CRYSTALLIZATION FOULING IN PACKED COLUMNS

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ABSTRACT

Fouling due to crystallization on structured packings has been studied in a 150 mm diameter column with 1800 mm packing height. A saturated solution of sodium chloride has been used in countercurrent flow to dry air. The pressure drop increase of the column during operation and the ratio between dry pressure drop before and after the experiments are used to quantify the degree of fouling. The data obtained show clearly that, as expected, rough and porous materials such as ceramics and steel as well as wire gauze packings tend to a stronger fouling than others.

INTRODUCTION

Due to their advantages, random and structured packings are widely used contact elements for fluids and gases in the process technology. Intense investigations over the last decades have made the description of the fluid dynamics possible with sufficient accuracy. In contrast to this, the well known problem of fouling in packed columns has met little interest in literature.

Fouling may be defined as the undesired accumulation of solids on a rigid surface. These accumulations influence the operation of packed columns severely by reducing the void fraction and thus the capacity of the packing (see Figure 1 and Figure 2). In this respect higher energy demand caused by increased pressure drop can be mentioned as well as costs for shutdowns, cleaning or replacement of packings.

Industrial applications which are typically affected by fouling are e.g. polymerization reactions in distillation columns (acrylic acid), gas cleaning and waste water operations. Many processes which are known to be endangered by fouling are carried out in dualflow tray columns or sprinkling towers. Using dualflow trays, the advantage of a sufficient fouling resistance has to be paid for with a massive pressure drop and, in turn, with a high energy demand. With sprinkling towers, only poor separation performance can be achieved. The aim is, therefore, to design packings that combine both sufficient pressure drop and good separating performance with high fouling resistance. The first step in this direction is the identification of materials and structures which could be used for this purpose.

Figure 1: Crystallization fouling on the surface of a single Pall-Ring (left side) / clean Pall-Ring (right side)

Figure 2: Crystallization fouling on a random packing

OBJECTIVES

The investigation consists of two parts, both dealing with the mechanism of crystallization fouling. In the first part, a simple experimental setup is used to examine single cylindrical rings. The objective here is to identify the fouling resistance of different materials (glass, ceramics, steel, PP, PTFE). A standard ring shape (15 mm diameter, 15 mm length, 1 mm wall thickness) is used for all materials to make sure that influences resulting from different shapes can be eliminated.

In the second part, several structured packings are investigated in a pilot plant. The aim is to identify packing structures with a high fouling resistance. These experiments deal with different shapes (packings with plane surfaces, wire gauze packings) as well as different materials (PP, steel, textile, glass). For the determination of the degree of fouling it is suitable to observe the increase of the pressure drop during operation. For the measurement of enduring fouling related changes, additionally the dry pressure drop is measured before and after the experiment. The investigated packings are listed in table 1.

Table 1: List of investigated structured packings

manufacturer	name	material	specific surface
Sulzer	Plastic Mellapak 250.Y	PP	250 m^2/m^3
Sulzer	Mellapak 250.Y	steel	250 m^2/m^3
Sulzer	Mellapak 350.Y	steel	350 m^2/m^3
Sulzer	Gauze packing BX	steel gauze	500 m^2/m^3
Sulzer	Gauze packing BX	plastic fibre gauze	500 m^2/m^3
QVF	Dura-Pack	glass	300 m^2/m^3

EXPERIMENTAL SETUP

System

For both the single ring and the packing experiments a saturated solution of sodium chloride serves as foulant. The deposits are generated when the solution gets into contact with dry air. The resulting evaporation of water leads to a fallout deposition of salt. The liquid inlet temperature in all experiments is held at 22 °C. In the relevant temperature range the solubility of sodium chloride can be considered as constant. As a consequence, a definite amount of evaporated water corresponds to a definite amount of salt that falls out.

Single Ring Experimental Setup

The single ring experiments are performed with the setup shown in Figure 3. It consists of a support to which the different cylindrical rings can be attached at an angle of 45° to the horizontal. The gas stream consists of dry air. The air load F is 1 $Pa^{0.5}$. A saturated solution of sodium chloride is fed to the rings. The effective liquid load B is varied in a range from 15 to 60 $m^3/(m^2h)$. The duration of the experiments is 10, 20 or 30 minutes. After each experiment the rings are dried at a temperature of 50 °C. The resulting masses of the deposits are measured with a scale.

