Effect of long-time immersion of soft denture liners in water on viscoelastic properties

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Aim of this study was to investigate the effect of long-time immersion of soft denture liners in 37°C water on viscoelastic properties. Six silicone-based and two acrylic resin-based soft denture liners were selected. Cylindrical specimens were stored in distilled water at 37°C for 6 months. Viscoelastic properties, which were instantaneous and delayed elastic displacements, viscous flow, and residual displacement, were determined using a creep meter, and analyzed with 2-way analysis of variance and Tukey’s comparison (α=0.05). Viscoelastic properties and their time-dependent changes were varied among materials examined. The observed viscoelastic properties of three from six silicone-based liners did not significantly change after 6-month immersion, but those of two acrylic resin-based liners significantly changed with the increase of immersion time. However, the sum of initial instantaneous elastic displacement and delayed elastic displacement of two acrylic resin-based liners during 6-month immersion changed less than 10%, which might indicate clinically sufficient elastic performance.

Keywords: Soft denture liners, Viscoelastic properties, Storage in water, Silicone-based liner, Acrylic resin-based liner

INTRODUCTION

Acrylic resins are the preferentially used denture base materials. Denture base acrylic resins do not exhibit shock absorbance capability upon loading of the denture. Thus, as a result occlusal impact forces are directly transmitted to the underlying tissues through the denture. In case of non-uniform distribution of the occlusal force on the thin mucosal tissue covering the alveolar ridge, the biting force may result in pain sensations and advancing alveolar bone absorption. Resilient denture liners are frequently used when complete denture wearers suffer from the symptoms mentioned, and clinical data have proven that soft denture liners improve the masticatory function when compared with conventional hard denture bases1-7.

Many commercial soft denture liners have been made available on the dental market. They can be categorized as acrylic resin-based and silicone-based materials, and their main characteristics vary according to the chemical type8. The soft denture liner must fulfill several essential requirements, such as high biocompatibility, good dimensional stability, permanent softness, compliance, viscoelasticity, strong bonding to the hard denture base, low water sorption and water solubility and color stability9. There is however no ideal soft lining material available, complying with all the requirements. In addition, initial characteristics of soft denture liners change often with time in service in the oral environment12,4,8. Several test methods have been used to evaluate viscoelastic properties, such as oscillating type and cone plate type rheometers9, dynamic mechanical analyzer10,11 and creep meter12,13. A schematic drawing of a creep meter is illustrated in Fig. 1. The properties obtained with the creep meter are interpreted as characteristics of the Voigt four-element model. A typical time-displacement curve obtained with the creep meter is shown in Fig. 2. The elastic deformation using this test is recorded as instantaneous and delayed elastic displacement, and the plastic deformation is recorded as the viscous flow and residual displacement. Viscous flow occurs during loading and unloading, whereas the residual displacement is the permanent deformation. The instantaneous elastic displacement of the indenter probe is h1, where the time-displacement curve deviates from the initial straight line. The time-displacement curve becomes linear again with the load continuously applied. The instantaneous elastic displacement determined with the present device is considered the elastic deformation of the serial spring (S1 in Fig. 2). The displacement of h2 is the intersection of the second linear line with the ordinate axis at time zero. The displacement at h3 is recorded 60 s after the load is applied. The delayed elastic displacement is h2-h1 and describes the elastic deformation superimposing the onset of the plastic deformation following the initial elastic deformation. The delayed elastic displacement is considered the elastic deformation of the parallel-connected spring (S2) and dashpot elements (D2). Thus, the viscous flow is the deformation h2-h3. The viscous
Fig. 1 Schematic drawing of the creep-meter used in this study.

