SIMULATION AND EXPERIMENTAL ANALYSIS OF OPERATIONAL FAILURES IN A METHANOL - WATER DISTILLATION COLUMN

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ABSTRACT

A rigorous model for the simulation of the dynamic behaviour of distillation column including pressure relief has been developed. This model allows the analysis of the influence of different disturbances (failures) on the dynamic behaviour of a methanol/water column. Disturbances of the cooling medium supply to the condenser and the reflux stream have been examined. In this paper the results of the simulation for a failure of the cooling water supply are presented and discussed. Experimental results, which should help to corroborate the simulation results as well as visual observations of the hydrodynamics on one of the distillation trays of the column during relief are also presented.

INTRODUCTION

Distillation columns are the most widely used separation operation in the process industry. Distillation columns present a special safety hazard because of their large inventories, which often involve flammable solvents. Disturbances during operation such as the failures of individual components can result in a deviation from the normal operation and could in the long run lead to a situation were the unit has to be depressurised (relief). That is, conditions which are not within the defined operating procedure may occur.

The new European control of major accident directive Seveso II will in its implementation make it necessary that plant operators deliver detailed evidence (i.e. Safety Report) that the plant design conforms to the required safety standard [1].

Although simulation studies are widely used to examine the operating behaviour of distillation columns, they are seldom used to carry out systematic studies of failures during column operation. Deerberg et al [2] state that in addition to the conventional methods, dynamic simulation can be a powerful tool for safety examinations in chemical engineering. Detailed dynamic simulation of operational failures gives a wide range of information about unknown internal process quantities. This leads to a deeper understanding of the process behaviour. With this instrument at hand risks can be assessed for normal operation states as well as in the case of operational failures so that applicable preventive measures can be defined. Testing improvements of the constructive and operational conditions with respect to important aspects of production processes is possible.

It is necessary to consider not only the result obtained within the column but also that of all other important components of the complete plant (valves, pumps, reboiler, condenser, steam supply, cooling medium supply etc.) as well as the important components of the process control system including the installed protective systems. By considering these influences on the process dynamics, many safety related questions can be answered.

The most important physical effects which must be investigated in a distillation column are:

⇒ Influence of the hydrodynamics and mass transfer
⇒ Stability of the control loop during non-standard operating conditions
⇒ Effects of operational conditions on process safety
⇒ Effectiveness of the protective system

To develop simulation tools for safety analysis, the characteristics of the process must be known. The effects most important for safety have to be detected and integrated into the process model.

DISTURBANCES IN DISTILLATION COLUMNS

A study carried out by Kister [3] showed that safety related incidences do occur in distillation columns and have in fact increased in recent years. Many different causes are reported, but imprecise design, operational errors and failure of plant components are mentioned quite often. The study also found that almost half of the failures could have been avoided, if better education and experience were available during design and operation. The ZEMA- Report [4] also relates incidences of accidents in which distillation column are involved.

Depending on the type of distillation column, different sources of hazard can be identified. Firstly there is the hazard of fire or explosion due to emission of flammable material from the column. Secondly, unwanted reactions such as Polymerisation or decomposition may result when there is an unwanted temperature increase due to deviation from the standard operating conditions. It is the job of safety technology to analyse all the hazard potentials as well as determine suitable preventive and operative measures. This includes recommendations on dimensioning and fail-safe design. A systematic examination of the causes that could lead to operational failures and the resulting dynamic behaviour in distillation column has not been done till now.

Kister [5] has listed some of the causes that could result in overpressure in a column. These are :

Utility failure such as

- Loss of coolant
- Loss of electric power
- Loss of steam
- Loss of instrument air

Controller failure

- Failure of steam controller
- Failure of pressure controller
- Failure of feed controller
- Failure of reflux controller

Extraneous sources

- Valve opening to external pressure source
- Loss of heating in an upstream column (dumping)
- Failure of an exchanger tube
- Exterior fire

Internal sources

- Accumulation of non-condensables
- Chemical reaction
- Closed column outlets
- Internal explosion

DYNAMIC MODELLING OF A DISTILLATION COLUMN WITH RELIEF STREAM

A rigorous dynamic column model was developed under gPROMS which can describe the dynamic behaviour of a distillation column. Figure 1 shows the scheme of a separation stage.

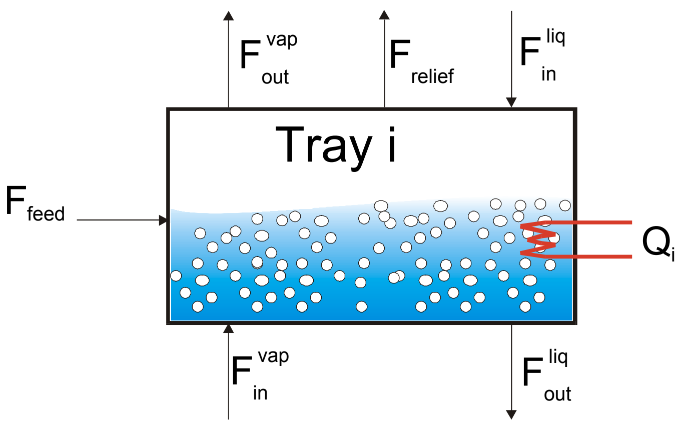

Figure 1: Model of a distillation separation stage with relief stream

The model consists of the following equations for each separation stage:

The mass balance of a separation stage including the relief stream is given as follows:

$$\frac{dM^{ges}}{dt} = F_{in}^{liq} + F_{in}^{vap} + F_{Feed} - F_{out}^{liq} - F_{out}^{vap} - F_{\mathrm{Re}lief} \tag{1}$$

The component balance for the component i on the relief tray is:

$$\frac{dM_i}{dt} = F_{in}^{liq} z_{i,in}^{liq} + F_{in}^{vap} z_{i,in}^{vap} + F_{Feed} z_{Feed} - F_{out}^{liq} x_i - F_{out}^{vap} y_i - F_{\mathrm{Re}lief} x_{\mathrm{Re}lief} \tag{2}$$

And finally the energy balance:

$$\frac{dU}{dt} = F_{in}^{liq} h_{in}^{liq} + F_{in}^{vap} h_{in}^{vap} + F_{Feed} h_{Feed} - F_{out}^{liq} h^{liq} - F_{out}^{vap} h^{vap} - F_{\mathrm{Re}lief} h_{\mathrm{Re}lief} \tag{3}$$

For single phase relief the enthalpy of the relief stream is calculated as usual for the vapour phase in the case of two phase relief the enthalpy is calculated assuming a homogenous mixture as described by the following equation:

$$h_{\mathrm{Re}lief} = h^{liq} + \dot{X}(h^{vap} - h^{liq}) \tag{4}$$

where $\dot{X}$ is the vapour mass fraction which is calculated as follows:

$$\dot{X} = \frac{\dot{m}^{vap}}{\dot{m}^{vap} + \dot{m}^{liq}} \tag{5}$$

Additionally the following equations need to be solved:

⇒ Phase equilibrium using Wilson equation for the calculation of the activity coefficients
⇒ Murphree tray efficiency
⇒ Francis weir correlation for the liquid hydrodynamics
⇒ Pressure loss correlation
⇒ Component physical properties

The relief stream is determine from the following equation:

$$F_{\mathrm{Re}lief} = \alpha \cdot \psi \cdot \sqrt{\frac{2 \cdot p_0}{v_0}} \cdot \frac{A_0}{M_{Wt}} \tag{6}$$

whereby α is the discharge coefficient, and ψ the discharge function. The equations for the calculation of this two parameters for different state of relief i.e. liquid, liquid-vapour or only vapour have also been included in the model. The equations are presented in the work of Schmidt and Westphal [6]. The values calculated in this work were $\alpha = 0.81$ and $\psi = 0.453$.

The remaining trays in the column are modelled in the same way only without considering the relief stream.

The pressure at the top of the column is fixed through the vapour pressure curve mainly by the temperature of the liquid phase in the condenser. The heat transfer in the condenser determines the pressure gradient which is required such that the steam generated in the reboiler flows through the column. It is therefore necessary for the modelling of the pressure control of the column that the dynamics of the vapour phase is considered. Through rigorous modelling of the heat transfer in the condenser, the condenser duty and column pressure can be varied using the cooling medium stream.

The sub models comprising the rigorous distillation are presented in the scheme shown in Figure 2.

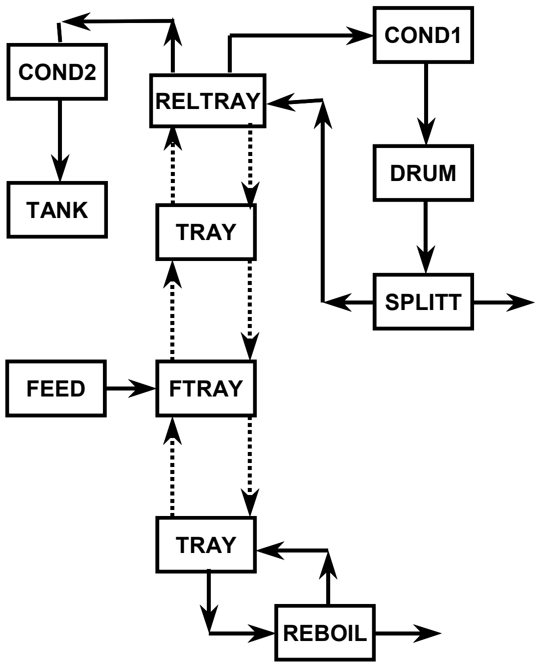

Figure 2: Configuration of the distillation column model

The basis for the model is described in detail and has been validated in previous works [6-9]. The validity of the model equations for the standard operating conditions was given. Disturbances near the operating point can be reproduced quite well. The model has to be validated for the operational failures considered in this work. For this purpose experiments have been conducted in a pilot plant column (100mm, 22 tray column).

As explained in the introduction to this article, simulation tools which are used for safety analysis must be adaptable to the specific process that is being studied. They should give the user the flexibility to included all necessary model details that are required. It is for this reason that gPROMS was used in this work. Commercial simulators such as Hysys are able to simulate specific operational failures but the models are not as flexible and adaptable to specific problems as are Speedup or gPROMS.

SIMULATION STUDY ON A METHANOL- WATER COLUMN

The simulation studies are done for the pilot plant described in the experimental section of this work. This column is equipped with a relief system so as to carry out experimental work on the combined column/safety valve system. Initial simulations were necessary to be able to design the safety valve as well as the dump tank B1.
The dynamic behaviour of a methanol water column during a disturbance of the cooling stream is examined. The scheme of the column is given in Figure 3.