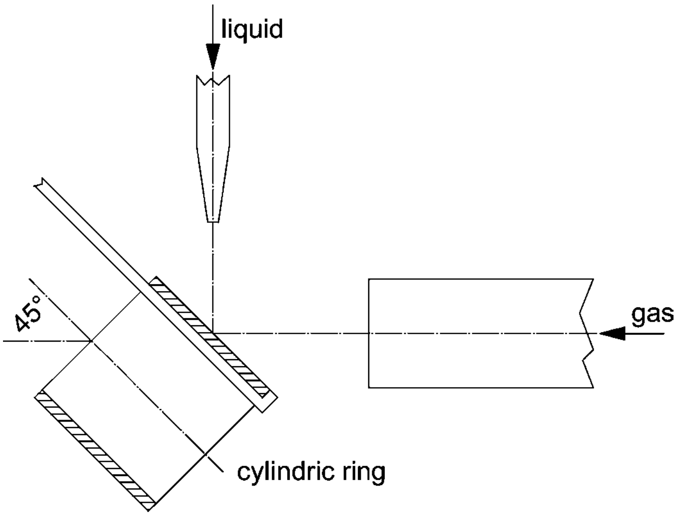

Figure 3: Single ring experimental setup

Pilot Plant

The experiments are performed in a glass column with 150 mm in diameter (see Figure 4). The column consists of 10 sections with a height of 150 mm each. Between these sections nozzles for the pressure drop measurement are provided.

The gas stream (dry air) is blown into the column below the bed support. Due to variable pressure drop during operation a roots fan is used. The saturated air leaving the top of the column is led trough a demister to separate small droplets from the gas stream.

The liquid circulates from a heated tank to the top of the column. There it is supplied to the packing trough a perforated distributor. At the bottom of the column the liquid is recollected and fed back into the tank.

The heated liquid tank serves on the one hand as a settler for the salt deposits washed out of the packing, on the other hand it is used to compensate for heat losses caused by the water evaporation and, thus, maintaining a constant liquid temperature. For cleaning purposes a separated water circuit is provided.

The instrumentation of the pilot plant contains a temperature measurement for the liquid at top and bottom of the column and in the heated tank. Temperature, pressure and relative humidity of the gas are measured at the inlet and outlet. Therefore, the amount of water in the air can be calculated.

The differential pressure measurement is connected to pressure nozzles in the column by magnet valves. With this system, the pressure drop can be measured in each section of the packing.Before starting an experiment, the dry pressure drop of the observed packing is measured at a gas flow rate F of 3 Pa$^{0.5}$. The experiment is then performed at a constant gas flow rate F of 1 Pa$^{0.5}$ and at a constant liquid flow rate. These flow rates are varied between 10 and 54.8 m^3/(m^{2}h). During the experiments the operating pressure drop is recorded. After 1390 g of water have been evaporated (corresponding to 500 g of salt-fallout) the experiment is stopped. After a constant drying time of 30 minutes the dry pressure drop of the fouled packing is measured again at a gas flow rate F of 3 Pa$^{0.5}$.

