PLATE DAMAGE AS A RESULT OF DELAYED BOILING

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ABSTRACT

After shutting down the direct steam feed and the vapour inlet to a continuos distillation column, repeated tray damages of up to 30 trays occurred. The cost of the damages amounts to 250 T€ in each case. A liquid level reduction in the column sump for a minute on the level display was observed each time.
To understand these events, first the steady state behaviour of the column was calculated by VT-Plan (an in-house process simulation tool) using LLVE description. Due to these calculations, three liquid phases could be distinguished. In the rectification section an organic liquid phase with 800 kg/m³ density exists and in the stripping section a hetero azetrope was found, with a liquid density of 1060 kg/m³ in the organic phase and the normal 980 kg/m³ in the aqueous phase. The difference in boiling temperature between the organic and the azetrope is 40 °C.

Experiments were carried out in the distillation laboratory in Building B310 in Leverkusen to understand the process during tray column shutdown. In these experiments, the mixing process in the sump was observed. Additionally the pressure drop over a lab scale trickle tray column and the amount of distillate was measured.

After shutting down the direct steam and the vapour inlet feed, the trays started to drain. First the heavy organic phase reached the sump. These droplets fell down through the water phase hold up. Due to the hydrostatic liquid height of the water phase, boiling of the heavy organic phase was suppressed. When the light organic phase from the rectification section reached the sump, a third light liquid phase was added above the water phase . This led to heavy boiling at the interface of light organic and water phase due to the high boiling temperature difference.

The pressure drop over the whole column is normally 200 mbar during steady state conditions. After shutting down of the inlet streams, the sump pressure decreased to a value equal to the column head pressure due to the lack of pressure drop across the column. When the decreasing pressure in the sump compensates the hydraulic hight of the water phase (and light organic phase) delayed boiling occurs in the lower organic phase. Due to mixing effects, the interface between the different liquid phases increases and causes violent boiling. All the liquid in the sump entrained to the first tray and damaged the supporting beams and the trays till the high kinetic energy was lost by expansion.

To protect the column internals, the sump hold up was reduced, a cooler was added to the column and the trays were strengthened by threaded spacers. Since these improvements were made the column has been subject to three emergency shut downs. These shutdowns were all carried out successfully without causing tray damage.

Column damage

Damage to distillation trays is not unusual. Damage is generally the result of poor maintenance, structural defects or a lack of the required measuring devices, which then results in normal loads being exceeded. However, damage occurred three times within 1½ years to column trays in a tray column, after the column had been shut down. Shutting down the column is the final safety precaution taken when critical conditions occur in any column. This is done primarily to protect the environment from the emission of hazardous substances and secondly to return the apparatus to a safe condition.

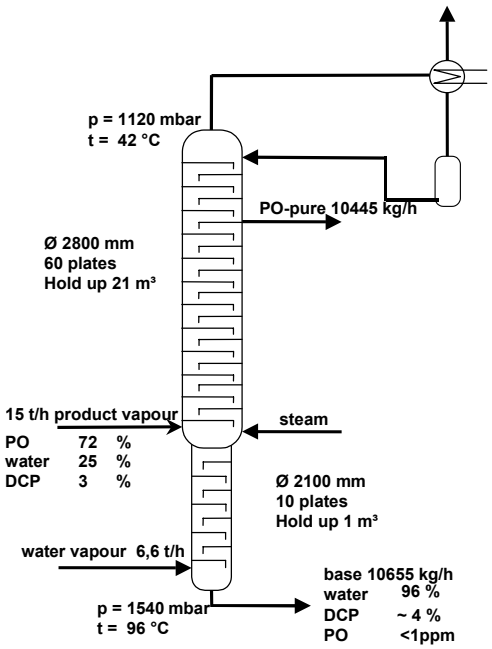
The two photographs shown below demonstrate very impressively, the extent of the damage. It can be seen quite clearly that the trays buckled or were split apart in an upwards direction, so the force being exerted must have come from below the trays. 20 to 30 trays were damaged during each of the three incidents. The repair costs were about 250 T€ each time. You can get an idea of the destructive forces involved when you realise that the tray sheet metal was designed for a mechanical load of 70 mbar pressure difference and the supporting reinforcement parts were actually designed for 200 mbar pressure difference. The noise made during these events was not noticed.

