Shore hardness and tensile bond strength of long-term soft denture lining materials

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Statement of problem. Reduced softness and separation from the denture base are the most significant problems of long-term soft lining materials.

Purpose. The purpose of this study was to evaluate the durometer Shore A hardness and tensile bond strength of long-term soft denture lining materials and to investigate the correlation between these 2 properties.

Material and methods. A group of 7 soft lining materials, 6 silicone based (Dentusil, GC Reline Soft, GC Reline Ultrasoft, Mucopren Soft, Mucosoft, Sofreliner Tough) and 1 acrylic resin based (Durabase), were evaluated for durometer Shore A hardness and tensile bond strength to heat-polymerized denture base resin (Lucitone 199). A specially designed split mold and loading assembly with a swivel connector were used for the durometer Shore A hardness test and tensile bond strength test to improve accuracy and facilitate measurement. Three specimens of each product were stored in a 37°C water bath, and durometer Shore A hardness tests were carried out after 24 hours and 28 days. A tensile bond strength test was carried out for 10 specimens of each product, which were stored in a 37°C water bath for 24 hours before the test. Repeated-measures ANOVA, the Kruskal-Wallis and Duncan multiple range tests, and the Spearman correlation were used for statistical analyses.

Results. The repeated-measures ANOVA found significant durometer Shore A hardness differences for the materials (P<.001) and the interaction effect (aging×materials) (P<.001). GC Reline Ultrasoft showed the lowest mean durometer Shore A hardness (21.30 ± 0.29 for 24 hours, 34.73 ± 0.47 for 28 days), and GC Reline Soft showed the highest mean durometer Shore A hardness (50.13 ± 0.48 for 24 hours, 57.20 ± 0.28 for 28 days). The Kruskal-Wallis test found a significant difference in the mean tensile bond strength values (P<.001). GC Reline Ultrasoft (0.82 ± 0.32 MPa) and Mucopren Soft (0.96 ± 0.46 MPa) had a significantly lower mean tensile bond strength (P<.05). GC Reline Soft had the highest mean tensile bond strength (2.99 ± 0.43 MPa) (P<.05), and acrylic resin-based Durabase showed a significantly different tensile bond strength (1.32 ± 0.16 MPa), except for Mucopren Soft, among the materials (P<.05). The tensile bond strength and Shore A hardness showed a statistically insignificant moderate positive correlation (r=0.571, P=.180 for Shore A hardness 24 hours versus tensile bond strength; r=0.607, P=.148 for Shore A hardness 28 days versus tensile bond strength).

Conclusions. Within the limitations of this study, significant differences were found in durometer Shore A hardness (with aging time) and tensile bond strength among the materials. Adhesive failure was moderately correlated with durometer Shore A hardness, especially after 28 days, but was not significant. (J Prosthet Dent 2014;112:1289-1297)

Clinical Implications
Clinicians need to be aware of the changes in hardness with the aging of denture lining materials in the mouth and need to select appropriate materials for specific clinical situations because the Shore A hardness and tensile bond strength values vary widely among commercial soft lining materials.

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A removable dental prosthesis is used for the restoration of the edentulous mouth when multiple teeth are missing. Decreasing stability and retention force of the denture base and the collapse of the vertical dimension of occlusion can be caused by the gradual resorption of the residual ridge under the prosthesis. In such a situation, soft lining materials are used to compensate for the resorbed zone of the ridge and to absorb some of the energy produced by masticatory impact. Plasticized acrylic resin-based, plasticized vinyl resin-based, polyurethane-based, polyphosphazene-based, and silicone-based materials are used as soft lining materials. Commercially, the most widely used long-term soft lining materials are either the plasticized acrylic resin-based or silicone-based materials; autopolymerizing and heat-polymerizing types are also available.

Long-term soft lining materials have several problems associated with their use, such as loss of softness, water sorption, colonization by Candida albicans, bonding failure between the denture base and the soft lining material, change of dimension or color during polymerization, and usage. Therefore, tear strength, tensile bond strength (TBS), elongation, and Shore A hardness (SH) have been determined to evaluate the properties of the long-term soft lining materials. The modulus of elasticity of acrylic resin denture base resin (2400 MPa) is significantly higher than those of the tissues on which it rests (1.25 to 5.0 MPa). Therefore, when stress concentrates in the denture-supporting mucosa or the stability of the denture base is poor, trauma or ulceration of the tissue can occur. In such a situation, soft lining materials provide a cushioning effect with a resiliency similar to that of the oral soft tissue, avoiding local concentration of stress, and provide even distribution of functional load with good elasticity. The SH test has been widely used to evaluate the elasticity of soft lining materials. This is a relatively simple test that determines the resistance to the indentation made by a rigid indenter on which a force is applied.