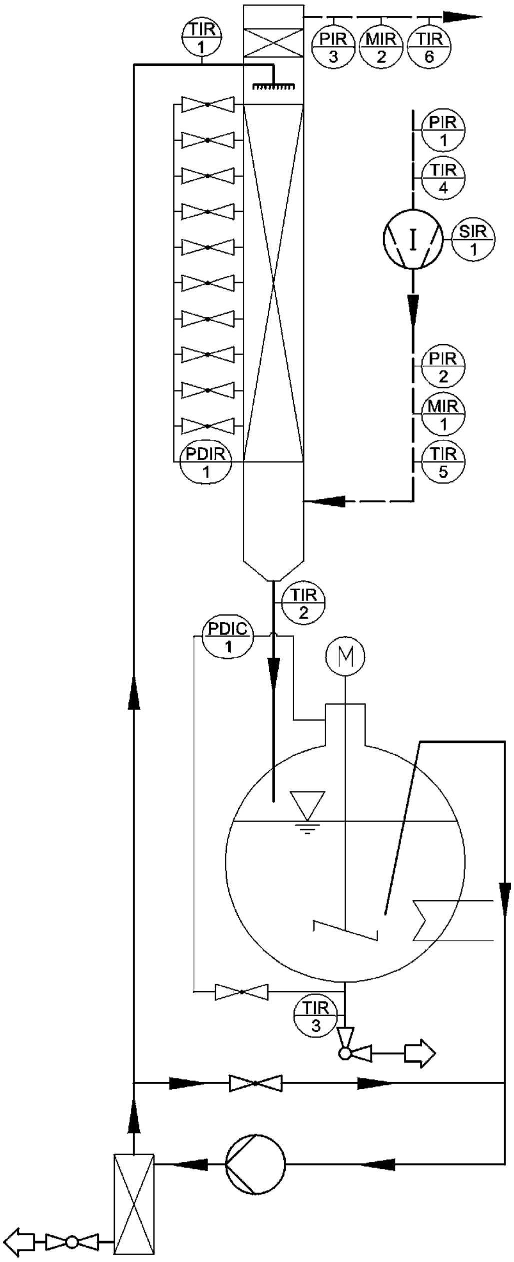

Figure 4: Pilot plant

EXPERIMENTAL RESULTS

Single Ring Results

Figure 5 shows the development of the deposits on the single rings over a period of 45 minutes. The data was obtained at a liquid load B of 40 m^3/(m^{2}h). It is obvious that the ring made of ceramics collects the most deposits. The results also show that the deposition rate is higher than with all other materials. The differences between PP, glass and PTFE are rather small. The deposition rates of PP, steel, glass and PTFE are of about the same order of magnitude. Figure 6 shows the average amount of salt deposits on the rings taken from all experimental data. Analogous to Figure 5, ceramics show the lowest fouling resistance. The deposits are stable and cannot be removed easily. Steel shows only about one fourth of the deposits of ceramics. Moreover, the salt crust is mechanically rather instable. PP, PTFE and glass show only few and unstable deposits.

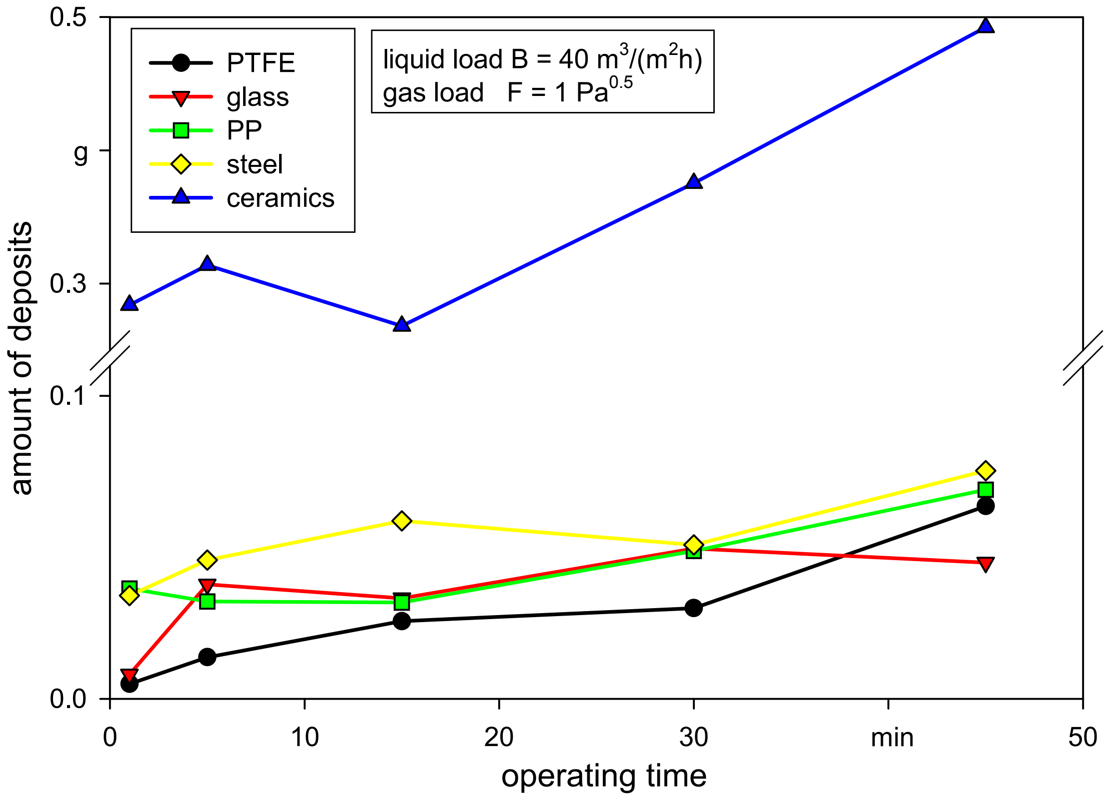

Figure 5: Amount of salt deposits on the single rings over time at a liquid load of 40 $m^3/(m^2h)$