Mode of column operation

First of all we need to describe normal operation of the column.

The distillation column is used to separate a volatile hydrocarbon from water. The column consists of 70 trays which contain Bayer bubble cups. The main product stream to the column is in the form of a vapour and consists of a light component as well as about 24 % water and about 3 % of another chlorinated organic secondary

product. The vapours are fed to the column on the 10th plate from the bottom. The column is heated by passing direct steam into the sump of the column.

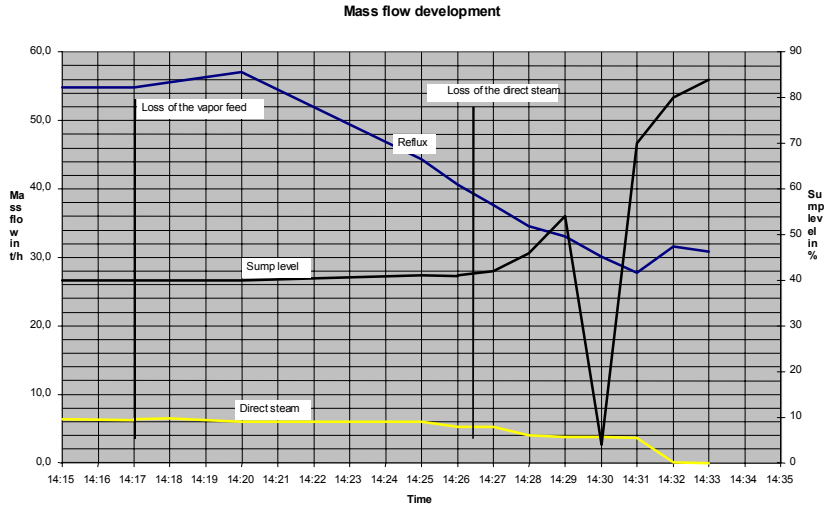


The head vapours from the column are totally condensed and returned to the 70th tray as reflux. The purified HC is withdrawn as a liquid side stream at the 58th tray. The water from the feed vapours , the direct steam feed, and the chlorinated component are drawn off as bottoms product.

The column is operated with a head pressure of about 1120 mbar (absolute); the pressure is controlled by means of cooling water flow rate. The 70 trays produce a pressure difference of 420 mbar. There is very little difference in temperature in the rectifying section, only about 12 °C across 60 trays. In contrast, in the stripping section, the temperature increases from 50 to 96 °C at the sump across only a few trays. The liquid hold-up in the column is approximately 22 m³, of which 21 m³ are allocated to the enricher and only 1 m³ to the stripper.

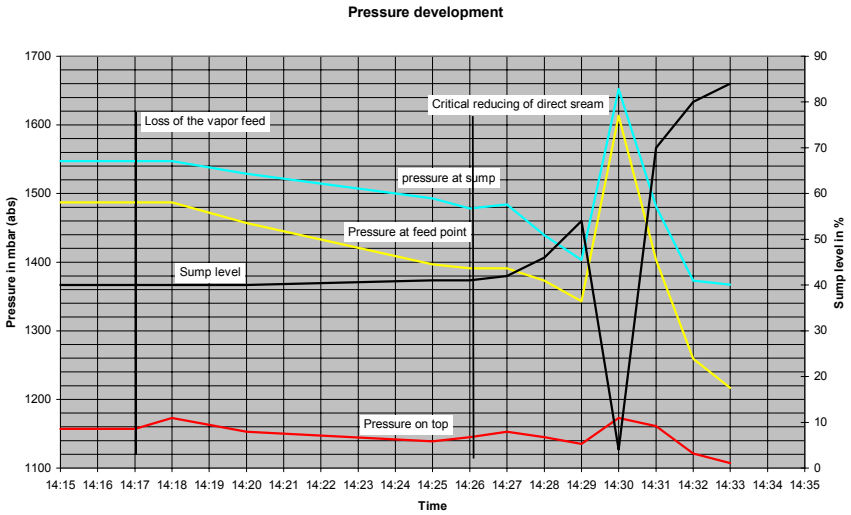
Change in pressure as seen from the data archiving programme 'IMPASS'

The processing data, which were recorded using the archiving system 'IMPASS', have only a limited resolution with a recording frequency of one minute.