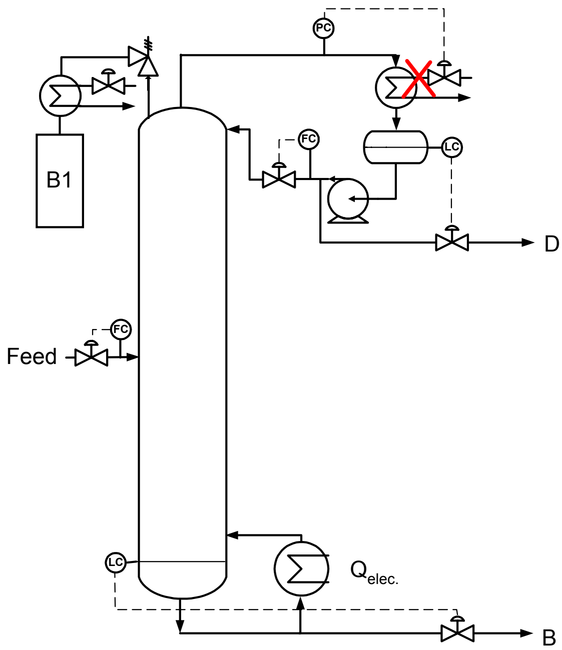

Figure 3: Scheme of the column

The standard operating conditions are given in Table 1.

Table 1: Operating conditions of column (simulation)

Components	Methanol/water
No. of trays	22
Feed (X_{meoh})	4 l/h (0,3)
Reflux	2.32
Q	2.6 kW
Cooling stream	260 l/h

After steady state operation of the column 5 minutes a cooling water disturbance is introduced to simulate the effect of cooling water loss in a distillation column. 260 l/h

cooling medium are supplied to the condenser during normal operation. The cooling water is reduced to 55 l/h.
This disturbance leads to a pressure increase in the column (Figure 4). Less cooling medium is supplied to the condenser, this leads to a substantial reduction of the rate of condensation. This can be explained from the equation for the condenser duty:

$$Q_{Kond} = \dot{m} \cdot C_P \cdot \Delta\vartheta_K = k \cdot A \cdot \Delta\vartheta_m \tag{7}$$

As the flow rate of the cooling medium $\dot{m}$ is reduced, the temperature difference $\Delta\vartheta_K$ of the cooling medium increases bearing in mind that the heat supply to the column remains constant.

This leads to an accumulation of vapour in the condenser with the effect that the pressure in the column increases. The heat input to the reboiler is required on one hand for the vapour build-up in the column, on the other hand, the column and liquid holdup have to be raised to the new temperatures. As such the geometry of the column at constant reboiler duty is of importance on the rate of pressure increase.

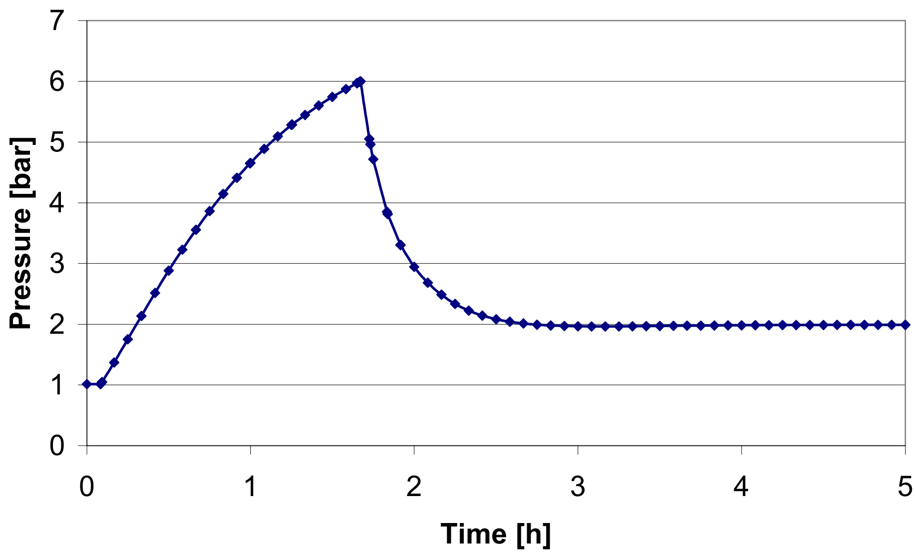

Figure 4: Column pressure after a failure of the cooling duty with subsequent relief

As Figure 4 shows, the pressure in the column (Tray 1) rises from 1.013 bar to 6 bar. A first-order response of the pressure can be observed. The column requires about 1,5 h to achieve the set pressure of the safety relief valve. At this point the valve is opened and the column is relieved. The pressure then sinks from 6 bar to 2 bar at which point it achieves steady state.

