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- Steam sterilization
- Air removal
- Fractionated vacuum
- Dilution factor
- Hollow device
- Process challenge device (PCD)
- Tubular model

A Quantitative Description of Air Removal from Hollow Devices in Steam Sterilization Processes

*D. Kaiser[1] *, U. Kaiser, H. Vogel[1]*

In steam sterilization processes hollow devices can be reliably sterilized only if the air trapped within these hollow devices has been quantitatively replaced by steam. This paper investigates the relationship between the behaviour exhibited by air removal mechanisms in steam sterilization processes and the type of fractionated vacuum used. The test models employed were plastic tubes of varying lengths and diameters. These were closed at one end with a metal capsule, into which a chemical indicator had been fitted. Substitution of air with steam is most difficult at this closed end of the tube. If the indicator positioned here indicates the presence of steam, it can then be assumed that steam has penetrated the entire tube.

The influence exerted by different air removal procedures was investigated in a parametric study.

Spicher et al. (1) devised a function that can be used for a quantitative description of air removal from porous items. The results obtained in this present investigation show that Spicher's approach cannot be applied to hollow devices. From the measurement results a relationship was inferred which describes air removal from hollow devices with diameters of less than 10 mm.

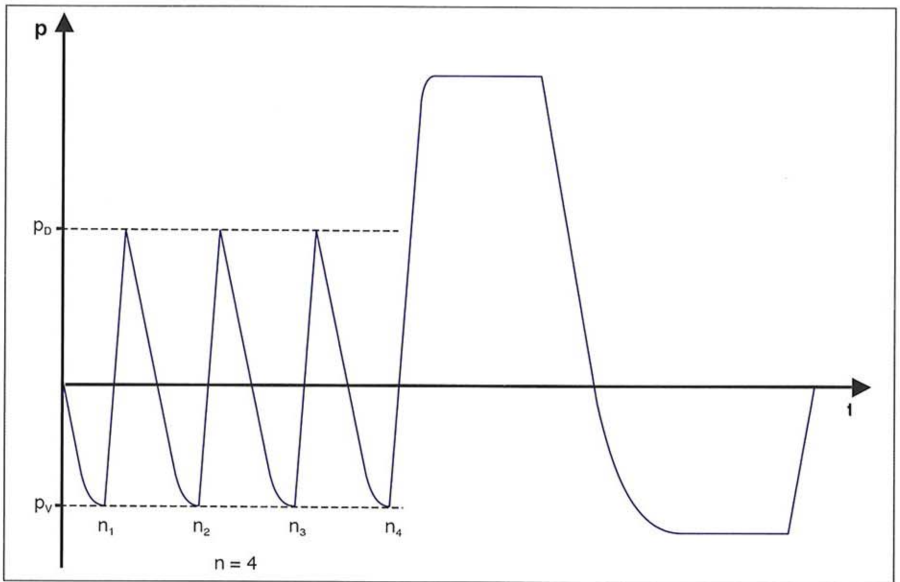


Fig. 1: Description of fictitious dilution factor variables

Introduction

In steam sterilization processes medical devices can be reliably sterilized only if the steam can gain access to all their surfaces. Air pockets or other types of noncondensable gases (NCGs) can impede complete steam penetration (2). To eliminate such impediments, all steam sterilization processes are preceded by air removal procedures of varying degrees of effectiveness:

- Gravity displacement procedures that remove the air only from areas directly exposed to flow, but not from well-wrapped items or hollow devices.
- Over-pressure cycles that are also very effective at removing air from wrapped, solid instruments and porous items (not effective for hollow devices).
- Subatmospheric cycles that are also in addition capable of removing air from intricate hollow devices.
- Combined procedures comprising elements of the methods mentioned above.