Fig. 2 Voigt four-element model and typical time-displacement curve obtained by creep meter. $S_1$ and $D_1$ are the serial spring and dashpot elements, respectively. $S_2$ and $D_2$ are the parallel-connected spring and dashpot elements, respectively. $h_1$: instantaneous elastic displacement; $h_2$: delayed elastic displacement; $h_3$: viscous flow; $h_4$: residual displacement.

flow and residual displacement are measures of the plastic deformation induced by the serial dashpot ($D_1$). The displacement recorded 60 s after removal of the load is the residual displacement $h_4$.

Several authors reported gradually changes in viscoelastic behavior, solubility, color, and bonding efficiency to acrylic resin denture base material with increasing time in service. According to investigations of Murata et al.,\textsuperscript{11} acrylic resin-based soft liners exhibited more viscous properties and became stiffer with time when compared with silicone-based materials. The viscoelasticity of silicone material did not change significantly even after 3 years storage. Abe et al. reported that the dynamic viscoelastic properties of silicone-based liners significantly changed until 30 days, and stabilized after 60 days upon wet storage\textsuperscript{10}. Dinckal et al. investigated water sorption and solubility during 16-week immersion of soft liners in artificial saliva. Irrespective of the type, acrylic resin-based or silicone-based, all soft liners tested showed both water sorption and solubility, which were approximately 0.9–1.2 and 0.7–3.4%, respectively\textsuperscript{15}. Elution of plasticizer components during storage in water is one of the more serious events, affecting the viscoelastic properties of soft denture liners. Previous research reports of soft denture liners’ characteristics evaluated after one-day to three-year immersion in water comprised mostly only a few products\textsuperscript{11,13,16–18}.

Aim of this study was to investigate the effect of 6-month immersion of 8 soft denture liners in 37°C water on their viscoelastic properties. The null hypothesis was that irrespective of the type of soft denture liners examined there was no significant effect of immersion of soft denture liners in water on viscoelastic properties.

MATERIALS AND METHODS

Eight soft denture lining materials, six silicone-based and two acrylic resin-based types were selected for this investigation. Table 1 shows the materials examined, their manufacturers, the curing types and the compositions, as publicly made available from the respective manufacturers. All materials were processed strictly following the manufacturers’ instructions for use.

Specimen preparation

From each material fifty cylindrical specimens, 10 mm in diameter and 10 mm in height were produced for investigation of viscoelastic properties. Specimens of the heat-cured type soft denture liner were heat-polymerized (DMP: 100°C for 120 min, DPS: 100°C for 15 min, NPS: 100°C for 60 min following 70°C for 30 min) in gypsum molds. Cylindrical silicone rubber patterns of the dimensions mentioned above were embedded in a flask with gypsum. After setting of the gypsum the silicone patterns were removed and the resulting molds were packed with the dough of the acrylic resin-based denture liner. Following heat polymerization according to the manufacturers’ instructions and cooling to room temperature, the specimens were removed and immersed into distilled water.

The cold-cured type soft denture liner specimens were produced in a 10 mm in diameter and 10 mm in height cylindrical split Teflon mold, placed on a Mylar strip covered glass plate. The mold was then filled with the mixed silicone-based product, covered with the Mylar strip and pressed flush with a glass plate. The mold was finally placed for 10 min in an incubator (TVN480DA, Advantec, Tokyo, Japan) at 37±0.2°C before the specimen after cooling to room temperature was removed. The specimens of the heat-curing product DMP were produced following the procedure described above for the acrylic resin-based liner.
Table 1  Materials examined