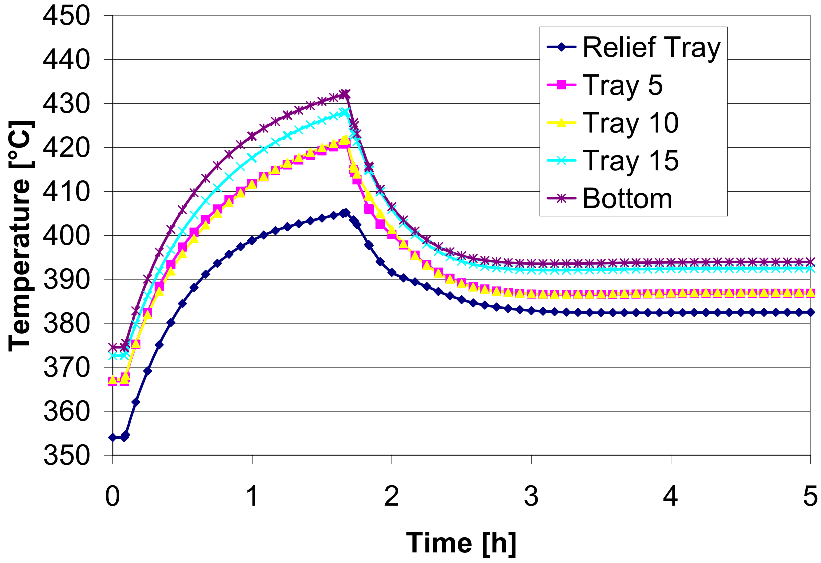

Figure 5: Temperature profile of the column

The temperatures that result with the change in pressure in the column are shown in Figure 5. The temperature at the bottom of the column (Tray 20) increases from 375 K at 1.013 bar to 432 K at 6 bar. This relates to the boiling point of almost pure water at the given pressures. At the column top the vapour consists of a mixture water and methanol, and its temperature increases from 352 K to 405 K.

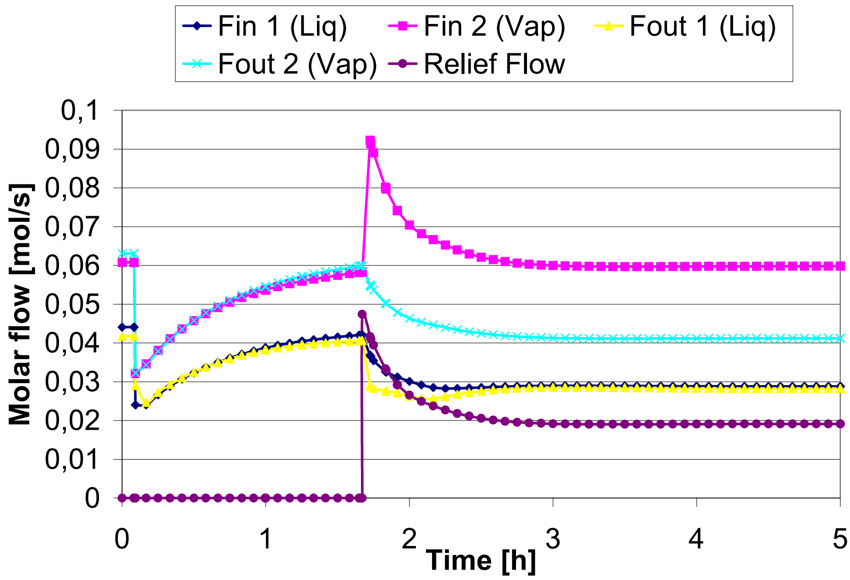

Figure 6: Molar flows for the streams of the relief tray

The input and output stream of the relief tray are shown in Figure 6. There is a strong reduction in the vapour flow within the column shortly after the disturbance, this is a result of the heating up process. The increased pressure and temperature in the column lead to a partial condensation of the vapour phase. The temperature difference between the hot stream and the cold stream in the in the column increases, which means that the condenser duty increases despite the decreased cooling stream. In this case the minimum vapour duty in the column is the limit that should be of interest. For our case the vapour duty remains within the limits.

The relief stream shoots up from zero to 0,045 mol/s and then reclines steadily until steady state is achieved.

The liquid load (Fout 1) in the column on the relief tray also decreases rapidly with an increase in pressure and then reduces again as the pressure tends toward a stationary value. On the lower tray (Figure 7) we see a reverse effect, the liquid streams here increase as the pressure rises and then drop off sharply as the column is relieved.

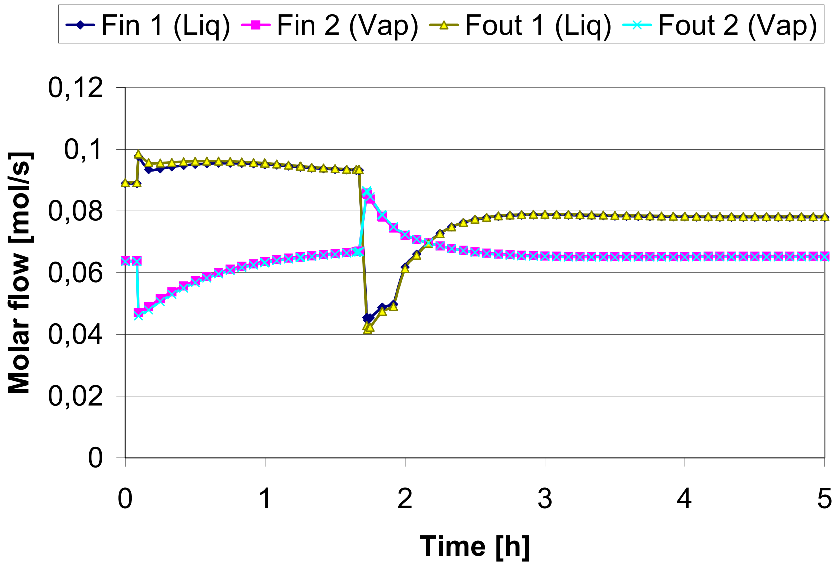

Figure 7: Streams on tray 15

EXPERIMENTAL EXAMINATION

For the purpose of examining the relief process in a distillation column as well as for the validation of the model and the design, experiments were carried out in the pilot plant. A mixture of water and methanol was used for the experiments. The effect of a disturbance in the cooling stream was examined.

