Fluid Separation in Two-Phase Flow by Depressurization of Chemical Reactors

S. MUSCHELKNAUTZ and F. MAYINGER
Technical University
Munchen, Munchen, FRG

Abstract

When a chemical reactor is depressurized or when pressure within a vessel, which is partly filled with saturated liquid has to be decreased, normally both vapor and liquid are coming out of the relief valve.
Due to the fact that usual chemical products can consist of toxic and explosive materials, the liquid vaporizing on the ground outside the vessel reaches dangerous concentrations around the vessel, which may cause explosions or accidents with poison gas.

During a research project some fundamentals have been developed for designing a vortex separator which separates the liquid from the vapor and stores it in a collecting chamber.
The theoretical investigation of the pressure distribution within the pipe to the relief valve and the vortex separator shows that the demand for reenchargement of the separated liquid into the vessel without any foreign energy can be satisfied within certain flow conditions.
Therefore, the separator has to be integrated in the pipe between the vessel and the relief valve in a special manner, so that the hydrostatic pressure corresponding to the difference in height is sufficient to lead back the separated liquid through another pipe, against the internal pressure of the vessel.
A vortex separator has been tested with air-water-mixtures and reaches a separation rate of over 90% at low pressure loss over the whole working range.

The problem that I am busy with is to separate the liquid from the vapor in a two-phase flow coming out of a chemical reactor during pressure relief, usually called blow-down. This operation becomes necessary if a chemical reaction is getting out of control.

Runaway reactions are usually bound up with strong exothermic energy turnover. Cooling by evaporation is the best way to lead off this energy. As shown in the Temperature-Entropy-Diagram (Fig. 1), the reaction enthalpy heats up the liquid to the saturation line (point 2). Then it starts to vaporize until the relief valve will open. By reasons of safety the free space sectional area in the valve is mostly oversized, thus leading to a temporal pressure drop within the vessel down to the closing level of the valve (point 4). During the pressure decrease both isobaric vaporization due to the reaction enthalpy and adiabatic flash evaporation are occuring.
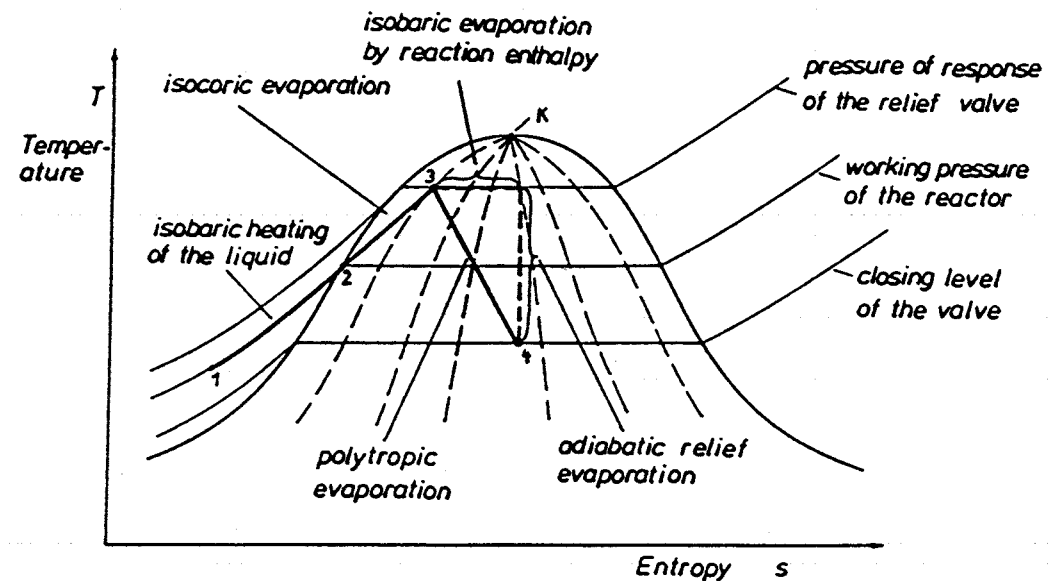