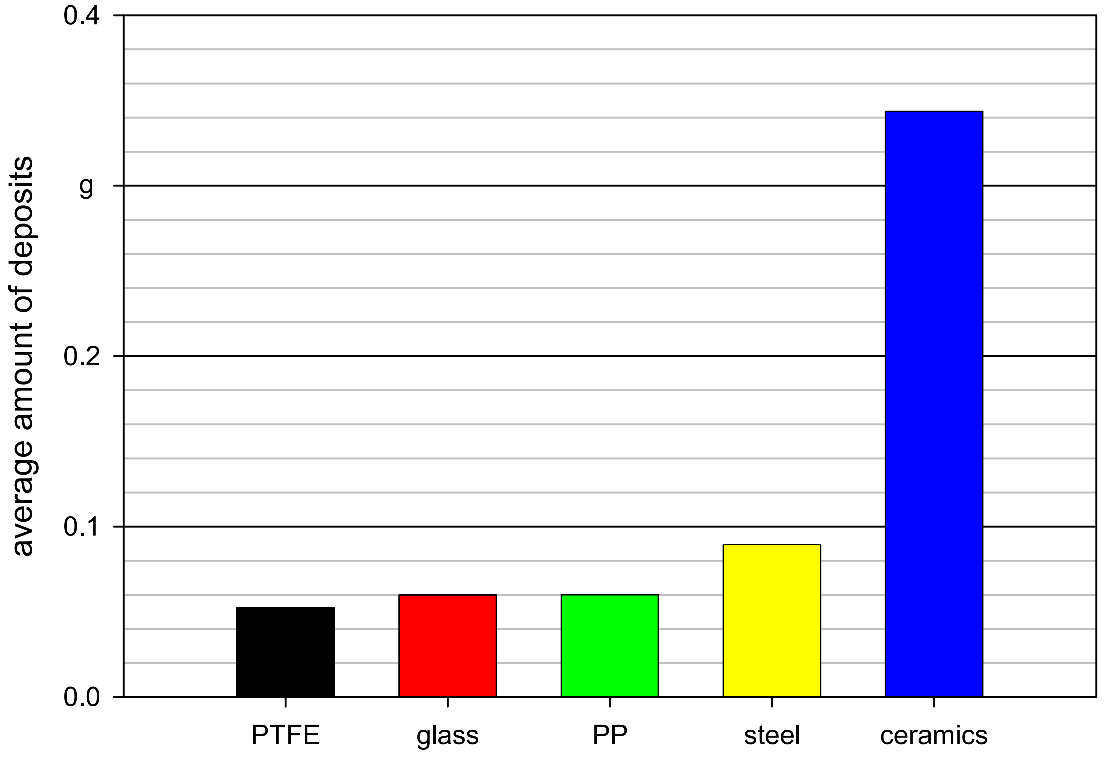

Figure 6: Average amount of salt deposits on the single rings

Pilot Plant Results

Figure 7 shows the operation pressure drop increase of the Sulzer Mellapak 350Y steel packing for the 10 sections. The graph indicates that the deposits are mainly concentrated in the lowest section of the packing. The differences in pressure drop in the sections 2 to 10 stays almost constant over the time of operation. The result is validated with the dry pressure drop of the different sections after the experiment (see Figure 8). Almost all the deposition is collected in the lowest section.