<table>
<thead>
<tr>
<th>Brand name</th>
<th>Manufacturer</th>
<th>Code</th>
<th>Type</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicone-based material</td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Molloplast B</td>
<td>Detax, Germany</td>
<td>DMP</td>
<td>Silicone-based</td>
<td>a, ω-Divinylpolydimethylsiloxane</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(Heat cured)</td>
<td></td>
</tr>
<tr>
<td>GC Reline Soft</td>
<td>GC, Japan</td>
<td>GRS</td>
<td>Silicone-based</td>
<td>Base: Hydrogen polysiloxane, Silica, Vinyl polysiloxane</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(Heat cured)</td>
<td>Catalyst: Vinylpolydimethylsiloxane, Platinum catalyst</td>
</tr>
<tr>
<td>GC Reline Ultra Soft</td>
<td>GC</td>
<td>GUS</td>
<td>Silicone-based</td>
<td>Base: Vinyl-polysiloxane, Silica</td>
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<td></td>
<td></td>
<td></td>
<td>(Cold cured)</td>
<td>Catalyst: Vinyl-polysiloxane, Silica</td>
</tr>
<tr>
<td>Sofreliner Tough Medium</td>
<td>Tokuyama Dental, Japan</td>
<td>TSM</td>
<td>Silicone-based</td>
<td>Base: a, ω-Divinylpolydimethylsiloxane, Silica</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(Cold cured)</td>
<td>Catalyst: Poly(methylhydrosiloxane), Platinum catalyst</td>
</tr>
<tr>
<td>Sofreliner Tough Super soft</td>
<td>Tokuyama Dental</td>
<td>TSS</td>
<td>Silicone-based</td>
<td>Base: a, ω-Divinylpolydimethylsiloxane, Silica</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(Cold cured)</td>
<td>Catalyst: Poly(methylhydrosiloxane), Platinum catalyst</td>
</tr>
<tr>
<td>Ufi-Gel SC</td>
<td>VOCO, Germany</td>
<td>VUG</td>
<td>Silicone-based</td>
<td>Vinyl and SiH-functional polydimethylsiloxanes, platinum catalyst</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>(Cold cured)</td>
<td></td>
</tr>
<tr>
<td>Acrylic resin-based material</td>
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<tr>
<td>Perma Soft</td>
<td>Dentsply, USA</td>
<td>DPS</td>
<td>Acrylic-based</td>
<td>Powder: PEMA</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>(Heat cured)</td>
<td>Liquid: Dibutyl phthalate, Ethyl acetate, Ethanol</td>
</tr>
<tr>
<td>Physio soft rebase</td>
<td>Nissin, Japan</td>
<td>NPS</td>
<td>Acrylic-based</td>
<td>Powder: PMMA</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>(Heat cured)</td>
<td>Liquid: Methacrylic acid ester, Fatty acid ester, Dimethacrylate</td>
</tr>
</tbody>
</table>

All specimens were stored in distilled water at 37±0.2°C for 1 day, 1 week, 1 month, 3 months and 6 months. For each soft denture lining material 10 specimens for each time storage period were produced.

Measurement of viscoelastic properties
Viscoelastic properties were measured using a creep meter (Rheoner RE3305S, Yamaden, Tokyo, Japan). The specimen was loaded with a 3-mm in diameter ball-shaped probe at a crosshead speed of 1 mm/s until the final load of 1.96 N (200 gf) was reached and kept constant for 60 s. After 60 s the load was removed and the displacement of the probe was continuously recorded until 120 s reckoned from the start of loading. Based on these recorded changes the viscoelastic parameters, instantaneous elastic displacement, delayed elastic displacement, viscous flow and residual displacement were calculated using the Voigt four-element model with a software program (Creep Analysis Ver.1.3, Yamaden).

Statistical analysis
The data for each of the determined parameters, instantaneous elastic displacement, delayed elastic displacement, viscous flow and residual displacement, were analyzed with 2-way analysis of variance (ANOVA) and Tukey’s comparison (α=0.05) using statistical software (JMP ver.11.2.0. SAS Institute, Cary, NC, USA).

RESULTS
The viscoelastic properties following water storage at the five different time points are shown in Figs. 3 to 6. Among the four properties determined, the instantaneous elastic displacement was dominant for all soft denture liners except for NPS. Two-way ANOVA of the four properties revealed that the two main factors, products and storage periods, and their interaction were significant.