FIGURE 1: Runaway reaction and pressure relief in the temperature-entropy diagram

Due to bubble growth the liquid level is rising up and in most cases it will reach the outlet of the reactor and then a two-phase mixture will flow put of the vessel. Since many fluids used in chemical reactors are either explosive or toxic, it is demanded to prevent discharge of these fluids into the environment. Therefore it is efficient to separate the liquid - which contains the mean part of mass - from the vapor and to collect it in an auxiliary vessel, so-called collecting chamber. In addition there is a demand to transport the separated liquid back into the reactor. For reasons of safety this transport must occur without any external energy source. This can be achieved by using the potential energy of the separated liquid. Fig. 2 shows the physical connection between the potential energy and the discharge of the separated liquid. As soon as the hydrostatic pressure difference between the separator- and the reactorlevel in the return tube is greater than the pressure drop from the reactor to the collecting chamber, the separated liquid will start to flow back into the reactor. Due to the critical flow condition in the relief valve at vessel pressures between 15 bar and 20 bar which are usual in chemical industry the valve generates a big pressure drop and therefore it is necessary to locate the separator between the reactor and the valve in a certain height above the vessel. If the cross-sectional areas of safety
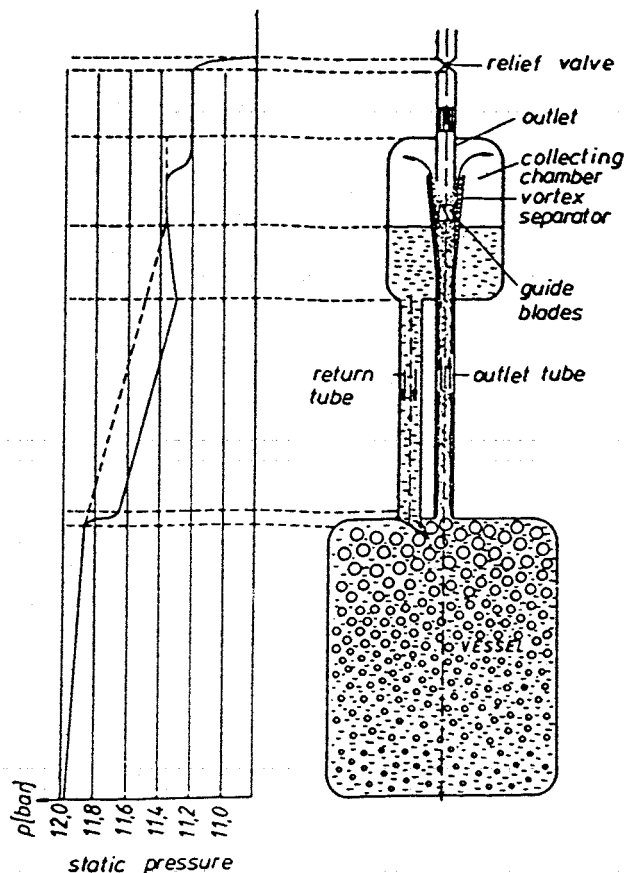

FIGURE 2: Pressure distribution in a separating device

valves are used to fix the diameter of the tube between the vessel and the valve, the velocities of liquid and vapor at the inlet of the separator will be in the range of 25 m/s and 50 m/s. The two-phase flow changes from churn-turbulent flow at the beginning of pressure relief to dispersed flow in a later period.

