibilities.

CAUSE	***REASON***	***RESULT***
High gas velocity	*Increase in plant throughput*	*Reentrainment of droplets & prorata Δp increase*
	Fibre blocked by solids or salts	*Reentrainment of droplets & big Δp increase*
Low gas velocity	*Capacity downturn*	*Poor collection of mist due to low saturation of fibre bed, so stack plume and lower than pro rata Δp*
High inlet liquid loading	*Liquid distribution problem or steam leak or process upset e.g.temperature or volume*	*Reentrainment of droplets & big Δp increase & higher liquid drainage*
Low inlet liquid loading	*Liquid distribution failure or process upset e.g.temper -ature or volume*	*Poor collection of mist due to low saturation of fibre bed, so visible mist and lower than pro rata Δp*
Gas by-pass of gasket	*Poor fitting / seal or gasket failure*	*Mist in small quantity*
Gas by-pass of drainpot	*Drainpot corrosion or not properly filled*	*Obvious mist and droplets & lower Δp*
Gas by-pass of fibre bed	*Corrosion or erosion of fibre or fibre bed collapse due to excessive liquid loading*	*Very obvious mist and droplets & much lower Δp*

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CAUSE	***REASON***	***RESULT***
Increased temperature	*Gas not properly cooled or vapourising H2O*	*Exit plume as H2O (& ?) vapourises in air*
Decreased temperature	*Excess gas cooling or shock cooling in or before tower*	*High amounts of very fine, submicron mist, so visible in gas & higher load and Δp*
Poor upstream filtration	*Poor filter performance or design*	*Increase in amount of solids blockage*
Particulates	*Dust from ductwork or atmosphere or hydrocarbons. Salts build up.*	*Acts as a nucleus for fine mist formation High Δp.*

View through a sight glass below I.A.T. filters, showing normal inlet mist load

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NOx

Some H2SO4 plant processes (particularly Spent Acid Regeneration and Non-Ferrous Metals Smelter off-gas but sometimes mixed feedstock or poorly combusted sulphur burners) have problems with NOx in the Final Absorber.
These NOx levels can be measured in the gas, and in the acid.

Below is a summary of the main (progressive) reactions and impacts that NO_X can have generally or under certain conditions in Metallurgical or Spent Acid Re-generation plants.

Formation of NO in H_2SO_4 Plants

- NO is formed at high operating temperatures from nitrogen in the combustion air (usually >1300 °C). Therefore it can be found in oxygen-enriched smelting operations (Isasmelt, Outokumpu, Inco, El Teniente, etc.)

- NO is formed from nitrogen compounds in the feedstock. Therefore it depends on the (secondary) feed material (feedstock impurities, spent acid decomposition)

Formation of NO_2 and N_2O_3 in the Converter

The NO then forms NO_2 and N_2O_3 in the converter

NO nitrogen monoxide	+	0.5 O_2 oxygen	→→	NO_2 nitrogen dioxide
NO_2 nitrogen dioxide	+	NO nitrogen monoxide	→→	N_2O_3 dinitrogen trioxide

Formation of NO_2 and N_2O_3 in the Absorbers

The NO_x and N_2O_3 then forms $NOHSO_4$ in the absorber(s)

N_2O_3 dinitrogen trioxide	+	2 H_2SO_4 sulphuric acid	→→	2 $NOHSO_4$ + H_2O nitrosyl sulphuric acid & water

$NOHSO_4$ is known for a long time to form in the acid in Brownian Diffusion type candle filters, but it can :

- form / increase the amount of small aerosols
- decompose to NO & NO_2 above 50 °C
- form clear crystals, generally soluble above 73 °C
- solubilise in 98 % H_2SO_4: 60 g per 100 g solution at 20 °C
  (rising with temperature to 75 g at 70 °C)

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Formation of NO_x and $NOHSO_4$ in Candle Filters

- Aerosols of $NOHSO_4$ are retained in / generated from the candle filters, and due to the preferential absorption of the NOx, less absorption of any SO_3 slippage is possible.

- High vapour pressure of $NOHSO_4$ results in NO_x slippage from the fibre

- Typically 85 - 250 g $NOHSO_4$ per kg H_2SO_4 is dissolved
  (up to 400 g/kg ~ 40 % $NOHSO_4$)

- Crystallisation of supersaturated acid can cause blockage of the candle filters (and therefore high pressure losses) especially in fluctuating process duties and abnormally dry / low H_2O presence conditions.

Operating and Maintenance Suggestions :

- <u>To reduce a stack plume :</u>
  Try operating the absorbers with slightly lower strength acid, e.g. 97.5-98%. The lower strength would allow more absorbtion of <u>both</u> the NOx & $S0_3$ in the candles' acid, and be drained away, before the acid strength there became strong enough to begin fuming.

- <u>To reduce pressure loss problems due to Nitrosyl acid crystals blockage of filters :</u>
  - Operate with gas at tower top above 73˚C
  - Operate with slightly lower strength acid so more crystals can be dissolved
  - Install a wetting system to pre-wet candles at start-up to prevent crystals formation, and run the wetting system during a purge period to wash out residual NOx content, which otherwise would solidify during shutdown, when candle filters become dry after draining.
    (Please contact Begg Cousland for details of effective wetting systems.)

- <u>To clean affected candles :</u>
  Wash NOx poisoned filters with Sulphamic acid.

- <u>To reduce NOx contamination of product acid :</u>
  If not already done, arrange a separate drainage for the acid from the candle filters. For Standing type candle filter arrangements, this can be done by draining from the tubesheet out of the tower, through a liquid seal bend. For Hanging type candle filter arrangements, this can be done by connecting all filter drains into a common drain pipe, going then out of the tower, through a liquid seal bend.