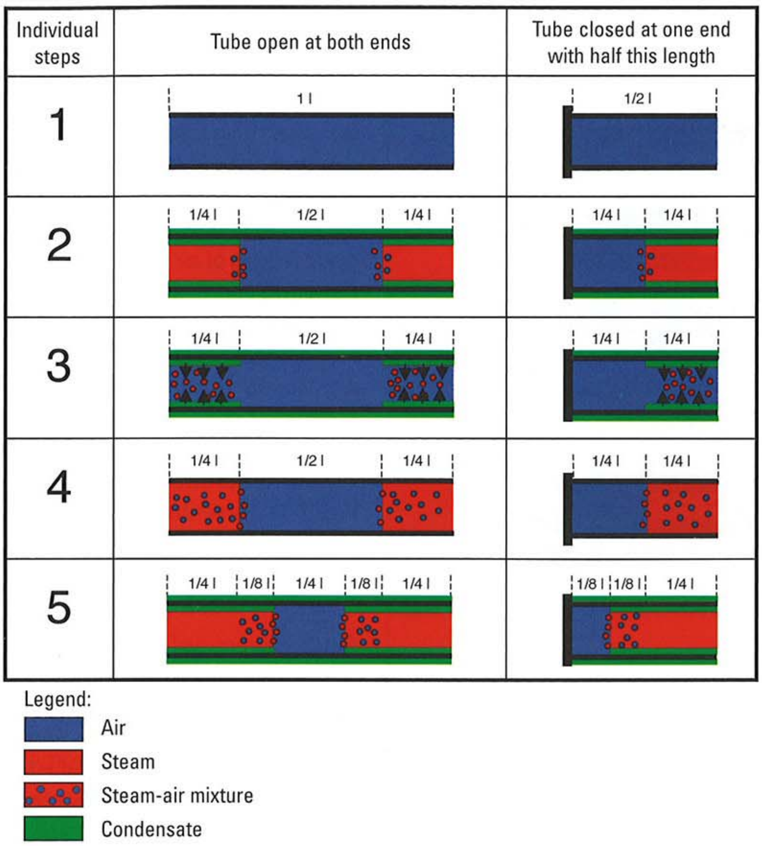
Even in respect of these processes there are major differences depending on the individual process used. Spicher et al. (1) investigated the effectiveness of these air-removal procedures and used a fictitious dilution factor V_f to characterise them.

However, this dilution factor is only valid if good mixing of NCGs and steam is assured through convection.

$$V_f = \frac{p_D}{p_V} \cdot \left(\frac{p_D}{p_V}\right)^{n-1}$$

(Dilution factor devised by Spicher)

1 Technische Universität Darmstadt, Ernst-Berl-Institut für Technische und Makromolekulare Chemie, Petersenstr. 20, 64207 Darmstadt, Germany



Description of individual steps

1. A tube open at both ends has the same air removal behaviour as a tube closed at one end with half the length of the former. Both are filled with air at the beginning of the sterilisation process before steam is supplied (pressure is 1 bar absolute).
2. The volume of air in the tube is reduced to half this amount when steam is introduced (the pressure rises from 1 to 2 bar absolute) and each of the open ends fills with steam, occupying 1/2 of the tube length. Steam condenses on the internal and external walls, heating the tube material.
3. After opening a valve, the positive pressure reverts to normal pressure, while the enclosed air volume decompresses again across the entire tube length in the region with the condensate film.
4. Due to the drop in pressure, the condensate evaporates on the walls, converting into a relatively large amount of steam that mixes with the expanding air and forces out yet more air.
5. During the 2nd compression phase the left air volume is once again reduced by half; and now 1/2 the tube volume is filled with steam and 1/4 with a steam-air mixture.

Fig. 2: Mechanism of steam penetration within tubes opened at one or both ends, when using an over-pressure cycle for air removal