The course of static pressure along the outlet-tube and the separator, as shown in fig. 2, is the real pressure distribution which was measured with a vortex separator during a blow-down experiment with refrigerant Freon 12. The difference in height between the vessel and the separator was about 4 meters. Due to the conical part of the vortex separator which is acting as a diffusor with an angle of 8°, the static pressure increases and reaches nearly the total-pressure of the flow at the end of the conical part. Therefore the pressure inside the collecting chamber is greater than the static pressure at the inlet of the separator. This fact is of great importance for the reenchargement of the separated liquid into the vessel. The more the pressure difference between the vessel and the collecting chamber can be reduced the more one can lower the height of the separator.
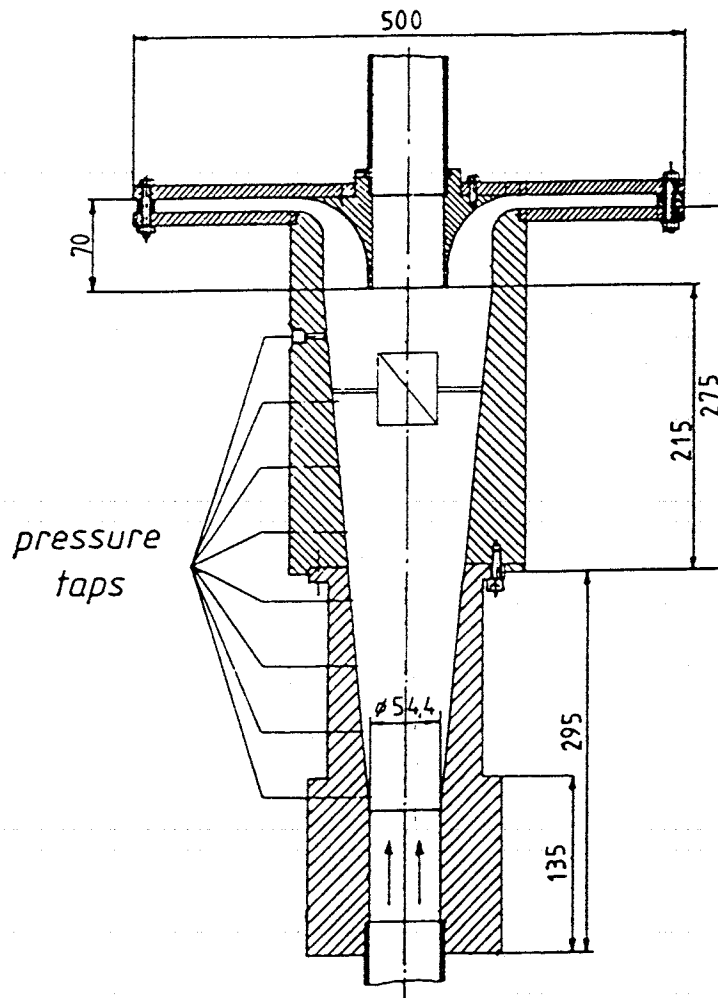
FIGURE 3: Vortex separator

During experiments with an air-water mixture a vortex separator, as shown in fig. 3, proved to be the required device.

After the two-phase mixture has entered the separator the conical expanding part of it leads to a deceleration of the flow which is combined with an increase of static pressure. Four plain guide blades are located centrical in the upper region of the conical part to keep the area under the outlet tube free from liquid. It has been found that there is no need in producing a centrifugal field to achieve a good rate of separation. The guide blades which cover only 20 % of the cross sectional area turn round the liquid to the slit between the outlet tube and the collecting chamber. The vapor leaves the separator through the outlet.

In the next figures a small part of the experimental results which were won by using an air-water mixture is shown.