Pilotplant

A flowsheet of the pilot column is given in Figure 8. The column has a nominal diameter of 100 mm and consists of 22 bubble cap trays. The reboiler of the column has a maximum heat rate of 6 kW. A relief system at the top of the column allows for relief process studies. This system consists of the relief device, a knock out drum, followed by the secondary condenser and a storage tank. The column is constructed using stainless steel piping. The column is fitted with extensive measurement equipment such that all the required operating conditions can be registered. The temperature on each tray in the column as well as of the relevant streams is measured using PT100- resistance elements. The volumetric flow rate is measured using flow meters. All the measured data are recorded using standardised signals in a process control system Freelance 2000™. This control system is coupled to the LAN so that the data are available on any of the connected Computer terminals. The pilot column is equipped with two sight glasses(tray 8 and 19) to enable the observation of tray hydrodynamics. A digital camera is used to document the hydrodynamic behaviour on the tray during the relief process.

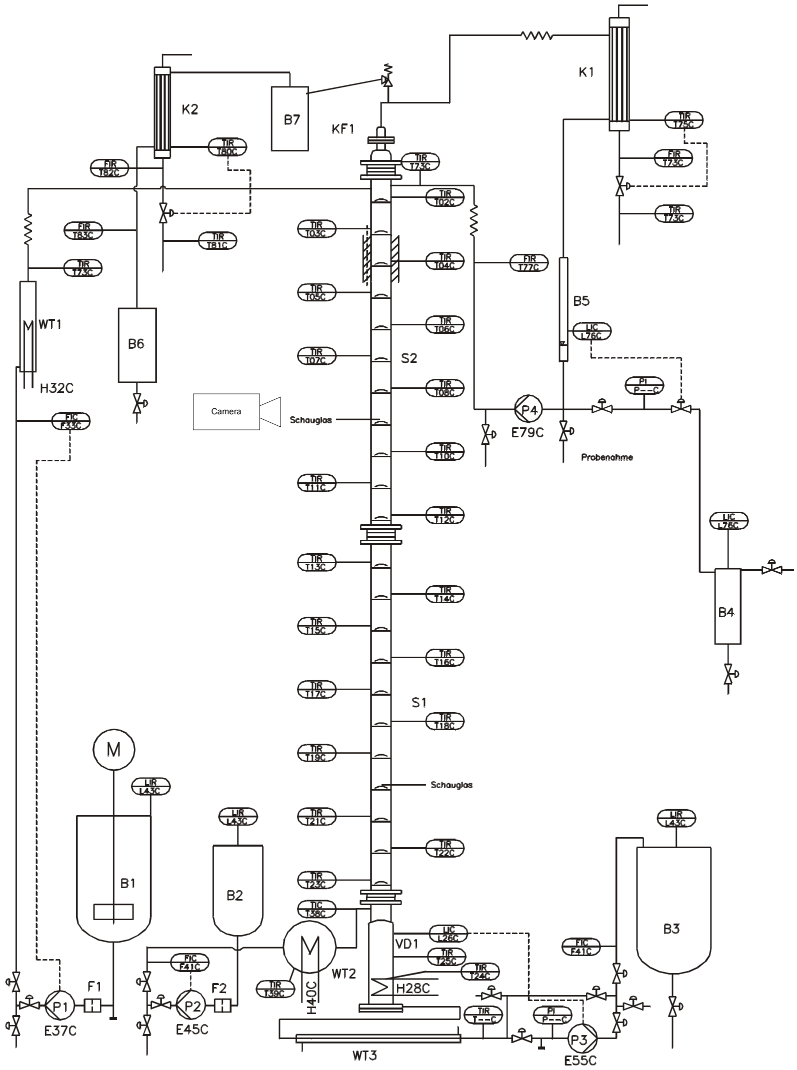

Figure 8: R+I-Flow sheet of the Pilot Plant

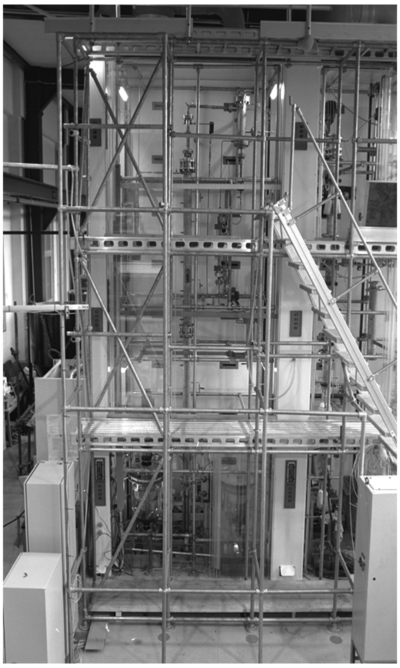

Figure 9: The distillation column pilot plant

Experiment

The plant was started up and brought to steady state operation (Table 2). A step change in the cooling medium supply from 160l/h to 20 l/h was then initiated (Figure 10). The behaviour of the column was then observed.