The three monitoring points: head pressure, absolute pressure at the feed point, pressure at the sump, as well as the level of liquid at the sump where the level is maintained using a pump are shown.

At about 14:17 the column was subject to an emergency shut-down due to a power failure. At this point the direct steam is taken over manually using the actual value. The head pressure in the column can be kept constant by means of regulation of the adjustable level. Due to the lack of product vapour, the pressure difference in the rectifying section decreases and the trays drain. The pressure at the feed point drops due to the reduced loss in pressure. The total pressure at the sump of the column also drops. The pressure drop in the stripping section however increased due to the liquid drained from the upper trays.



After 14:25, the direct steam at the sump of the column is reduced stepwise. Due to the increased amounts of liquid on the trays in the stripping section, these trays also start to drain, which is indicated by a rise in the bottoms level. The pressure drop at

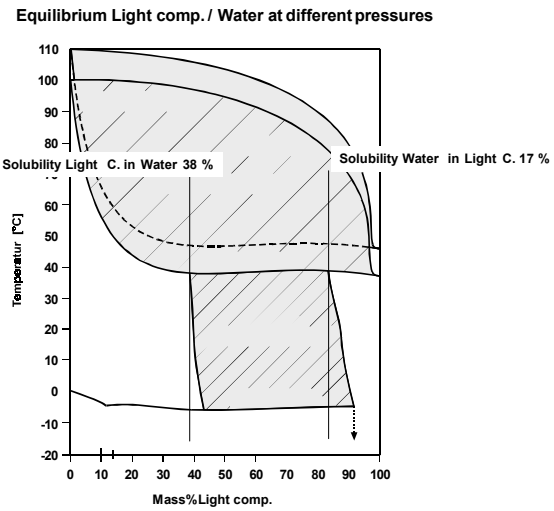
the feed point and at the sump of the column takes place more rapidly. At about 14:27 the bottoms level falls spontaneously from 54 to 5 %. Simultaneously the pressure gauges at the sump and at the feed point increased rapidly by 250 mbar. At the column head, the pressure increased by only 38 mbar. This shows that an extreme pressure difference over the column trays was generated.

Phase equilibrium of the components involved

The phase equilibrium diagram for the light component/water system shows that the system is only partly mutually soluble and that with no azeotrope is formed. Thus there are reasons for supposing that the partial solubility of the system is the cause of the damage to the trays. The density difference between the two liquid phases is about 160 kg/m³, with water being the denser phase.

Thus, there was reason to believe that the light component draining downwards formed a second phase on the aqueous sump product. The liquid phases have a boiling temperature difference of about 40 °C. The sump of the column, about 5 m³ of water, contains enough energy potential to vaporise part of the organic liquid and produce amounts of vapour which could lead to the damage observed.

The chlorinated component, present at only 3 % in the feedstock, is virtually insoluble in water and forms a heteroazeoptrope. The phase equilibrium with the light component is ideal. As is normal with chlorinated products, the density of the liquid is greater than that of water.