Fig. 4 shows the measured rate of separation versus an adjusted quality. At low qualities the separation rate is nearly 100 % which is important for a good total rate of separation during a pressure relief. At high qualities the two-phase mixture consists of small droplets which are hard to separate and this leads to a decreasing separation rate. Still, it is better than 80 % over the whole range of quality and thus sufficient for industrial applications. We also made experiments with water mixed with foaming chemical additives. The results show that this has almost no effect
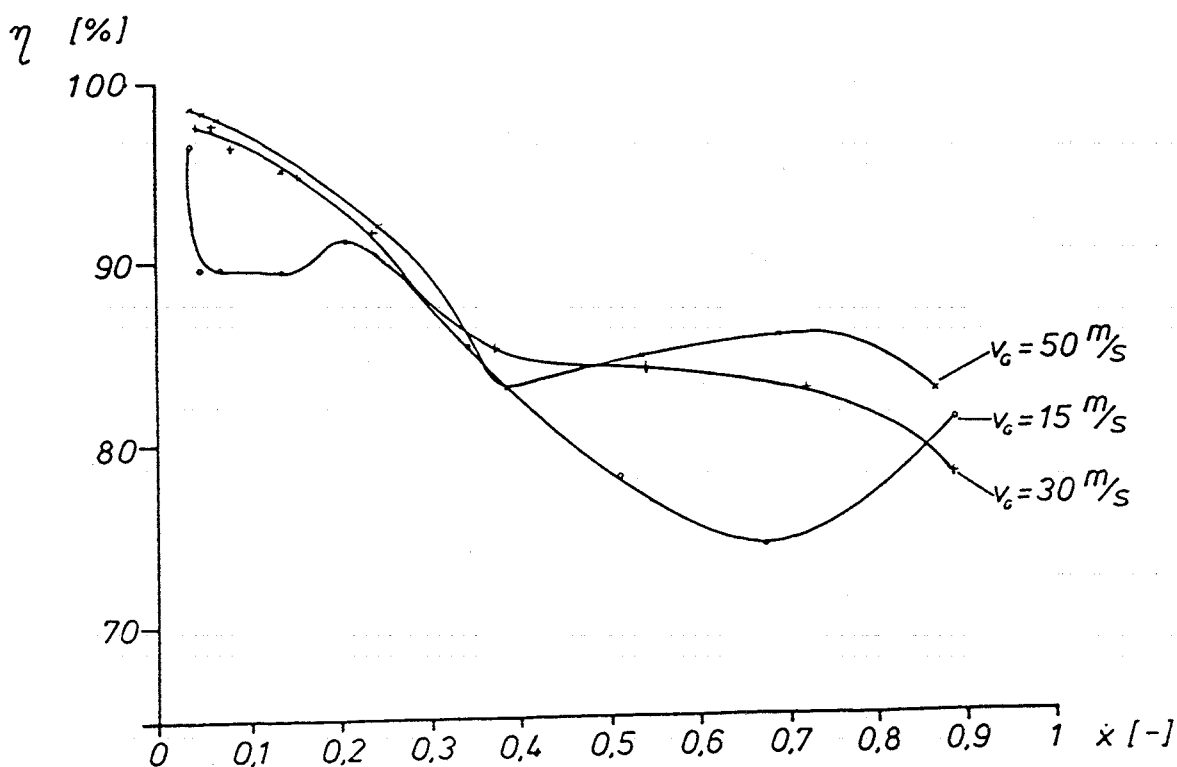