p_0 = *local atmospheric pressure*

p_V = *final pressure obtained after evacuation*

p_D = *final pressure obtained after a steam pulse*

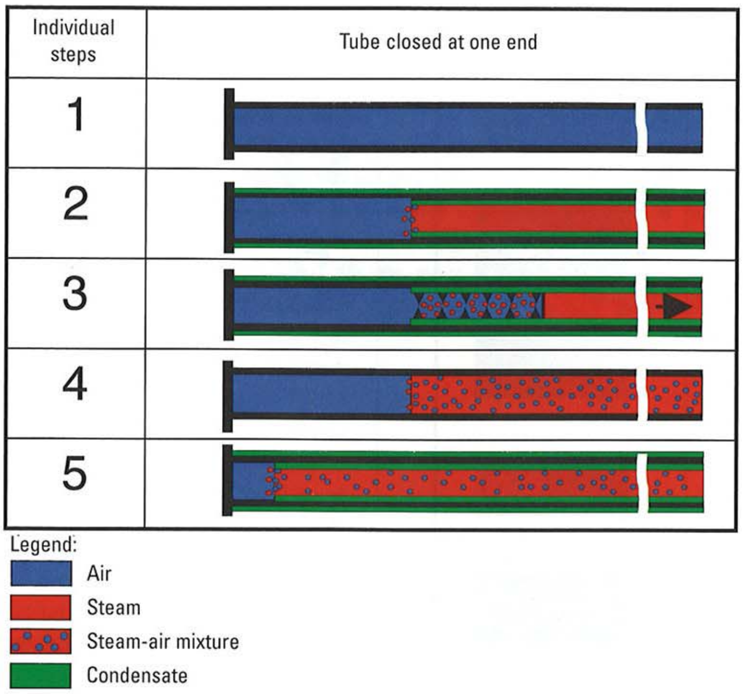
n = *number of vacuum cycles*

The fictitious dilution factor is characterised by the number of air removal cycles and by the upper and lower pressure-change points.

In general, convection assures good mixing within porous items and unoccupied volumes in loaded trays of packets. But within empty chambers stratification of steam and residual air can take place. As a result of stratification within the chamber, the incoming steam will not be able to make contact with any packages located in areas harbouring NCGs, because of poor mixing activity. To prevent this, autoclave chambers with differential pressure processes are equipped with special plates to ensure that the incoming steam creates turbulence. Gravitiy displacement autoclave chambers have a steam inlet at the highest point and an exhaust pipe at the lowest point in the chamber. In this manner the air is forced out of the chamber by the lighter, incoming steam, thus precluding stratification.

In differential pressure procedures, air removal from hollow devices unfolds in a completely different manner from e.g. porous items or empty spaces (3).

A tube that is open at both ends exhibits the same air-removal behaviour as a tube measuring half this length, but closed at one end. If a hollow device that is filled with air is exposed to a steam pulse, the NCGs within will be compressed (4). In view of the relatively small area between NCGs and steam there is only slight turbulence-mediated mixing. The intensity of this mixing process will depend on the flow profile, which in turn is largely determined by the flow velocity. And the flow velocity is a function of the magnitude of the pressure gradient and of the tube length, but is virtually independent of the tube diameter. It is greatest at the tube outlet, whereas it is zero at the geometric mean of a tube open at both ends or at the closed end of a tube. In the case of tubes measuring more than 20 cm in length, mixing activity will be negligible at the area between steam and NCGs, compared with that seen throughout the entire tube length; mainly movement of the gas columns is achieved by compression and decompression. The proportion of axial mixing attributable to turbulence at the boundary surfaces is within the centimetre range. Hence a very large number of compression/decompression cycles would be needed to assure adequate air removal, if such air removal were to be mediated exclusively by turbulence. Spicher's dilution factor cannot be extrapolated to hollow devices in view of the fact that turbulence-mediated mixing of NCGs takes places only at very small boundary surfaces and not within volumes. This is also borne out by the fact that only by performing a very large number of over-pressure cycles are these able to remove air from tubes approximately longer than 50 cm. To use over-pressure cycles is not a practical approach due to the time and energy consumption involved. Using vacuum or transatmospheric cycles, too, turbulence-mediated mixing is not very effective at the areas between steam and residual air. However, here the mixing activity resulting from evaporation of condensate from the inner wall surfaces in ar-



Description of individual steps

1. When the pressure drops from 1000 to 100 mbar, 90% of the air is removed, leaving 10% residual air across the entire tube length.
2. When the pressure is increased from 100 to 1000 mbar as steam is supplied, the residual air is compressed, causing the steam to condense on the tube walls and thus heating them. Now 90% of the tube is filled with steam and 10% with residual air.
3. When the pressure drops from 1000 to 100 mbar, the encapsulated air volume expands again in the region containing the condensate film.
4. The reduced pressure causes the condensate to evaporate on the walls, giving rise to relatively a lot of steam that mixes with the expanding air and forces out yet more air.
5. Increasing the pressure again to 1000 mbar by supplying steam leads to compression of the residual air to 1%, to the air-steam mixture as well as to steam condensation once again on the tube walls, thus heating the walls.