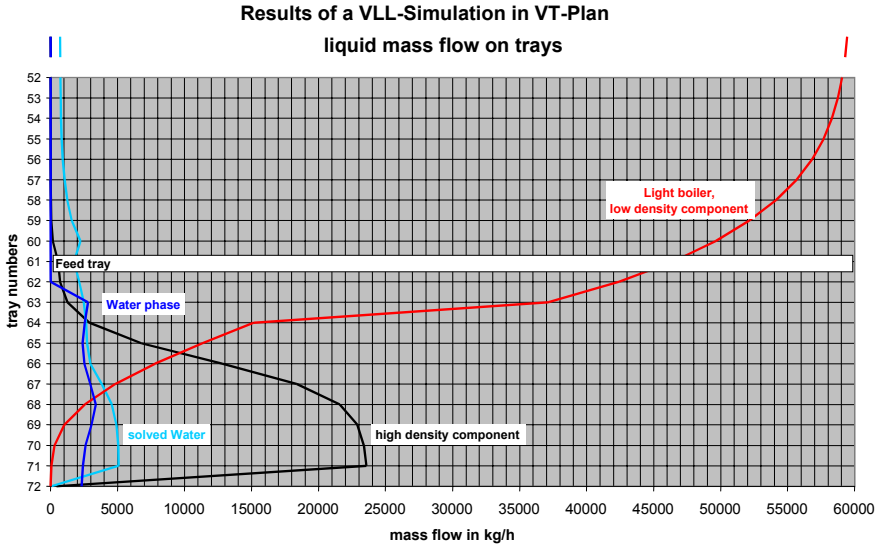


3-phase flash calculation

To determine the profile under normal operating conditions, the column was calculated using VT-Plan, an internal Bayer simulation programme. The equilibrium was described using the NRTL approach and the liquid was assumed to be in 2 phases. The results of the calculation are shown on the following slide. The curves show the component streams over the last 10 rectifying trays and all the stripping section trays.

It can be seen quite clearly that an aqueous phase is formed only in the stripper section. The aqueous phase is between 2.3 and about 3 t/h. The proportion of organics in the aqueous phase is less than 40 kg/h and can be ignored here.
The column separates the organic phase into 2 definite zones:

- The 4-5 trays directly above the sump show a pronounced intermediate boiler bulge which mainly contains the chlorinated component. The maximum mass flow of the chlorinated component is 23 t/h.
- Above this zone, the concentration of light component increases steadily until it reaches 60 t/h in the non-aqueous region. The amount of water dissolved in the organic phase, at a maximum of 5 t/h, is greater than that in the pure aqueous phase.



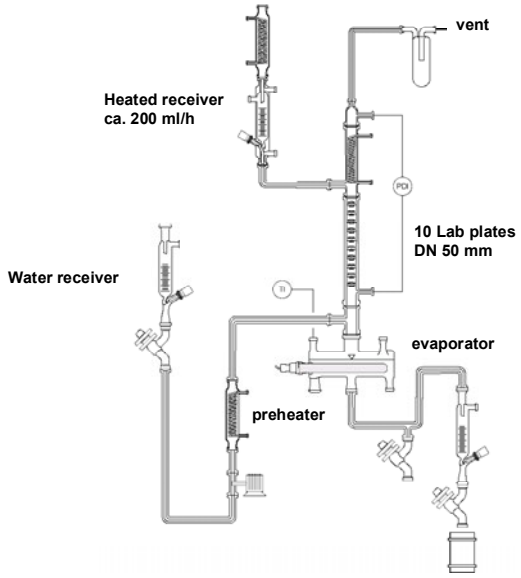
The composition on the stripper trays was estimated using this calculation.

Laboratory experiment

The cause of the damage was thought to be the presence of two liquid phases. As it drains downwards, the organic phase forms a layer on the aqueous phase, the temperature difference between the phases then leads to a vigorous evaporation process with delayed boiling.

To support this hypothesis, the following trials were performed:

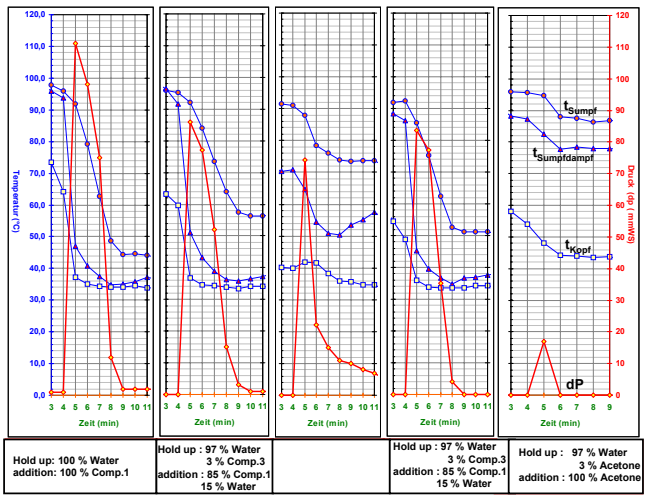
A column, consisting of 10 trickle trays, was first operated with infinate-reflux. For this purpose, boiling water was initially fed to the sump of the column. A preheater produced the required water vapour. When the system had reached a steady state condition, the heating was turned off, thus letting the boiling water drain to the bottom of the column. When the temperature at the sump of the column reached 96 °C, the organic liquid in the heated container was suddenly applied to the head of the column.
The temperature drop at the head and at the sump of the column and the loss in pressure over the column were measured and the evaporation process at the sump was observed.