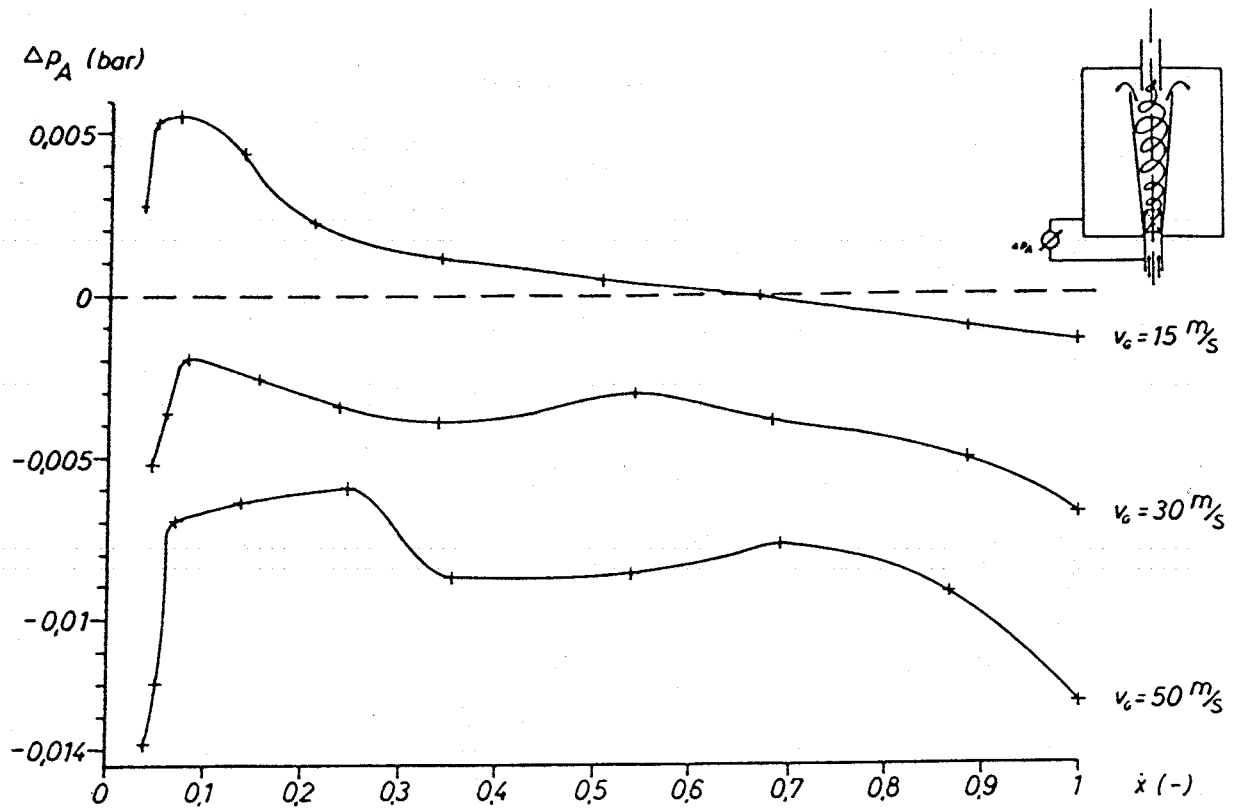FIGURE 4: Rate of separation versus quality of the vortex separator

FIGURE 5: Pressure difference inlet-collecting chamber versus quality of a vortex separtor.

on the rate of separation. The mean velocity of the air-flow has no significant influence on it, too. The according pressure difference p from the inlet to the collecting chamber is shown in fig. 5.

At air-velocities between 30 m/s and 50 m/s the pressure difference is negative within the whole range of quality. Its high gradient at low qualities is due to the increase of the average density of the mixture. Provided that the separator is only operating with liquid flow,the pressure comes near to the velocity pressure of single-phase liquid flow which reaches an amount of several bar in the mentioned velocity range.

In the case of pure gas flow the pressure difference p also turns to its corresponding velocity pressure. Although the diffusor performance is quite well near these limits its efficiency is apparently low within the remaining range of quality. At an air-velocity of 15 m/s the pressure decrease in the conical part of the separator due to the hydrostatic pressure increase due to deceleration of the flow.