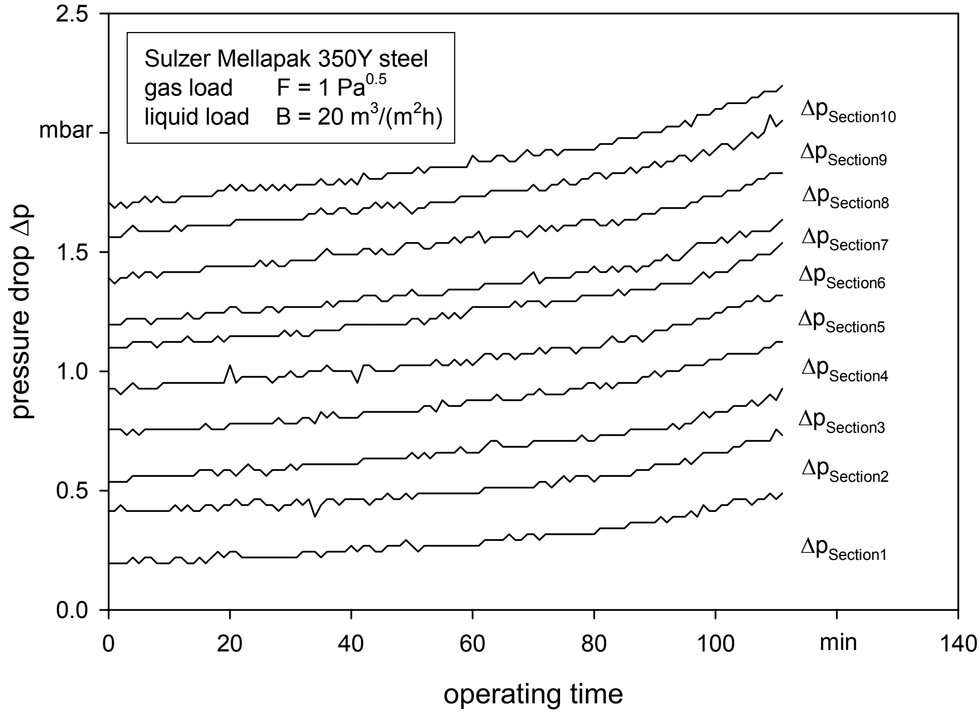

Figure 7: Development of pressure drop over operating time

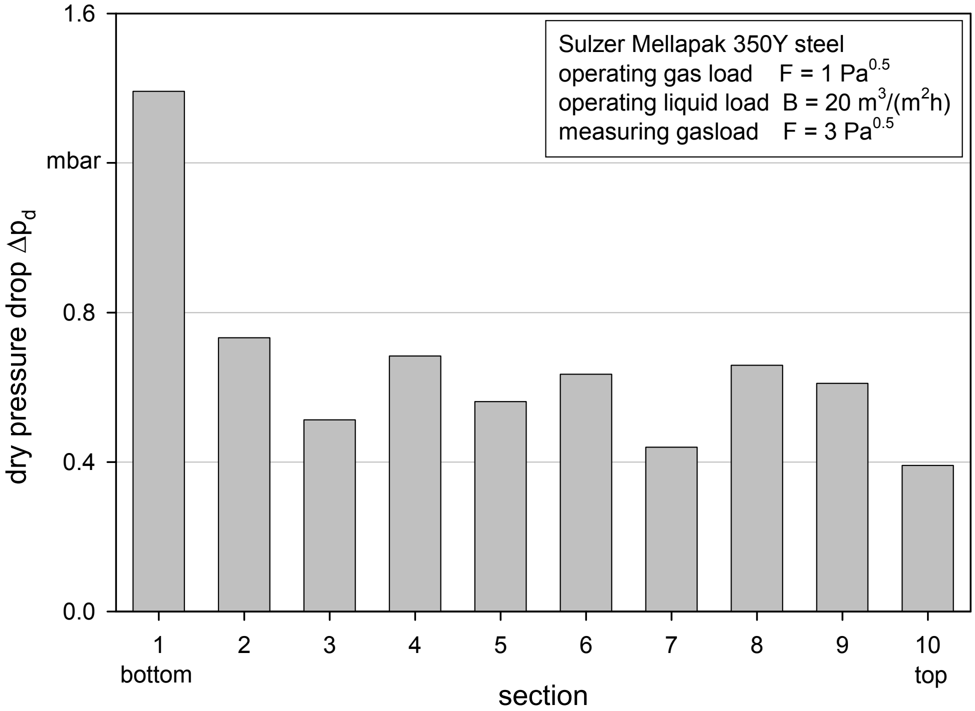

Figure 8: Dry pressure drop of the 10 packing sections

The comparison of the results of the different packings is shown in figure 9. Obviously, both gauze packings show a strong pressure drop increase during the operation. Especially the steel gauze packing plot displays an almost constant increase versus the liquid load. The textile gauze packing ranges in about the same order of magnitude for a liquid load of less than 30 $m^3/(m^2h)$. With higher liquid loads, a significant fraction of the deposits is obviously washed out of this packing.