Laboratory results

The slide shows some examples of trial results.

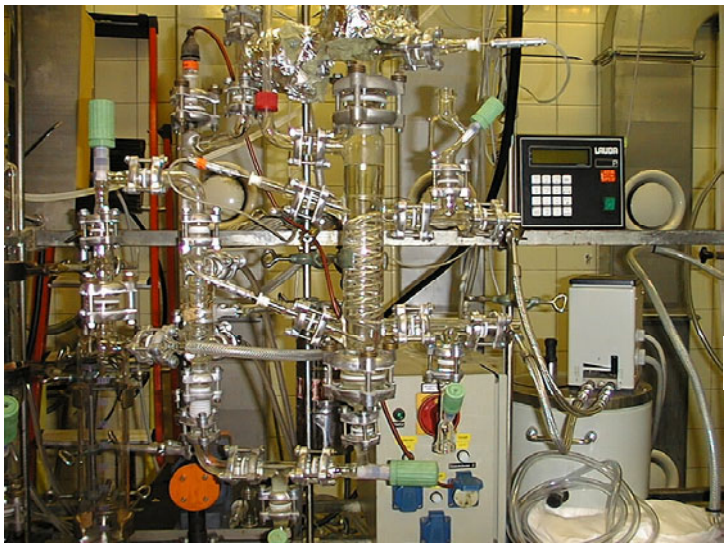
The first curve shows the results when pure water is introduced to the column and pure light component is applied to the column. The sudden pressure difference and the temperature drop in the column are the most pronounced of all the trials. Variations in the product added, such as for example a light component with maximum water-solubility or a representative mixture, lead to lower pressure differences and smaller temperature drops at the sump of the column. Curves 2 and 4 show that the results are very reproducible.
The same trial with water and acetone, which is completely miscible with water, shows for similar boiling point differences only a small increase in pressure difference and only a small temperature change at the sump of the column.
The energy differences are partly compensated for by good mixing in a soluble system.



Modifying the laboratory apparatus

Even though the laboratory results clearly demonstrate that intense evaporation does take place when insoluble components are mixed, the laboratory trials could not at first also demonstrate effects such as a drop in level. Rather, the sump of the column appeared to operate within the range of normal column trials.

Perhaps the horizontal evaporator had too small a depth and too large a surface area for such effects to be produced at all. The sump of the column was therefore redesigned in accordance with the following photograph. The diameter of the sump was now the same as the diameter of the column, 50 mm. The ratio of sump hold up to volume of product was the same as the ratio of the volume of the stripper section to the volume of the operational sump, approximately 3. However, this ratio had also been achieved in the horizontal evaporator.

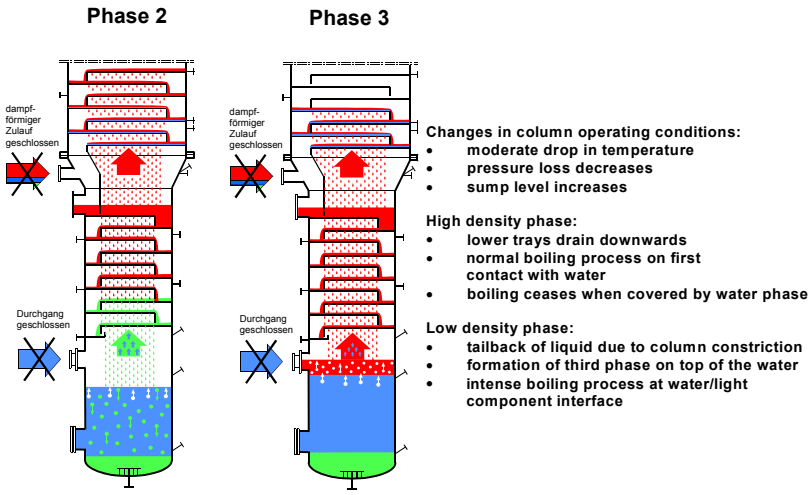


In the optimised lab configuration eruptive evaporation was observed and led to better understanding of the processes.

What happens when the steam is switched off ?