Fig. 3: Mechanism of steam penetration within tubes closed at one end, when using a fractionated vacuum for air removal

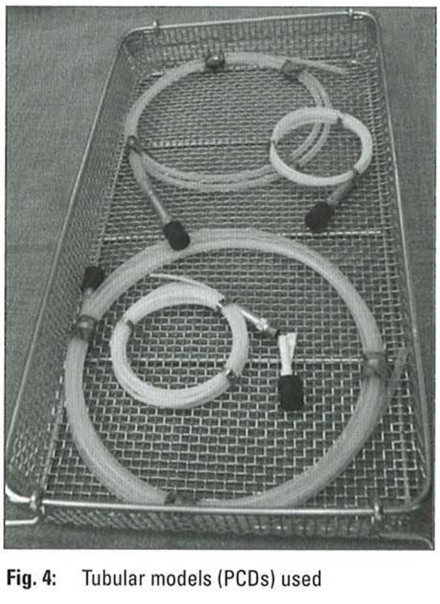


Fig. 4: Tubular models (PCDs) used

eas with residual air is essentially better. Likewise, a large amount of the air initially present in the tube is removed through air expansion on generation of a vacuum, whereas the air within the tube is only compressed if no vacuum is previously generated. This is illustrated in figures 2 and 3.

Kaiser et al. (4), too, have demonstrated in experimental studies that only fractionated vacuum cycles are effective at removing air from longer hollow devices.

Conclusion: Air is removed from hollow devices during fractionated vacuum cycles first by increasing the volume of air within the tube, thus forcing out the biggest amounts. Only to a small extent is there mixing of air and steam at the boundary surfaces. Most of the residual air is removed by steam condensation on the walls, followed by evaporation of the condensate again (see figure 3, step 4). From this mechanism it can be concluded that Spicher's fictitious dilution factor cannot be applied to hollow devices since it calls for good mixing of air and steam without condensation and evaporation. These requirements are not met in the case of hollow devices.

Mixing forced by condensation of steam and evaporation of condensate into the areas with residual air complies with a different mathematical model, which is presented below.

Task Definition

Since air removal and thus steam penetration of hollow devices cannot be explained on the basis of Spicher's fictitious dilution factor, there are no process engineering data for air removal cycles available to remove the air from hollow devices and minimal invasive surgery (MIS) instruments. Today, more than 30% of surgical operations in Western Europe are carried out using MIS techniques. To that effect, MIS instruments, most of which are of the hollow device type, are used.

The aim of the present study was to formulate a process engineering description for air removal from hollow devices analogous to Spicher's fictitious dilution factor, which is a convenient description of air removal from empty spaces and porous items (5).

Experimental Setup

The hollow devices used were 4 tubular models called Process Challenge Devices (PCDs) made of PTFE (1.5 m + 3 m; each 2 mm + 5 mm diameter), which represent typical and difficult to sterilize hollow devices (see table 1 and figure 4). The tubes were closed at one end with a metal capsule, into which a chemical indicator was fitted to proof steam penetration at the closed end.

A test sterilizer described in EN 867-4 and EN-ISO 11140-4 was used. The sterilization cycles could be programmed as required via a PC.

To study the influence of the different air-removal cycles, different sterilization programmes were used, consisting of:

PCD	Length	Diameter
1	1.5 m	2 mm
2	3.0 m	2 mm
3	1.5 m	5 mm
4	3.0 m	5 mm

Table 1: Dimensions of tubular models (PCDs) used

	1st Experim. Series	2nd Experim. Series
Upper pressure-change pt. p_D	1000, 1500 & 2000 mbar	< 3000 mbar
Lower pressure-change pt. p_V	> 20 mbar	50, 100 & 200 mbar
Number of air-removal cycles n	1 to 10	1 to 10

Table 2: Overview of the series of experiments carried out

Fig. 5: Steriliser chamber 6, 6, 6 with a volume of 318 litres

- an air-removal process,
- an exposure time of 3.5 min at 134 °C
- and a subsequent drying phase.

Only the air-removal process was varied for the experiments, and the parameters changed were the number of air-removal cycles n, the lower pressure-change point p_V and the upper pressure-change point p_D (see figure 7).