Many studies have used the SH test with variations for polymerization methods, aging, primer application, or pretreatment methods for soft lining materials. In dental practice, the separation of soft lining materials from the denture base is observed frequently, and this is one of the most significant problems of long-term soft lining materials. Various evaluation methods such as peel strength, shear strength, and tensile strength tests have been used to determine the bond strength of soft lining materials to denture bases. The TBS test has been recognized as an appropriate method, but the results are not in agreement because of the differences between the evaluation methods of tensile strength. Therefore, comparing the bond strengths of the soft lining materials is not easy. Emmer et al pointed out that the axial self-alignment of the specimen is an important factor for the precise evaluation of TBS.

The purpose of this study was to evaluate the durometer SH (with aging) and TBS and to investigate the correlation between these 2 properties in 7 commercially available long-term soft lining materials. The null hypotheses were that no difference would be found between the mean values of durometer SH (with aging), that no difference would be found between the TBS values of the materials, and that no correlation would be found between the mean values of durometer SH and TBS.

MATERIAL AND METHODS

The materials used in this study are listed in Table I; the long-term soft lining materials were 6 silicone-based materials (Dentusil, GC Reline Soft, GC Reline Ultrasoft, Mucopren Soft, Mucosoft, Sofreliner Tough) and 1 acrylic resin-based material (Durabase). Lucitone 199 was heat polymerized and used as a denture base material. The durometer SH tests were carried out in accordance with the International Organization for Standardization (ISO) standards ISO 10139-2:2009 and ISO 7619-1:2010. The TBS tests were carried out in accordance with ISO 10139-2:2009. A water bath with inner dimensions of 300 × 200 × 270 mm was used for storage. The sample sizes were determined by G-power software v3.1.9 (Heine Heinrich University) with a medium effect size of 0.5 and with the significance level set at .05 and the power at .80. The calculated total sample size for a repeated-measures ANOVA within factors was 14, and for within interaction it was 21. Therefore, the sample size of 21 was determined for the durometer SH test. For the TBS test, the calculated total sample size was 63; when considering the measurement error, a sample size of 70 was chosen.

Durometer Shore A hardness

Three test specimens per product were prepared for the durometer SH test in accordance with the manufacturers’ instructions. Six silicone-based soft lining materials were supplied by cartridge type and were mixed and dispensed with a specific mixing tip and dispenser. Acrylic resin-based Durabase liquid was added to the powder in a mixing cup with a volume proportion of liquid to powder at 1:2, and the mixture was stirred thoroughly for 30 seconds with a spatula. The mixture was placed into the split mold (Fig. 1), which was specially designed for the specimen preparation and locked tightly as a locking screw advanced along the tapered outer surface of the split mold. The specimens were 35 mm in diameter and 6 mm in thickness. The mold assembly consisted of a 2-piece split ring, screwed mold base, joining screw cap with tapered inner surface, and specimen support; undistorted specimens with accurate dimensions were obtained.

After the setting time specified in the manufacturers’ instructions, the specimen was removed from the mold and stored in a 37°C water bath for
24 hours before the durometer SH measurement. By turning the knob of the vertically driving ratchet holding the durometer SH tester (Durometer A, WHS 150/A; Wolpert Wilson), the indenter foot was gradually lowered onto the surface of the specimen in such a way that the indenter foot just touched the specimen surface. The value was recorded after 5 seconds of loading, and the measurements were repeated 5 times for each specimen. At this time, the distance of the loading points from the edge of the specimen was at least 5 mm. The durometer SH was calculated by averaging the 5 determined values, and this was carried out for each specimen. The total test time was less than 3 minutes for each specimen. After the determination of values, the specimen was stored in a 37°C water bath for another 27 days, and the water was changed every 7 days. At 27 days after the previous test, the specimen was removed from the water bath, and the 28-day durometer SH was measured by using the same procedure as that used for the 24-hour durometer SH test. The new loading points were located at a minimum