Table 2: Operating conditions (experiment)

Components	Methanol/Water
No. of trays	22
Feed (X_{meoh})	4 l/h (0.3)
Q	2,7 kW
Cooling stream	160 l/h
Reflux	4 l/h

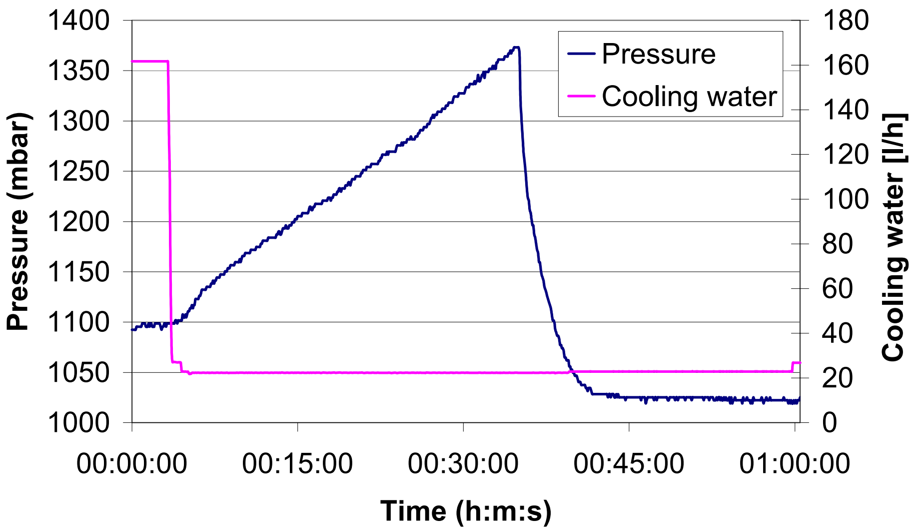

Figure 10: Column pressure and cooling water

The pressure in the column rises from 1.10 to 1.37 bar as shown in Figure 10. At this point the column was depressurised manually by opening of the relief valve at the top of the column, this had to be done at this point as the cooling water exit temperature had reached 65 °C. This temperature is a limit that is imposed on the pilot plant. The pressure in the column then decreased rapidly from 1.37 bar to 1.02 bar.
The temperature in the column is shown in Figure 11. The temperatures generally increase and decrease with the pressure in the column. On some of the trays there is however some variation at the point of insipient relief. At this point there is a slight jump in the temperature as indicated in the Figure 11. This observation is an indication that there is a disturbance of the tray equilibrium as liquid from the lower trays it entrained to those above.

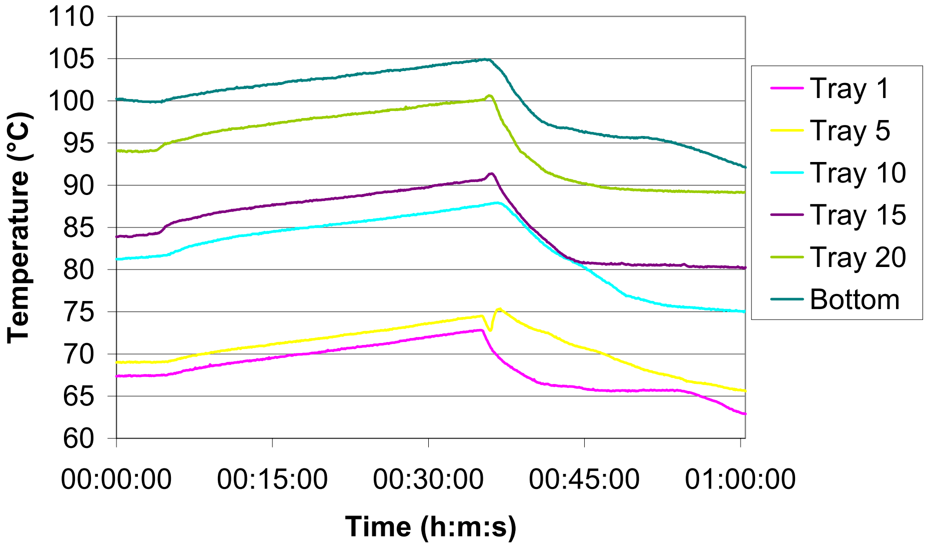

Figure 11: Column temperatures

Hydrodynamic behaviour of the tray during relief

The dynamic behaviour of the tray froth during the relief of the column is shown in the following :

a) Before relief at elevated pressure
b) Start of column relief [t_{rel} = 0]
c) Relief [t_{rel} = 192 ms], the froth height increases.
d) Relief [t_{rel} = 256 ms]
e) Relief [t_{rel} = 1,025 s], froth height at maximum
f) Relief [t_{rel} = 12,917 s], the liquid content of the froth increases. The liquid from trays below is carried over to tray. This is also explains the increase in temperature on so of the trays in the
g) Relief [t_{rel} = 15,69 s], the liquid phase now seems to be the continuous phase
h) Relief [t_{rel} = 31,08 s], the vapour content of the froth is increasing again
i) Relief [t_{rel} = 59,74 s]
j) Relief [t_{rel} = 73,01 s], with lower pressure in the column the relief stream is sinking, and as such the froth height is also sinking.
k) Relief [t_{rel} = 84,22 s],the froth height has now returned to the level before relief.
l) Relief [t_{rel} = 120,13 s], at the start of relief the reboiler duty was turned of. This means that less vapour is now being produced, such that weeping increases and the column begins to dump. For this reason the level of clear liquid begins to rise momentarily.
m) Relief [t_{rel} = 126,72 s], the clear liquid level reaches its maximum.
n) Relief [t_{rel} = 197,21 s], the level of clear liquid is now sinking.

The various stages of the relief process in relation to the pressure in the column are indicated in the following Figure 12.