All rising and falling pressure gradients were kept constant and set to 1.5 bar/min (there were minor deviations caused by the vacuum pump in the region of the deep vacuum). The number of air-removal cycles as well as the upper and lower pressure-change points were varied until complete steam penetration had been assured. Several tests had to be carried out to accomplish this. To begin with, air-removal cycles that were not sufficiently effective at removing air from the tubular models were set. Then the pressure difference was increased in small steps until complete steam penetration was assured. These values are outlined in tables 3 to 8.

Two series of experiments were conducted, with the upper pressure-change point being kept constant in the first series and the lower in the second series (see table 2).

Test Results

The tables 3 to 5 show the test results obtained for the 4 PCDs for 4 different lower pressure-change points p_V and correspondingly adapted upper pressure-change points p_D and exact number of air-removal cycles n needed to assure complete air removal.

The tables 6 to 8 show the measured values obtained using a constant upper pressure-change point, but with variation of the lower pressure-change points and of the number of cycles.

Three equal PCDs were used for each experiment in order to gain insights into the reproducibility. Running up to five air-removal cycles n, the results were eminently reproducible in the same three PCDs. On increasing the number of cycles n > 5, there was a noticeable decline in the reproducibility of the results. On average, five measurements had to be conducted for each result in order to be able to determine the limit value. This means that overall about 1,200 individual measurements had to be carried out with 3 PCDs in each case.

Discussion

When the values obtained for the air-removal parameters n, p_V and p_D are inserted into Spicher's dilution formula, a constant dilution factor for all data sets could not be observed, as it is the case for porous systems, but rather a very scattered data range was obtained. This confirms, as already pointed out in the quali-

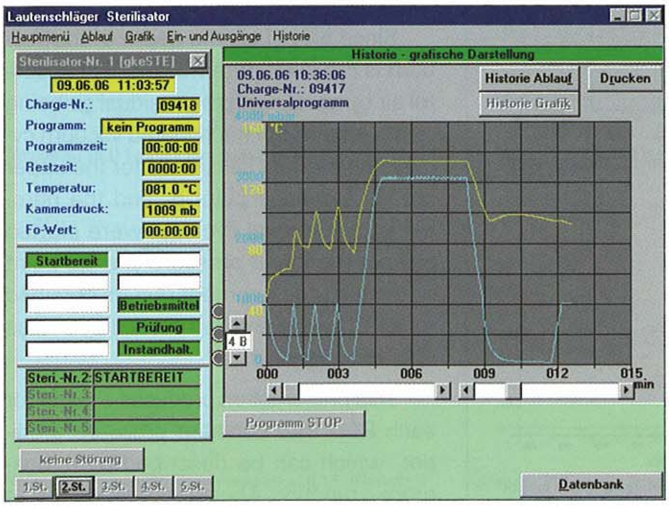


Fig. 6: Screen shot of a programme cycle

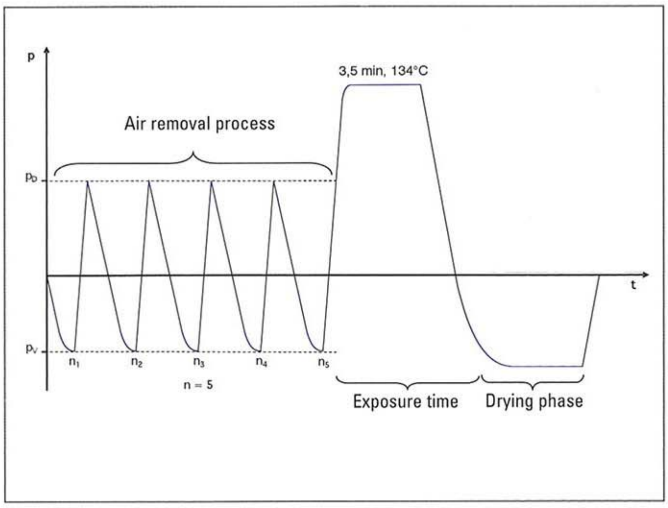


Fig. 7: Design of a sterilisation programme with fractionated vacuum

n	PCD 1 p_V [mbar]	PCD 2 p_V [mbar]	PCD 3 p_V [mbar]	PCD 4 p_V [mbar]
1	20	–	–	–
2	50	40	40	30
3	110	100	60	40
4	170	110	80	60
5	200	150	100	70
6	210	160	110	80
7	220	180	130	90
8	230	210	140	100
9	250	240	150	110