Fig. 6 shows the course of the static pressure along the center line of the separator related to the static pressure at the beginning of the conical part, which was also measured by using an air-water mixture with an air-velocity of 50 m/s.
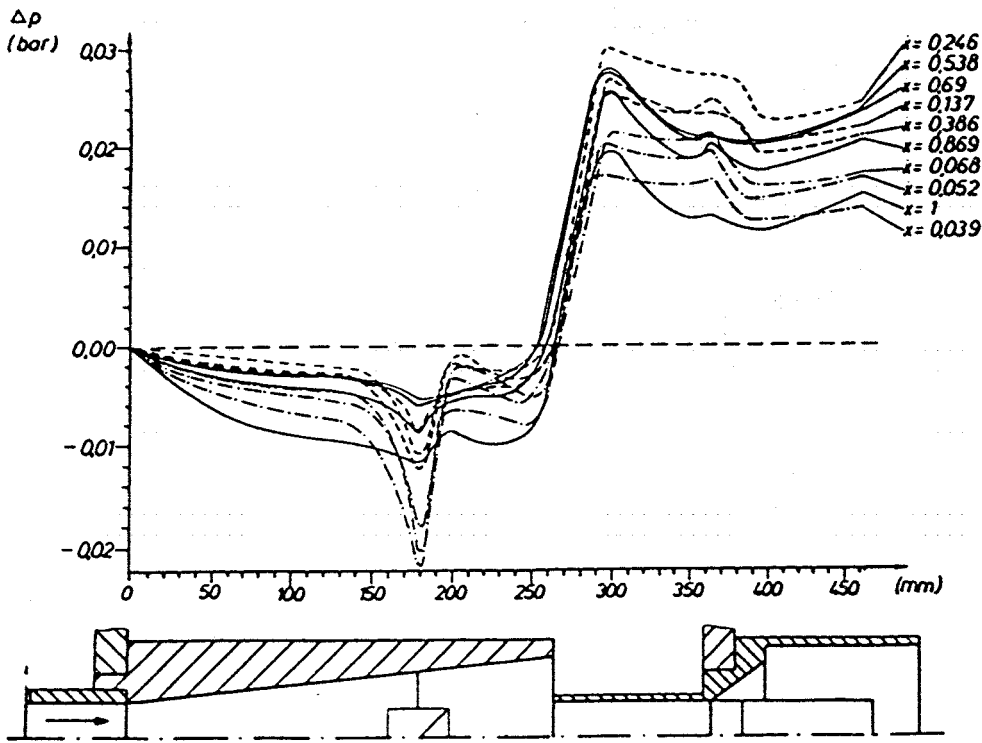
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FIGURE 6:Distribution of the static pressure along the center line of the separator related to the static pressure at the inlet

The pressure increase in the lower conical part of the diffusor depends on the quality of the flow as mentioned above. The small guide blades produce only a low pressure loss while the mean part of pressure drop is caused by the acceleration of the gas flow from the end of the diffusor to the outlet tube. The static pressure reaches its lowest value at the point of maximal flow-contraction which is located in a short distance from the beginning of the outlet tube. Additional measurements of the velocity distribution in the outlet tube have shown that there occurs almost no vortex in it. This is the main reason for the low pressure loss of this separator in comparison to a cyclon separator. Apart from the difference in the course of static pressure along the diffusor, the pressure drop at the outlet remains almost constant for all qualities. This is quite clear since the gas flow beneath the outlet tube carries almost no liquid within it. The pressure distributions at lower air-velocities are similar to the results shown in fig. 6.

In order to examine the rate of separation and the pressure distribution in a separation device under the conditions of pressure relief, an experimental set-up was built for the depressurization of refrigerant Freon 12. Fig. 7 shows a sketch of the plant.

The pressure vessel on the left side has a volume of 200 litres and represents the chemical reactor. It can be depressurized by opening a relief valve which is fixed in the tube between the vessel and the vortex sepa-