Using these reproducible trials in the modified sump of the column, the post-evaporation process is explained as follows:

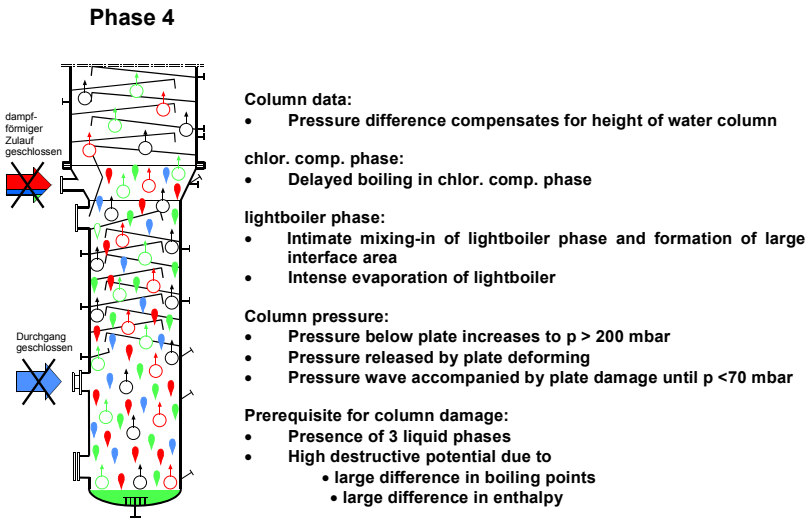
When the vapour feed and the heating steam are switched off, the pressure difference across the trays decreases and the trays begin to drain. That means that the heavy phase consisting of the chlorinated component accumulates at the sump of the column. The droplets fall through the aqueous phase causing partial evaporation. With increasing hydrostatic coverage of the droplets by water, the ambient pressure of the drops is higher than the pressure at the boiling point. Evaporation again ceases. After drainage of the chlorinated component, the volatile organic phase (the light component) can reach the sump of the column. This forms a third layer on top of the aqueous phase. Vigorous boiling takes place at the interface between the light component and the aqueous phase.



The cause of delayed boiling

The still decreasing pressure compensates for the hydrostatic height of the liquid column above the chlor. component phase and boiling is initiated at the water/chlor. comp. interface. The chlor. comp. starts to evaporate. As the bubbles of vapour rise, the density of the liquid column decreases and the pressure at the interface is reduced further and there is delayed boiling in the chlor. comp. phase.

The intimate mixing of all the liquid phases produced in this way results in a large interface and thus intense evaporation of the lights from the hot aqueous phase. The pressure below the 1st tray increases to the point where the moment of resistance of the tray and the support is exceeded and the support and tray deform. The pressure surge on the other trays which then takes place, leads to damage until the pressure loss has become < 70 mbar. At this point the vapour expansion after the 20th or 30th tray no longer causes tray damage.



Inclusion in the PLS system

Even when the heating steam is not turned off at precisely the same time as the loss of product, the processes described above are still observed, as can be seen from the curves for changes in level and pressure.

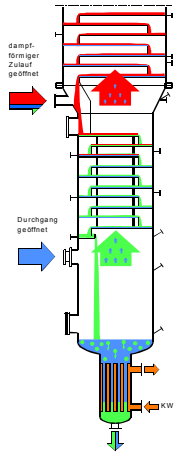
Proposals for avoiding post-evaporation

Based on the findings given above, the column was changed as follows:

To minimise the energy potential, the liquid volume in the column sump was reduced. This was done by reducing the sump level significantly.

The energy from the falling dense chlorinated phase is transferred to the surroundings by an integrating a cooler in the sump of the column.

In the event of a loss of the feed and of steam, emptying of the sump of the column is maximised by means of the control system. A rise in sump level is therefore avoided. In addition, the trays are reinforced with distance bolts which increase the strength so as to resist a difference in pressure of 500 mbar.



Proposals to avoid post-evaporation

Minimising the energy potential

- **Reduce volume of sump**
- **Remove energy by using integrated sump-cooler**
- **Maximum sump drainage if steam fails**

Reinforce the plates

- **Increase strength of plates to 500 mbar pressure difference, instead of 200 mbar**

Now, after operating the column for over 1 year using the plant shown, no more damage has occurred, although in the meantime the column has been subject to three emergency shut-downs.

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