The instantaneous elastic displacements after 1-day storage were from 0.52 to 1.31 mm; those after 6-month storage were from 0.40 to 1.23 mm (Fig. 3). The instantaneous elastic displacement of all products except for DMP, TSS and TSM decreased significantly after 6-month storage in water.

The delayed elastic displacement except for NPS was the second greatest deformation following the instantaneous elastic displacement. The delayed elastic displacements after 1-day storage were from 0.04 to 0.50 mm; those after 6-month storage were from 0.01 to 0.63 mm (Fig. 4). The delayed elastic displacements of the silicone-based liner did not change throughout the 6-months storage time, but the acrylic resin-based liner significantly increased. For NPS, the values of the delayed elastic displacement were larger than those of the instantaneous elastic displacement after 1-, 3- and 6-months of storage in water.

The values for viscous flow after 1-day storage were
Fig. 3 Changes of instantaneous elastic displacement after water storage. Values with the same letters are not significantly different \((p>0.05)\).

Fig. 4 Changes of delayed elastic displacement after water storage. Values with the same letters are not significantly different \((p>0.05)\).

Fig. 5 Changes of viscous flow after water storage. Values with the same letters are not significantly different \((p>0.05)\).

Fig. 6 Changes of residual displacement after water storage. Values with the same letters are not significantly different \((p>0.05)\).

from 0.01 to 0.14 mm; those after 6-month storage were from 0.00 to 0.13 mm. The values of the acrylic resin-based liner were significantly larger than those of the silicone-based liner. The viscous flow data throughout the 6-months storage did not significantly change except for DPS that decreased significantly after 1-week storage, without further changes until 6-month.

The residual displacements after 1-day storage were from 0.01 to 0.22 mm; those after 6-month storage were from 0.00 to 0.22 mm (Fig. 6). The residual displacements throughout the 6-month storage period for the silicone-based liner were not significant, whereas those for DPS significantly decreased until 1-month storage, and that for NPS significantly increased until 1-week storage.

**DISCUSSION**

The results of the present study showed that the viscoelastic properties of soft denture lining materials varied depending on the products and that changes of the viscoelastic properties by storage time were different among the products. Therefore, the null hypothesis that irrespective of the type of soft denture liners examined there was no significant effect of immersion of soft denture liners in water on viscoelastic properties has to be rejected.

This study evaluated the effect of immersion in water for 6 months. There are varieties of storage media\(^{14,17}\), but immersion in 37°C water is the most common environment because it is easy, reproducible, and useful. The testing device used was developed for industrial purposes, presumably mainly for use in quality control. Therefore, the manufacturer made no effort to design the machine in an attempt to simulate the clinical oral conditions. The load and the loading time are arbitrarily selected. Even if the device were adjustable, clinical information on the settings are not available. In spite of these limitations, this device may be useful to produce relative data or product rankings that might be relevant for the assessment of soft denture lining products.

The instantaneous elastic displacement of DMP, TSS and TSM did not change with increase of immersion
time, whereas the others decreased. The delayed elastic displacements of silicone-based liners did not change with time but those of acrylic resin-based liners increased. The viscoelastic properties of DMP were almost same as reported previously. The viscous flow and residual displacement of the silicone-based liner examined were almost identical and did not change with time. However, the residual displacement of acrylic resin-based liners was larger than the corresponding viscous flow and changed with time. Moreover, the delayed elastic displacement, viscous flow and residual displacement of acrylic resin-based liners were significantly larger than those of the silicone-based liners. These results agreed with that the elastic recovery of silicone-based liners required 1–2 s but that of acrylic resin-based liners more than 30 s. These results suggested that the silicone-based liner comprise well cross-linked siloxane structures. Therefore, the silicone-based liner is a predominantly elastic compound with rather small viscous flow. On the other hand, the acrylic resin-based liner is a thermoplastic resin containing plasticizers that weaken the intermolecular bonding. This structure might be the reason why the acrylic resin-based liner is more viscous with a resulting smaller elastic recovery after deformation than the silicone-based liner.