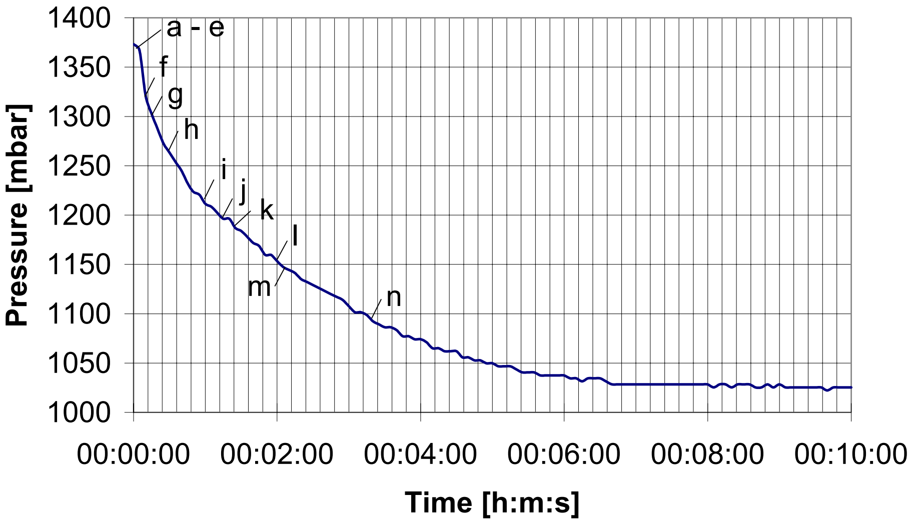

Figure 12: Pressure at the top of the column during relief

Observation during the experiment showed that the relief was two phase i.e. liquid-vapour.

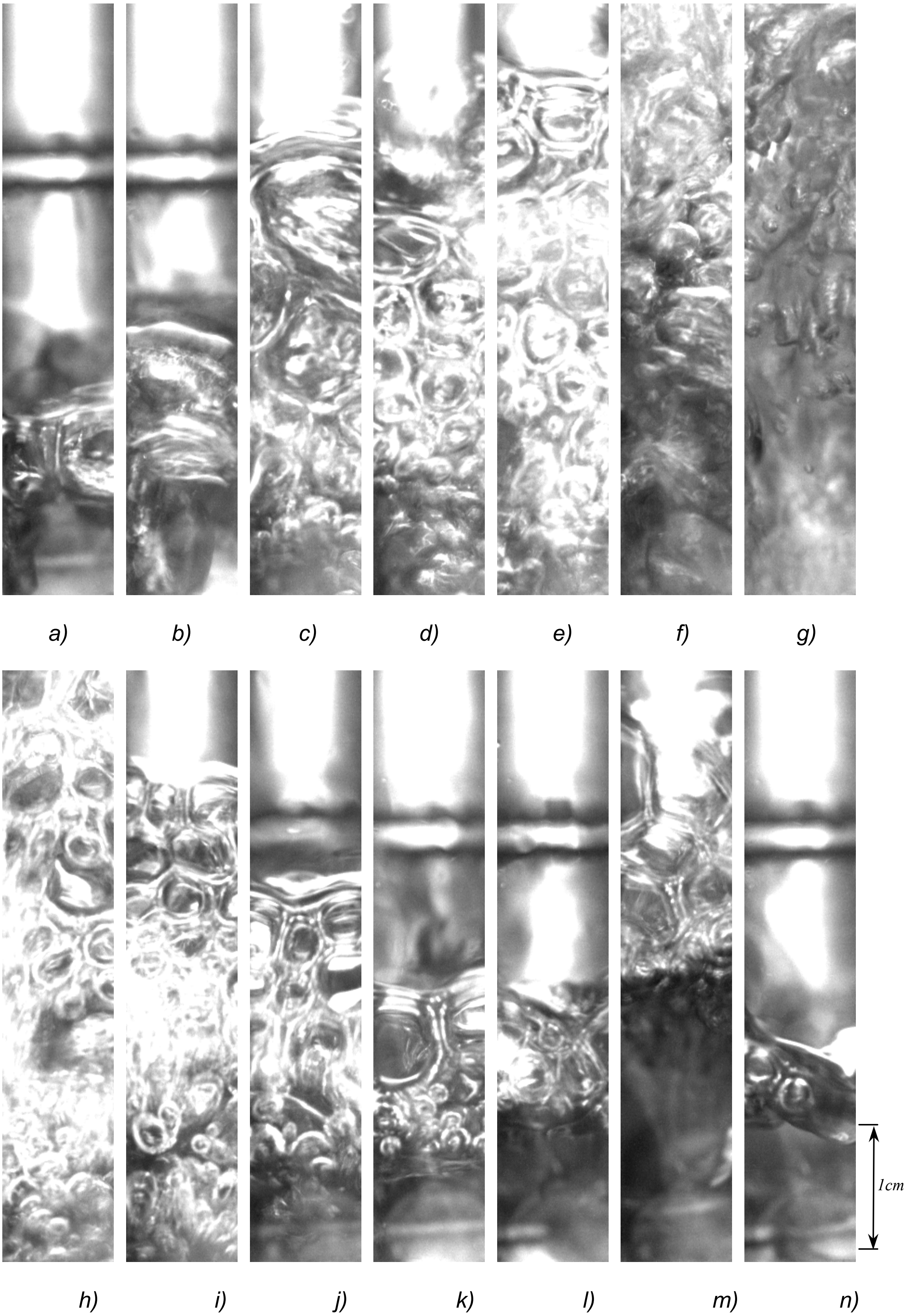

Figure 13: Froth behaviour on Tray 6

COMPARISON OF EXPERIMENT WITH SIMULATION

The following diagram show a comparison of a simulation of the experiment carried out above.

