Investigation and adjustment of the polarographic measuring method ISO 18369-4 for determining the oxygen permeability of silicone hydrogel contact lenses

Summary

Aim: the oxygen permeability coefficient (Dk) of modern silicone hydrogel contact lenses was determined with an optimised polarographic measuring method according to ISO 18369-4.

Attention was paid to the reliability and reproducibility of measuring results. In addition, the determined Dk values were compared with the manufacturers’ information and with measurements published in the literature.

Method

In this study the Dk values of eight commercially available medium to high gas permeable silicone hydrogel (Si-Hy) materials (status: 2008) were compared using the optimised ISO 18369-4 measuring method. The optimised method involves measuring the Dk value by stacking five contact lenses of the same material (S’ = -1.00 D) on the polarographic measuring electrodes. Subsequently the geometric central thickness of the contact lens was determined and the Dk value was calculated. The resulting Dk values were corrected for edge and interface film effect using the calculation method laid down in the ISO standard.

The Dk measurements obtained were calibrated to a reference group comprising two hydrogel and two RGP materials, and the Dk values thus determined were compared with those stated by the manufacturers.

Results

The measuring results of the Dk values (95% confidence interval) are corrected for edge and interface film effect. They are shown in the dimension $10^{-11}$ (cm$^2$/s) x [mlO$_2$/(ml x mmHg)] and (cm$^2$/s) x [mlO$_2$/(ml x hPa)] for the following Si-Hy materials, given below only with the unit of pressure:

<table>
<thead>
<tr>
<th>Material</th>
<th>[mmHg]</th>
<th>[hPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lotafilcon A</td>
<td>106.10 ± 1.88</td>
<td>79.58 ± 1.41</td>
</tr>
<tr>
<td>Comfilcon A</td>
<td>93.37 ± 2.57</td>
<td>70.03 ± 1.93</td>
</tr>
<tr>
<td>Lotrafilcon B</td>
<td>85.47 ± 3.72</td>
<td>64.11 ± 2.79</td>
</tr>
<tr>
<td>Senofilcon A</td>
<td>86.90 ± 2.22</td>
<td>65.18 ± 1.67</td>
</tr>
<tr>
<td>Balafilcon A</td>
<td>74.90 ± 0.28</td>
<td>56.18 ± 0.21</td>
</tr>
<tr>
<td>Sifilcon A</td>
<td>59.23 ± 0.17</td>
<td>44.43 ± 0.13</td>
</tr>
<tr>
<td>Hydrosifilcon A</td>
<td>10.97 ± 0.05</td>
<td>8.23 ± 0.04</td>
</tr>
<tr>
<td>Galysilcon A</td>
<td>57.57 ± 0.53</td>
<td>44.33 ± 0.13</td>
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</table>

<table>
<thead>
<tr>
<th>RGP materials</th>
<th>[mmHg]</th>
<th>[hPa]</th>
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<tbody>
<tr>
<td>Paflufocon D</td>
<td>116.18 ± 3.89</td>
<td>87.14 ± 2.92</td>
</tr>
<tr>
<td>Paflufocon A</td>
<td>70.92 ± 1.91</td>
<td>53.20 ± 1.43</td>
</tr>
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</table>

<table>
<thead>
<tr>
<th>Hydrogel materials</th>
<th>[mmHg]</th>
<th>[hPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Etafilcon A</td>
<td>16.73 ± 0.41</td>
<td>12.51 ± 0.37</td>
</tr>
<tr>
<td>Ocufilcon D</td>
<td>14.27 ± 0.33</td>
<td>10.70 ± 0.25</td>
</tr>
</tbody>
</table>
Conclusion

This study showed that silicone hydrogel contact lenses can be reproducibly measured with the polarographic measuring method. In particular, the results obtained corroborate the practicability of the optimised polarographic measuring method for determining the $D_k$ values of medium to high gas permeable Si-Hy contact lenses. It was also noticeable that the $D_k$ values determined for six of the eight Si-Hy materials measured correlated with the $D_k$ values given by the manufacturers, if edge correction is not taken into consideration.

A complete, and above all, uniform specification of $D_k$ values (in respect of error correction and unit of pressure used) is therefore urgently required to allow realistic comparisons of $D_k$ values.

(Key words: oxygen permeability ($D_k$ value), silicone hydrogel (Si-Hy), oxygen flow, edge correction)

Introduction

An adequate oxygen supply is vital for long-term, physiologically compatible contact lens wearing, in order to avoid functional impairment to the cornea. This is why contact lens (CL) materials are produced which ensure high oxygen permeability ($D_k$ value), such as, e.g., the silicone hydrogel materials. Currently Si-Hy contact lenses have $D_k$ values in the range of 55 to 140 Barrer. It has become customary to use mmHg as the unit of pressure when stating $D_k$ units (also known as Barrer or Fatt units). However, the ISO Norm 18369-4:2006 recommends using hPa units. Since the indication of $D_k$ values in Barrer is widespread and common practice, a gradual switch to the hPa unit and lower numbers is unlikely, even in the longer term.