For liquid loads below 30 $m^3/(m^2h)$ the other 4 packings show a nearly identical performance. For liquid loads higher than 40 $m^3/(m^2h)$, higher pressure drop increases can be observed. In contrast to this, the glass packing's pressure drop stays rather constant for all liquid loads.

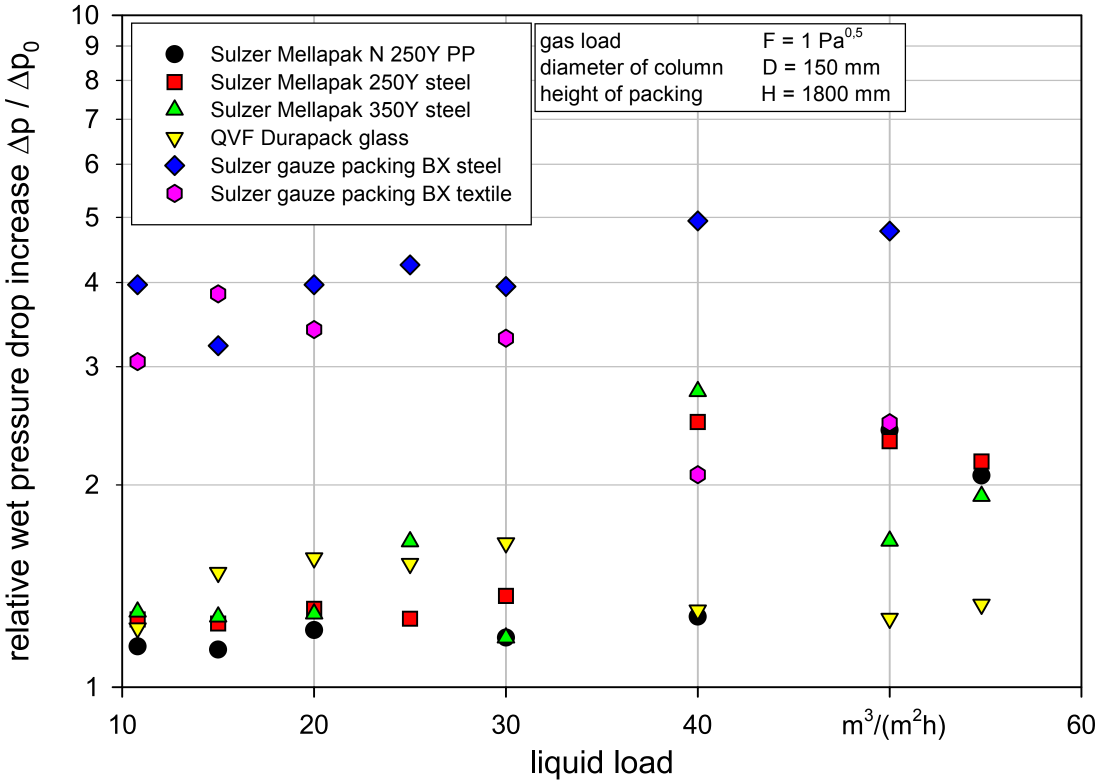

Figure 9: Relative wet pressure drop increase of packings

Additional information can be derived from figure 10, where the dry pressure drop of the fouled packings is refered to that one of the clean packing. Taking into consideration the results shown in figure 9, the overall range of pressure drop ratios for the different types of packings is smaller than presumed.

This indicates that the strong increases of pressure drops during operation are caused by local flooding zones within the packings. This is the case especially for the steel gauze packing where relatively low ratios of the dry pressure drops are observed. The packings Mellapak 250Y, 350Y and N 350 Y show a rather similar behavior. For all three packings liquid loads larger than 30 $m^3/(m^2h)$ lead to large pressure drop increases. As these increases cannot be found in figure 10, it is not possible that the deposits are the only reason for the increases. Rather the deposits cause zones of local flooding with a high pressure drop.

The only packing that shows a consistent plot in both the figure 9 and 10 is Durapack. Due to its plane surface the deposits can only grow to a certain extent. As soon as flooding starts, a significant fraction of the deposits is washed out.