Elastic properties of soft denture liners are classified by Shore A hardness at 24 h and 28 days according to ISO 10139-2: “Dentistry-Soft lining, materials for removable denture —Part 2: Materials for long-term use”. According to the manufacturers’ information, GUS and TSS are classified as Type B (extra soft), DPS is not specified, and the remaining products tested as Type A (soft) liners. The Shore A hardness recorded 5 s after loading is the criterion used for this classification. Therefore, the instantaneous elastic displacement is related to the Shore A hardness determined after 5 s. The manufacturers’ information of GUS and TSS as type B is in accord with the larger instantaneous elastic displacements recorded at 1 day and 1 week. However, after 1 month of storage in water GUS exhibited a similar elastic displacement as the other type A products tested. Generally, water absorption and elution of plasticizers are presumably responsible for the changes observed after immersion in water. Acrylic resin-based soft denture liners contain plasticizers such as dibutyl phthalate and fatty acid ester, which will elute during storage in water. Therefore acrylic resin-based soft denture liners will partially lose their instantaneous elasticity with time.

When the sums of instantaneous and delayed elastic displacements were calculated as the elasticity, the changes of elasticity of the acrylic resin-base liners, DPS and NPS, during 6 months were 0.14 and 0.08 mm, which were approximately 10% of the elasticity. On the other hand, some silicone-based liners also partially lost their elasticity with time. The changes of the elasticity of some cold-cured type silicones, GUS, VUG, and GRS, were 0.53, 0.27 and 0.13 mm, respectively, which were more than 20%, whereas those of the remaining cold-cured type silicones, TSS and VUG, were less than 0.1 mm. The heat-cured type silicone, DMP did not change elasticity. Similar decreases of the elasticity were observed in silicones for maxillofacial prostheses. These changes were considered due to additional cross-linking during 37°C water immersion.

Time-dependent changes of the elasticity of two acrylic resin-based liners and three silicone-based liners did not change dramatically. Thus, these soft denture liners were considered having stable elastic properties even after 6-month immersion which might indicate clinically sufficient elastic performance.

The test method used overemphasizes the clinically occurring duration of loading of a denture and the magnitude of stress transferred. Noteworthy, the thickness of the specimens used is very different from the soft liner thickness under a denture base. Therefore, the deformations encountered after short-time loading of a lined denture are presumably very low and practically not significant. The method used serves primarily as a screening tool to discriminate among different brands of soft denture liners. The direct clinical relevance of the test and thus, the differences found among the materials tested are certainly a matter of dispute. Anyway, high instantaneous displacement and short delayed elastic recovery times are desirable characteristics of soft denture liners that should guide the practitioner in selecting appropriate materials.

Several unfavorable changes after longer usage might happen such as discoloration, colonization of microorganisms, and odor. Therefore, future studies are requested to evaluate how long the soft denture liner could be used in oral cavity without adverse effects.

CONCLUSIONS

Within the limitations of this study, it is concluded that:

1. Viscoelastic properties and their time-dependent changes upon storage in water were varied among six silicone-based and two acrylic resin-based soft denture liners.
2. The delayed elastic displacement, viscous flow, and residual displacement of the acrylic resin-based liners were significantly larger than those of the silicone-based liners.
3. The viscoelastic properties of three of the six silicone-based liners did not significantly change after 6-month immersion, whereas those of the acrylic resin-based liners significantly changed with increasing time of storage in water.
4. The change of the sums of instantaneous and delayed elastic displacements encountered for two acrylic resin-based and three silicone-based liners throughout the 6 months of storage were less than 10%.

CONFLICTS OF INTEREST

The authors declare no potential conflicts of interest to this study.
ACKNOWLEDGMENTS
The authors appreciate GC, Tokuyama Dental, and Voco for donating their products for this study. The authors thank Prof. Werner J Finger for discussions and preparation of this article.

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