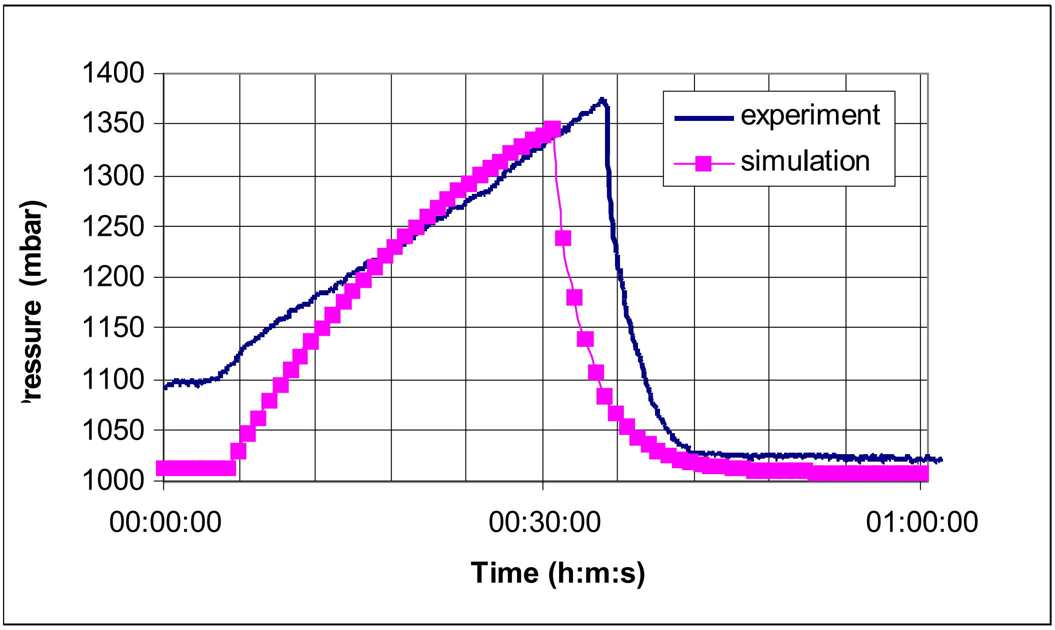

Figure 14: Comparison of the simulated and experimental dynamic pressure

The simulation results show good agreement with the experimental results. The Time contants for the pressure rise fit quiet well. In the case of the relief process they do not fit that well. This may be due to imprecise discharge coefficient for the flow through the relief valve.

DISCUSSION OF THE SIMULATION AND EXPERIMENTAL RESULTS

The simulation results give information about the process behaviour during a particular disturbance. It can be seen that by using dynamic simulation important information can be obtained, which would not have been obtained using steady state calculations. The steady state method can only determine the initial and final states of the system. This means that, the rapid increase or decrease of the vapour or liquid streams in the column cannot be considered. This could lead to inadequate dimensioning of the column.

When conducting safety valve design it is important to know what amounts of vapour or/and liquid would be emitted in the case of a relief. Here again dynamic simulation can give the required information about the actual amounts of vapour and/or liquid that are available at relief time.

The experimental results show that the simulation results qualitatively reproduce the experimental results. The experiments show that it is necessary to know the state of the relief stream as this can be liquid , liquid-vapour or only vapour. It was shown that due to the relief process there is level swell on the tray.

CONCLUSIONS

Using dynamic modelling of the column behaviour during different operational disturbances, the effects of such disturbances can be systematically characterised. For this reason the pressure in the column has to be introduced as a state variable so that the short non-steady state processes can be described. The pressure relief, that could result through the increase in pressure and temperature needs to be examined more closely.

Futher combined analysis of the dynamic column behaviour during non-standard operation together with the relief process should give a deeper understanding of the combined column/safety valve system. The simulated results need to be verified in detail on the pilot plant. The possibilities and timing of preventive measures are to be examined. The results could then be used in the development of new design regulations that would help to achieve hazard free operation or at least help in identifying the hazard potential of the column under consideration.

The criteria for the determination of the state of the relief stream in the model need to be examined more precisely as the knowledge of the state plays an important role in the design of the pressure relief system

ACKNOWLEDGEMENT

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NOMENCLATURE

A	condenser area	[m²]
A_0	relief cross sectional area	[m²]
B	bottom product flow rate	[mol/s]
C_p	heat capacity	[kJ/kg K]
D	distillate flow rate	[mol/s]
F	flow rate	[mol/s]
h	enthalpy	[J/mol]
M	molar holdup	[kmol]
k	heat transfer coefficient	[W/m² K]
$\dot{m}$	cooling medium mass rate	[kg/s]
$\dot{m}^{vap}$	vapour relief mass rate	[kg/s]
$\dot{m}^{liq}$	liquid relief mass rate	[kg/s]
p_0	pressure at point of relief	[bar]
Q	heat supply	[kW]
Q_{Kond}	condenser duty	[kW]
v_0	specific volume	[m³/mol]
$\Delta\vartheta_K$	cooling flow rate temperature difference	[K]
$\Delta\vartheta_m$	log mean temperature difference	[K]

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