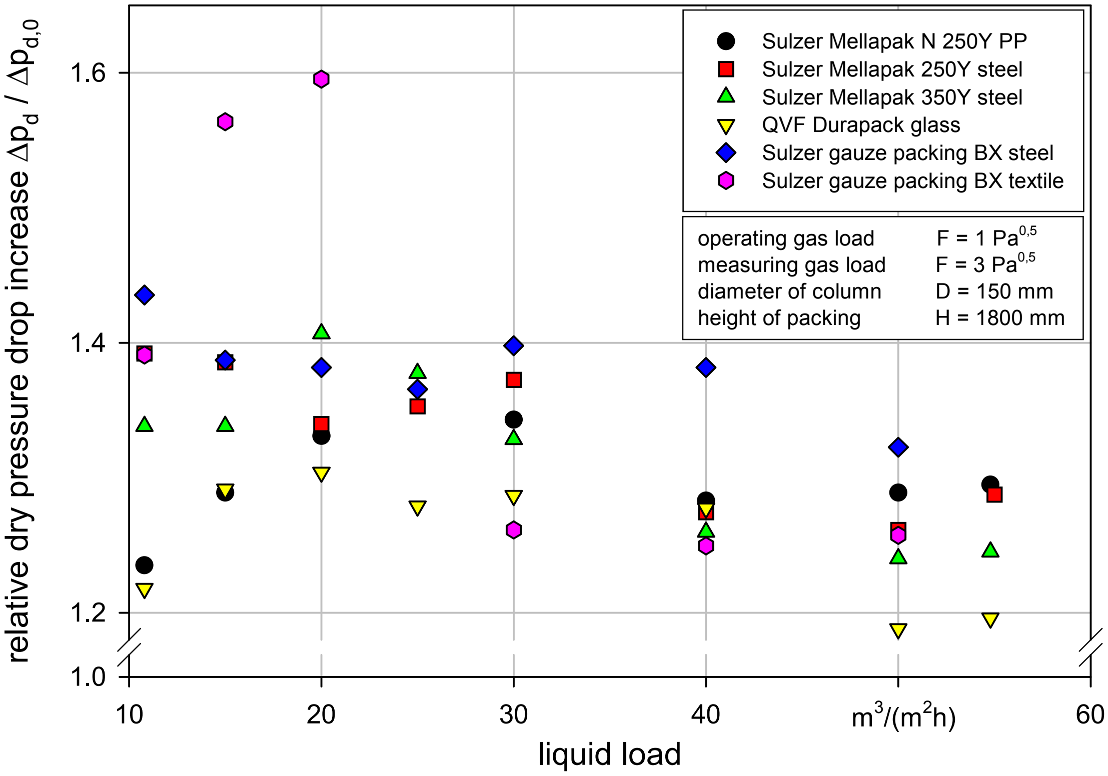

Figure 10: Relative dry pressure drop increase of packings

CONCLUSION

As a result from the single ring experiment it can be stated that porous materials like ceramics are not suitable for fouling applications. Though the investigation was performed with an aqueous solution, the strongly hydrophobic PTFE does not turn out to be as resistant as presumed. Despite its hydrophilic character glass appears to be quite resistant against deposits. Obviously, the roughness of a surface plays a decisive role for fouling.

The main conclusion from the pilot plant experiments is that packings with rough or porous surfaces like gauze packings should not be used for fouling applications. The results coincide well with the ones from the single ring experiments. For some types of packings a low amount of deposits contributes to a strong increase of the liquid holdup. As a consequence local flooding and, thus, strong increases in pressure drop occur. In contrast to this, fouling resistant packings like the durapack do not show flooding during the experiment.

NOTATION

B	liquid flow rate	$m^3/(m^2h)$
D	diameter of column	mm
F	gas flow rate, $F \equiv w_G \cdot \sqrt{\rho_G}$	$Pa^{0.5}$
H	height of packing	mm
Δp	operation pressure drop	mbar
Δp_0	pressure drop at operation start	mbar
Δp_d	dry pressure drop before the experiment	mbar
$\Delta p_{d,0}$	dry pressure drop after the experiment	mbar
w_G	superficial velocity of the gas	m/s
ρ_G	density of the gas	kg/m^3

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