Table 3: Measurement results obtained on varying the lower pressure-change point p_V, while maintaining the upper pressure-change point p_D constant at 1000 mbar

n	PCD 1 p_V [mbar]	PCD 2 p_V [mbar]	PCD 3 p_V [mbar]	PCD 4 p_V [mbar]
2	60	60	40	30
3	160	90	90	60
4	220	130	130	80
5	260	160	140	90
6	290	190	140	100
7	310	210	150	110
8	340	230	160	120
10	400	260	190	120

Table 4: Measurement results obtained on varying the lower pressure-change point p_V, while maintaining the upper pressure-change point p_D constant at 1500 mbar

n	PCD 1 p_V [mbar]	PCD 2 p_V [mbar]	PCD 3 p_V [mbar]	PCD 4 p_V [mbar]
2	70	50	40	30
3	180	160	100	60
4	250	210	150	80
5	300	230	170	90
6	330	260	180	110
7	370	300	190	120
8	400	340	190	130
10	410	380	210	140

Table 5: Measurement results obtained on varying the lower pressure-change point p_V, while maintaining the upper pressure-change point p_D constant at 2000 mbar

n	PCD 1 p_D [mbar]	PCD 2 p_D [mbar]	PCD 3 p_D [mbar]	PCD 4 p_D [mbar]
2	1000	1100	1200	
3	300	300	400	900
4	200	200	200	600
5	100	100	100	300
6	75	100	75	300
7	60	70	70	300
8	60	70	65	200
10	60	70	60	200

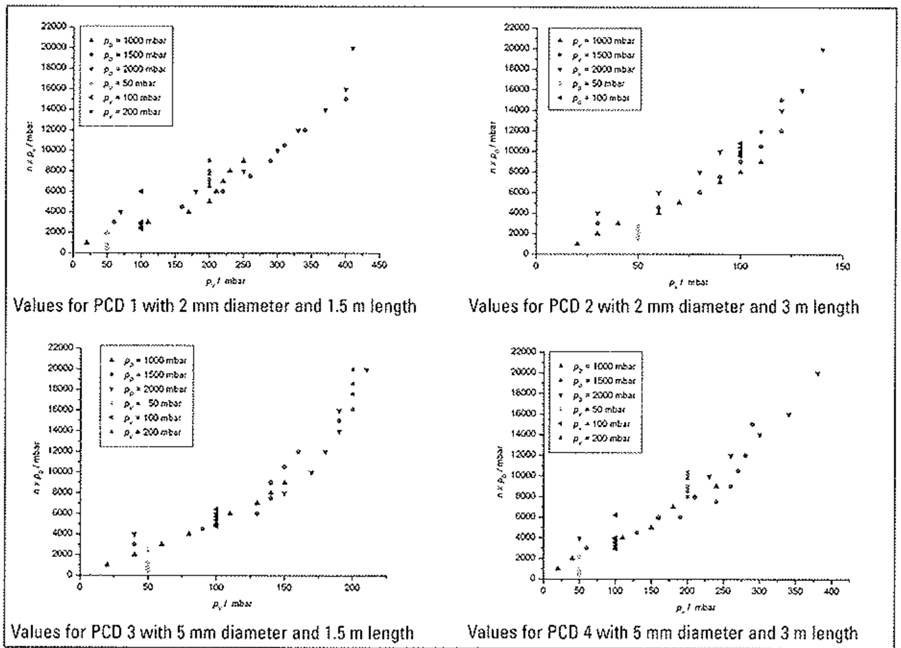
Table 6: Measurement results obtained on varying the upper pressure-change point p_D, while maintaining the lower pressure-change point p_V constant at 50 mbar

n	PCD 1 p_D [mbar]	PCD 2 p_D [mbar]	PCD 3 p_D [mbar]	PCD 4 p_D [mbar]
2	3000	3100		
3	1000	1300	1900	
4	600	1000	1600	2700
5	500	700	1000	2000
6	400	600	900	1600
7	400	500	700	1400
8	350	400	600	1300
10	300	300	600	1000