orifice

rectifier

vortex separator

guide blades

p/T

return tube

outlet tube

relief valve

depressurized vessel
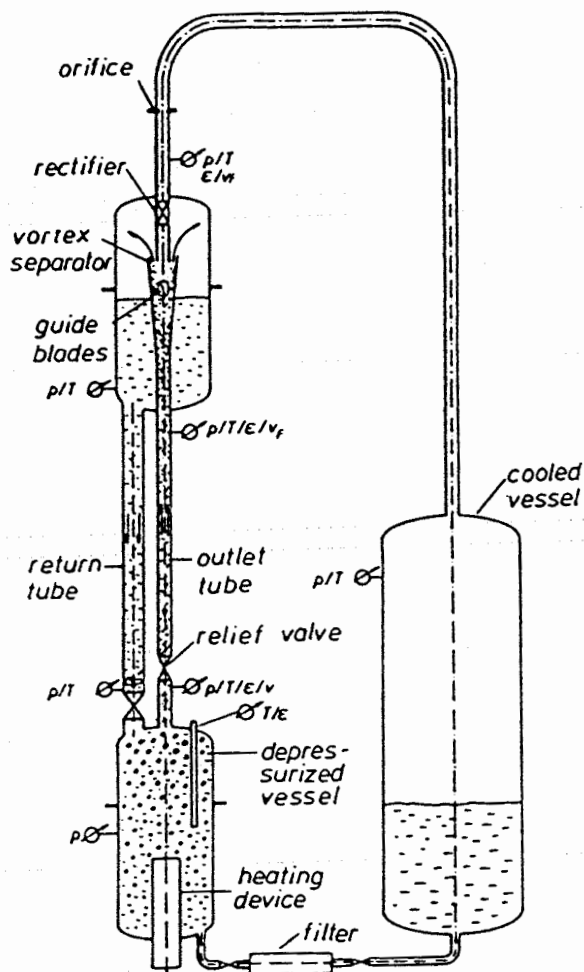
heating device

filter

cooled vessel

p/T

FIGURE 7: Experimental set-up for pressure relief

tor. Since then the wall temperature of the collecting chamber and the return tube is much lower than the saturation temperature of the fluid inside the vessel, the separated liquid will be subcooled during the pressure relief. Provided that the wall temperature is set equal to the saturation temperature of the stored liquid, this will begin to vaporize due to the pressure decrease or a chemical reaction, respectively, thus leading to a reduction of the average density of the vaporizing two-phase mixture in the return tube. At the worst the vaporization of the stored liquid can lead to a flooding of the collecting chamber which will prevent the function of the separator. Therefore, there is a demand for subcooling the collecting chamber and return tube either by use of an external cooling liquid or by keeping the wall on the temperature of the environment which then has to be much lower than the saturation temperature of the fluid inside the vessel. In order to minimize the pressure loss in the relief valve, it is necessary to adjust the cross sectional area of the outlet tube to that one of the valve. The vapor flow must be throttled by an orifice which is installed behind the separator.

The environment is simulated by a large vessel with a volume of 700 litres where the vapor will be condensed at a temperature of 243 K according to a static pressure of 1 bar. The diameter of the outlet tube is

54 mm and the difference in height between the vessel and the separator is about 4 meters.

In order to obtain clarity about the possiblities of the reenchargement of the separated liquid into the vessel, the pressure distribution from the vessel to the throttle has been calculated for pressure relief with refrigerant Freon 12. Bernoulli's equation gives the pressure drop at the vessel outlet.

vessel outlet (0) – (1)

$$\frac{p_1}{p_0} = 1 - \frac{\dot{M}_{L_1}^2}{2 \cdot \rho_L \cdot A_{L_1}^2 \cdot p_0} \qquad \text{(liquid)}$$

$$\frac{p_1}{p_0} = \left[1 - \left(\frac{\varkappa - 1}{2\varkappa}\right) \frac{\dot{M}_{V_1}^2 \cdot \rho_{V_0}}{\rho_{V_1}^2 \cdot A_{V_1}^2 \cdot p_0} \right]^{\frac{\varkappa}{\varkappa - 1}} \qquad \text{(vapor)}$$