### Background

Various efforts have been made world-wide to determine the oxygen permeability of medium to high gas permeable contact lenses $D_k$ values. The measurement of Si-Hy materials, either in accordance with ISO standards or modified ISO standards, as well as non-standardised measuring methods has played an important role here. However, this means it is much more difficult to compare the published data on the $D_k$ measurements of the many different products.

Most of the published oxygen permeability values for hydrogel contact lenses were determined according to DIN ISO 9913-1 $D_k$ values, also known as the “Fatt method”. However, the polarographic method ISO 9913-1 $D_k$ values which are exceeded in some Si-Hy materials.

Following the revision of this standard, using DIN EN ISO 18369-4, it has been possible, since 2006, to determine the $D_k$ values of all types of materials in the range from 0 to 145 $D_k$ values.

Since the revised ISO standard of 2006 does not describe any significant differences in implementation compared with ISO Norm 9913-1, this study investigated to what extent Si-Hy materials can be reproducibly measured using the polarographic measuring method or by optimising the process.
Material and Methods

The Dk values were measured according to DIN EN ISO 18369-4:2006, using the polarographic measuring method developed by Prof. Dr. Irving Fatt. In the measuring apparatus a spherical measuring electrode is used which produces measurable electricity proportional to the oxygen flow of a CL material (Fig. 2).

Measurements took place in a water vapour-saturated air chamber at 35 ± 0.5°C. While measuring the oxygen in a CL, the measurement set-up causes two systematic measurement errors which must be considered, the edge and interface film effects (Fig. 3). If the edge is discounted, for example, the Dk measurement is inflated by about 30%. This error is eliminated by applying the edge-correction formula in the standard.

The interface error is cancelled out by measuring the material through several CL thicknesses (stacking technique) so that the proportion of additional resistance is automatically eliminated 1,12.

Applied optimised measuring methodology

Many preliminary investigations were undertaken in order to be able to better assess the influencing factors when determining Dk values. The findings resulted in the optimised polarographic measuring method, involving stacking technique 1.

This method is used instead of measuring CL materials of different thicknesses. In stacking technique 1, five contact lenses of the same material with an S’ = -1.00 D are stacked one above the other in a 15 minute measuring rhythm until the oxygen molecules have diffused through 5 contact lenses to the electrode. Stacking took place with the help of saline. Following a 24 hour hydration phase the geometric CL central thicknesses were measured using an Electronic Thickness Gauge Model ET-1 (RDC). The geometric central thickness is used because with CL S’ = -1.00 D the geometric central thickness corresponds to the average central thickness (t)13. When calculating the Dk value, the corrected t/Dk values were entered in a regression plot against the sum of the stacked central thicknesses and calculated through the reciprocal formation of the slope of the straight line of the Dk value.

Fig. 2  Polarographic measuring method with magnified spherical measuring electrode (courtesy of Wöhlk-Contact-Linsen GmbH)

Fig. 3  Diagram of edge and interface film effect
Study design

In the present study the randomised Dk values of eight medium to high gas permeable Si-Hy products from different manufacturers (Table 1) were measured and compared three times using the optimised polarographic measuring method. By determining the Dk values of two hydrogel and two RGP materials, the Si-Hy could be calibrated against these reference materials. All Dk values were corrected with regard to the edge and interface film effect in accordance with ISO 18369-4:2006.

Results

The following Tables 2 and 3 and Figures 4 to 6 show the results of the analyses. The results of the measurements (Table 2) were calculated with relatively small standard deviations, whereby some of the high gas permeable materials Dk > 60 hPa show a 4 to 8-fold higher standard deviation than the medium gas permeable materials (Dk < 60 hPa). With a probability of 95% the repetition precision of all materials lies in the standardised range of ± 10% and above. The evaluation of the regression analysis (graph of t/Dk against t) showed a high linear correlation for all results with an r = 0,98 and $r^2 = 99\%$ and better.
Figure 4 shows the calibration of the Si-Hy materials measured (blue) with the measured and known reference materials (orange). The linear dependence of the calibration curve shows an $r^2$ of 99.80%, an increase of +0.93 and the intercept with the ordinate ($y$) at +2.89.

The calibration shows clearly that the measured Si-Hy materials lie near the calibration curves and between the reference products. A survey of the measurements compared with the manufacturers’ data in the literature (unit of pressure mmHg and hPa) is shown in Table 3.
Diversity of Dk value specifications

Closer observation of Figure 5 shows that the uncorrected Dk values are about one third greater than the edge-corrected Dk values, and almost twice as much as the corrected Dk value in hPa. Figure 5 therefore illustrates the necessity for a uniform, and above all complete, specification for Dk values, because superficial statements of figures with a supplement "x 10^{-11}", "according to Fatt / ISO" or simply "Dk units" is no longer adequate to be able to make a realistic comparison of different products.