Table 7: Measurement results obtained on varying the upper pressure-change point p_D, while maintaining the lower pressure-change point p_V constant at 100 mbar

n	PCD 1 p_D [mbar]	PCD 2 p_D [mbar]	PCD 3 p_D [mbar]	PCD 4 p_D [mbar]
3	2200	2900		
4	1700	2000		
5	1300	1700		
6	1200	1500	3100	
7	1100	1400	2300	
8	1000	1300	2200	
10	900	1000	2000	

Table 8: Measurement results obtained on varying the upper pressure-change point p_D, while maintaining the lower pressure-change point p_V constant at 200 mbar



Values for PCD 1 with 2 mm diameter and 1.5 m length

Values for PCD 2 with 2 mm diameter and 3 m length

Values for PCD 3 with 5 mm diameter and 1.5 m length

Values for PCD 4 with 5 mm diameter and 3 m length

Fig. 8: Diagrams of measured results

tative analysis in the introduction to this paper, that Spicher's dilution formula cannot be extrapolated to hollow devices, so as to ascertain the air-removal parameters capable of assuring good air removal.

Since first of all, the depth of the vacuum is the chief determinant of successful air removal, with the residual air being removed through fractionated vacuum cycles, the product calculated for the upper pressure-change point p_D and the number of air-removal cycles n were plotted against the lower pressure-change point p_V. All the measurement results obtained for a PCD were entered into a diagram, as shown in figure 8.

This presentation produces a curve for each PCD at a constant pressure gradient, which can be described by a logarithmic function. The geometric values as from 50 mbar and up to 8 cycles showed

Length L [cm]	Diameter d [mm]	HPR* [cm]	Measured value calculated HPF**/10^{-2}	Measured value calculated A**
150	2	30	0.22	3.33
300	2	60	0.32	3.25
150	5	75	0.49	3.24
300	5	150	0.73	3.23

Table 9: Comparison of HPR and HPF values for tubes with a wall thickness of 0.5 mm
* valid only for PTFE tubes with 0.5 mm wall thickness
**valid only for a pressure gradient of 1.5 bar/min and ≤ 8 air removal cycles

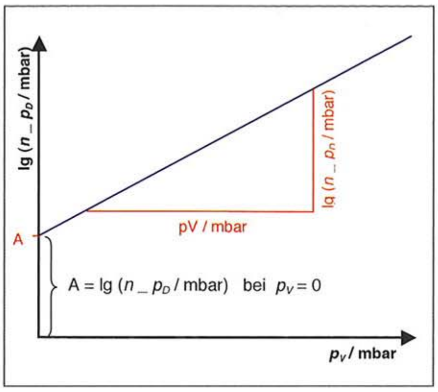


Fig. 10: Diagram showing the straight line equation

a little scatter during tolerance testing. Deviations of more than n > 8 occurred because of the scatter detected between the same PCDs, while deviations below 50 mbar were attributable to the fact that the test sterilizer was no longer able to maintain a constant pressure drop of below 50 mbar.

Logarithmization of the product n x p_D produces a straight line with the equation (see figure 9):

$$\lg(n \cdot p_D/\text{mbar}) = HPF \cdot p_V/\text{mbar} + A$$

n = *number of vacuum cycles*

p_D = *final pressure obtained using steam pulses*

HPF = *Slope of straight line (Hollow Penetration Factor)*

p_V = *final pressure obtained for evacuation cycles*

A = *y-axis section*

Once converted, this gives:

$$HPF = \frac{\lg(n \cdot p_D/\text{mbar}) - A}{p_V/\text{mbar}}$$

Hollow Penetration Factor (HPF) in the region p_V > 50 mbar, n < 8

In respect of the measurement accuracy, the y axis section A is similar for all PCDs tested and has the value A = 3.25. But this value is assured only for the air-removal programme tested so far. It is astonishing that, despite the fact that the air-removal parameters n, p_D and p_V change, the y axis section A remains constant nonetheless. This finding is true at least for a sterilizer with the same pressure gradient and flow profiles.