The well-known equations of Martinelli et al. have been used for the calculation of the friction pressure loss within the tube between the vessel and the separator. A dimension analysis applicated on the results of the experiments with an air-water mixture leads to an equation enabling calculation of the total pressure drop in the separator. In order to simplify the calculation the pressure inside the collecting chamber was set equal to the static pressure at the separator inlet.

separator (2)-(3) and (2)-(3')

$$\frac{p_3}{p_2} = 0.98 - 0.0075 \cdot k \qquad\qquad \frac{p_{3'}}{p_2} = 1$$

$$k = \left[-(\sin \alpha)^3 \cdot \ln\left(\frac{v_\ell}{\sqrt{g \cdot D}}\right) \cdot \ln \dot{x} \cdot \left(\frac{\rho_{V_1}}{\rho_\ell}\right)\left(\frac{v_{V_1}^2}{g \cdot D}\right) \right]$$

With the assumption of full separation the critical one-phase vapor flow conditions at the orifice can be obtained with sufficient accuracy from the equations of the dynamics of gases.

The result of the theoretical analysis is shown in fig. 8.The curves characterize the flow conditions which allow reenchargement of the separated
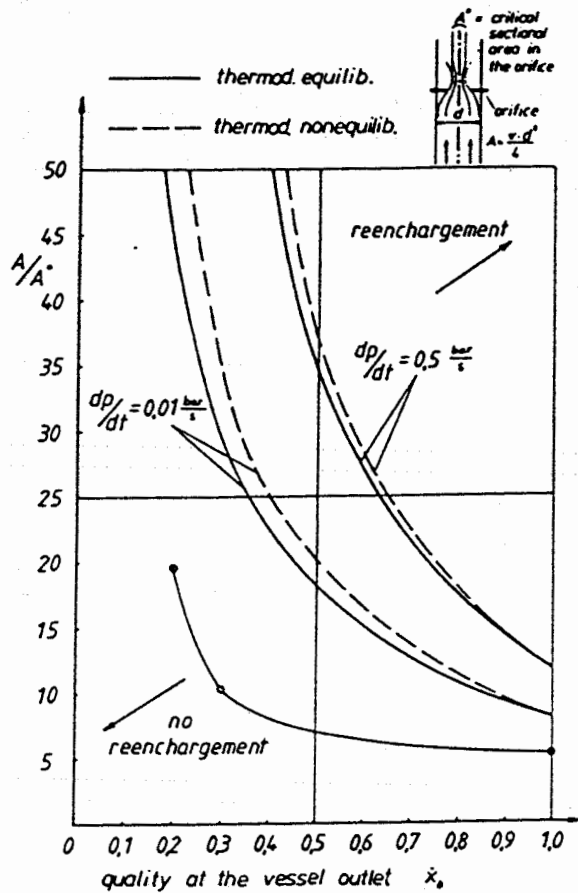
FIGURE 8:Flow conditions at reenchargement of separated liquid into the
vessel

liquid into the vessel. The axis' show the vessel quality at the outlet
and the ratio of the sectional area of the tube before the orifice and
the critical free space sectional area in the orifice. It is supposed in
the calculation that the temporal pressure drop inside the whole system
can lead to an additional boiling of the stored liquid in the return tu-
be, thus reducing the hydrostatic pressure difference of the separated
two-phase mixture compared to the case of single-phase return flow. The
higher the quality at the outlet of the vessel, when the separated liquid
starts to flow back into the vessel.

The experimental results show that the reenchargement fortunately is pos-
sible at lower qualities than predicted by the calculation. However it
is plain to see that the separated liquid cannot flow back into the ves-
sel, if the cross sectional area ratio is smaller than 5. Adjusting a ra-
tio of 20, for example, reenchargement will occur at a quality of 0.2. At
this quality the vessel is filled to about 60 % with liquid. Supposing
an initial charge of 80 % the volume of the collecting chamber must be
20 % of the vessel volume in order to store the separated liquid until
reenchargement begins.

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