Comparison of the study results and data in the literature

The comparison of the study results with the manufacturers' data given in the literature is particularly interesting. Fig. 6 shows the data in the literature compared with the mean values of the non-edge corrected and edge corrected study results for the different materials (in mmHg). The diagram shows that it is difficult to compare the edge corrected measurements in the study with the manufacturers' data in the literature because the former are much lower. It was only possible to measure Dk values for the
Galyfilcon A and Senofilcon A materials which were similar to the values in the literature.

The uncorrected (not edge-corrected) measurements correlate better with the manufacturers’ data. Very similar and some slightly higher Dk values were determined. It was conspicuous that the Dk value of the Hydrosilfilcon A material was just 20% of that specified by the manufacturer. This might be due to material-related reasons.

Discussion

The results obtained show that Si-Hy materials can be realistically measured with the adjusted polarographic measuring method. One indication for this is that there was only a slight standard deviation, and another, that the Dk values determined were within the desired measuring tolerance (repetition precision ± 10%). Larger relative measuring errors in the high gas permeable Si-Hy materials are probably due to the measuring electrode reacting sensitively to the increased oxygen flow. The siloxane in the CL polymer structure supports the oxygen flow, and thus ensures a greatly increased electrical current flow in the electrode. The applicability of the revised ISO Norm 18369-4:2006 is supported by the always positive linear curves in the Si-Hy and reference materials (regression analysis with r = 0.98 or r² = 99.00 % and higher). In particular, the stacking technique 1 is a method for obtaining high correlations for low to high gas permeable Si-Hy materials.

However, in order to show a high positive correlation, the stacking process must be carried out very exactly. It was therefore an advantage to stack CL (-1.00 D) because of their regular, almost plane cross-section geometries 13. These could be stacked exactly on the measuring electrode, thus minimising the risk of slipping. This resulted in measurements of reproducible Dk values with minimal standard deviations. If our study results are compared with measurements in publications (Table 4), the measured Dk values without edge correction (in mmHg) correspond very closely with those of Compan et al.3, also without edge correction (in mmHg). Compan et al. calculated very similar values for the Lotrafilcon A and Balafilcon A materials with the polarographic method, also using a contact lens stacking technique (S’ = -3.00 D). For the sake of completeness the manufacturers’ Dk values (in mmHg) are included in the third column. It is interesting to note that stacking of CL with -1.00 D results in lesser standard deviations than the -3.00 D CL stacked by Compan et al.

Further comparisons with Dk values in similar publications are not considered here because of the large volume. Similar, and sometimes higher, Dk values were found for some materials, although these results were obtained with a variety of measuring methods and measuring procedures. However, it is interesting to note that some manufacturers 14 currently state the Dk values of their products with the adjunct: “non-edge corrected”. This transparency enables a much better comparison and sets a good example. Accordingly, in Table 5 the manufacturers’ data regarding the “non-edge corrected” Dk values of two materials are compared with the non-edge corrected study results. The correlation of the respective “non-edge corrected” Dk values underlines the practicability of the optimised polarographic measuring method (ISO 18369-4:2006).

<table>
<thead>
<tr>
<th>Material</th>
<th>Dk value (study)</th>
<th>Dk value (Compan et al.)</th>
<th>Dk value (manufacturer)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lotrafilcon A</td>
<td>142.3 ± 3.0</td>
<td>141.0 ± 5.0</td>
<td>140.0</td>
</tr>
<tr>
<td>Balafilcon A</td>
<td>106.9 ± 0.4</td>
<td>107.0 ± 4.0</td>
<td>99.0</td>
</tr>
</tbody>
</table>

Tab. 4 Comparison of Dk value study results with Compan et al. and manufacturers’ data (in Barrer)

<table>
<thead>
<tr>
<th>Material</th>
<th>Dk value (Studie) non-edge corrected</th>
<th>Dk value (Compan et al.) non-edge corrected</th>
</tr>
</thead>
<tbody>
<tr>
<td>Galyfilcon A</td>
<td>73.1 ± 0.69</td>
<td>70.0</td>
</tr>
<tr>
<td>Lotrafilcon B</td>
<td>113.0 ± 5.63</td>
<td>110.0</td>
</tr>
</tbody>
</table>

Tab. 5 Comparison of study and manufacturers’ non-edge corrected Dk values (in Barrer)
Conclusion

In summary, the present study has shown yet again that Si-Hy materials can be realistically measured using the polarographic measuring method. The study results show that, with optimisation, the oxygen permeability of Si-Hy materials can be measured so that they lie within the required measuring tolerances of the norm. Furthermore, the measuring results support the usability of the optimised method for the current product portfolios of medium to high gas permeable CL materials.

Outlook

Further technical development of the electrode is required to be able to measure even higher gas permeable materials in the future. A continuation of this project is also desirable, in order to corroborate the results obtained to date and to measure other contact lens materials. In order to be able to place more credibility on Dk values given, a uniform specification of Dk values stating the pressure unit and error correction is urgently required. This should be defined by the standardisation institutes and applied as a guide line by all contact lens manufacturers. Because ultimately comparisons of Dk values will only be meaningful and afford the necessary comparability for the contact lens specialist when uniformity prevails in the “Dk value jungle”.

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