In contrast to axis section A, the slope differs for each PCD tested, and is called the "Hollow Penetration Factor" (HPF). This is a characteristic variable for air-removal from hollow devices.

By defining the parameters for two air-removal cycles capable of effecting just enough air removal, the HPF can be calculated with the following formula:

$$HPF = \frac{\lg(n_2 \cdot p_{D2}/\text{mbar}) - \lg(n_1 \cdot p_{D1}/\text{mbar})}{p_{V2}/\text{mbar} - p_{V1}/\text{mbar}}$$

The axis section A is calculated after insertion of HPF and a data set for the air-removal parameters with the following formula:

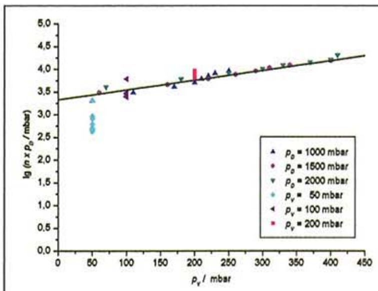
$$A = \lg(n_1 \cdot p_{D1}/\text{mbar}) - HPF \cdot p_{V1}/\text{mbar}$$

If the HPF as well as axis section A are known, the third parameter can be calculated by specifying two of the three air-removal parameters p_D, p_V or n. In this way different fractionated cycles, all of which are capable of effecting air removal from the specified hollow device, can be determined. The HPF value and axis section A always refer to the same pressure gradient and the same flow characteristics within the sterilizer chamber.

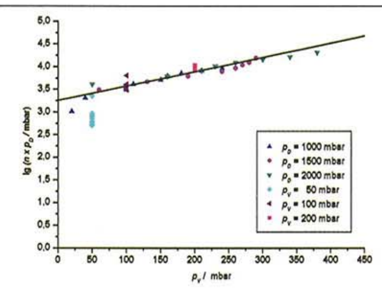
Below axis section A of the converted straight line equation is replaced by the value 3.25 which has been calculated for these experiments:

$$HPF = \frac{\lg(n \cdot p_D/\text{mbar}) - 3{,}25}{p_V/\text{mbar}}$$

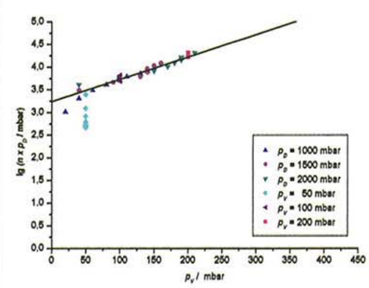
(Valid only under the test conditions used here)



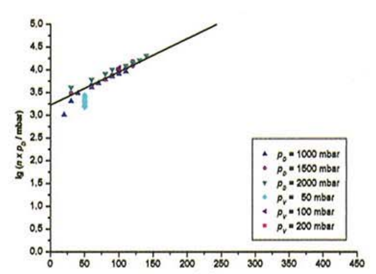
Values for PCD 1 with 2 mm diameter and 1.5 m length



Values for PCD 2 with 2 mm diameter and 3 m length



Values for PCD 3 with 5 mm diameter and 1.5 m length



Values for PCD 4 with 5 mm diameter and 3 m length

Fig. 9: Linearised curve depicting values after logarithmization

Kaiser et al. (4) have demonstrated that different hollow devices made of the same material and with the same wall thickness show the same air removal difficulties under the same air removal conditions. This level of difficulty is expressed by the term "Hollow Penetration Resistance" (HPR) and can be calculated from the product of tube length L and diameter d.

$$HPR = L \cdot d$$

Table 9 shows a comparison of the HPR and HPF values obtained.

Outlook

Since presumably the HPR value and/or the y axis section A are also a function of the pressure gradient, these interrelationships should be further investigated. It may be possible to extend the HPR value on the basis of the material properties, and once a correlation can be established between these data and the HPF, the air-removal effectiveness needed in a fractionated vacuum steam sterilization process can be calculated only if one has knowledge of the tube material and its geometry.

To achieve that goal, the following principles should be further investigated:

- Influence of the pressure gradient on HPF and/or axis section A
- Incorporation of material properties and wall thickness into HPR
- Quantitative relationship between HPR and HPF ❊

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