**Table I. Materials used**

<table>
<thead>
<tr>
<th>Brand Name</th>
<th>Material Type</th>
<th>Lot No.</th>
<th>Manufacturer</th>
<th>Processing Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lucitone 199</td>
<td>Denture base polymer, heat-polymerizable, powder and liquid</td>
<td>1108291</td>
<td>Dentsply Prosthetics</td>
<td>Compression-molding technique, heat processed at 72°C for 90 min, boiling water for 30 min, cooling at room temperature for 30 min, cool water for 15 min</td>
</tr>
<tr>
<td>Durabase</td>
<td>Plasticized methacrylate-based soft liner</td>
<td>Powder: 021210 Liquid: 031010</td>
<td>Reliance Dental Mfg Co</td>
<td>Auto polymerize by mixing powder and liquid</td>
</tr>
<tr>
<td>Durabase Repair Liquid</td>
<td>Adhesive</td>
<td>031010</td>
<td></td>
<td>Applied to the surface and gently air dried</td>
</tr>
<tr>
<td>Dentusil</td>
<td>Auto polymerizing polyvinyl siloxane-based soft liner</td>
<td>1112-583</td>
<td>Bosworth Co</td>
<td>Autopolymerizing</td>
</tr>
<tr>
<td>Dentusil Silicone Soft Reline Adhesive</td>
<td>Adhesive</td>
<td>1110-498</td>
<td></td>
<td>Dried for 1 min</td>
</tr>
<tr>
<td>GC Reline Soft</td>
<td>Auto polymerizing polyvinyl siloxane-based soft liner</td>
<td>1109061</td>
<td>GC Dental Products Corp</td>
<td>Autopolymerizing</td>
</tr>
<tr>
<td>GC Reline Primer R</td>
<td>Primer</td>
<td>1109291</td>
<td></td>
<td>Applied to the surface and gently air dried</td>
</tr>
<tr>
<td>GC Reline Ultrasoft</td>
<td>Auto polymerizing polyvinyl siloxane-based soft liner</td>
<td>1107051</td>
<td>GC Dental Products Corp</td>
<td>Autopolymerizing</td>
</tr>
<tr>
<td>GC Reline Primer R</td>
<td>Primer</td>
<td>111251</td>
<td></td>
<td>Applied to the surface and gently air dried</td>
</tr>
<tr>
<td>Mucopren Soft</td>
<td>Auto polymerizing polyvinyl siloxane-based soft liner</td>
<td>100232</td>
<td>Kettenbach GmbH &amp; Co KG</td>
<td>Autopolymerizing, 3 min 15 s followed by 15 min in a 50°C water bath</td>
</tr>
<tr>
<td>Mucopren Adhesive</td>
<td>Adhesive</td>
<td>80321</td>
<td></td>
<td>Applied twice. Dried for 30 s with air after first application. Dried for 90 s with air after second application</td>
</tr>
<tr>
<td>Mucosoft</td>
<td>Auto polymerizing polyvinyl siloxane-based soft liner</td>
<td>MU-11333</td>
<td>Parkell Inc</td>
<td>Autopolymerizing</td>
</tr>
<tr>
<td>Mucosoft Bonding Liner</td>
<td>Adhesive</td>
<td>ML-08316-1</td>
<td></td>
<td>Dried for 2 min with air</td>
</tr>
<tr>
<td>Sofreliner Tough</td>
<td>Auto polymerizing polyvinyl siloxane-based soft liner</td>
<td>081</td>
<td>Tokuyama Dental Corp</td>
<td>Autopolymerizing</td>
</tr>
<tr>
<td>Sofreliner Tough Primer</td>
<td>Primer</td>
<td>032B</td>
<td></td>
<td>Dried sufficiently</td>
</tr>
</tbody>
</table>
distance of 2 mm from the previous loading points.

**Tensile bond strength**

The denture base materials were prepared in plate form with a dimension of 25 ±3 mm square and 3 ±0.5 mm in thickness. The denture base resin was mixed according to the manufacturer's instructions and packed into the mold of a dental stone cast in accordance with the manufacturer's instructions. After polymerizing, the specimens were removed from the mold and wet polished with P500 SiC abrasive paper in a polishing machine (MetaServ 250; Buehler Ltd) while both surfaces of the specimen were kept plano-parallel. Polished specimens were stored in a 37°C water bath for 28 days before the test and used as adherent plates. After removing the acrylic resin plates from the water, they were air dried, and an adhesive was applied to both surfaces of the acrylic resin plates involved in bonding according to the
manufacturer's instructions. A specific adhesive was used for each material supplied by the same manufacturer. The adhesive was applied evenly with a dry, clean brush. A collar made of polytetrafluoroethylene, 10 mm in inner diameter and 3 mm in height, was positioned on the acrylic resin plate on which the adhesive was applied, and the slight excess of the mixed soft lining material was injected into the collar. Another acrylic resin plate with a surface on which the adhesive was applied was gently pressed over the overfilled lining material, and the plates were clamped and maintained for 1 hour at room temperature. The bonded specimen was stored in a 37°C water bath for 23 ±1 hours. The specimen was removed from the water bath, and a polymethyl methacrylate rod, which was screwed with a ring bolt into the upper side, was fixed to the specimen with cyanoacrylate luting resin. The lower acrylic resin plate of the adherent specimen (Fig. 2) was slid into the bracket slot. In this study, a sliding-insert bracket fixed on a ball-and-socket joint assembly was used to hold the lower acrylic resin plate. As the crosshead of the testing machine moved up, the lower acrylic resin plate was secured to the lower surface of the bracket, avoiding preload. By using a ball-and-socket joint in the lower fixture and by pulling the rod fixed to the upper acrylic resin plate through an s-hook, the adherent surface was aligned perpendicular to the loading axis, and the upper and lower axes of tensile force were in colinear alignment. The loading assembly used in this study was designed to minimize the shearing or torsional forces, which adversely influence the TBS.

For the TBS test, a universal testing machine (4302; Instron) was used at a crosshead speed of 10 mm/min. The test was carried out until separation occurred, and the TBS was calculated by dividing the maximum load by the adherent area assessed from the inner
diameter of the collar. Ten specimens for each soft lining material were evaluated.

Statistical analyses

A repeated-measures ANOVA was used to test the mean values of durometer SH for the main effects (aging and materials) and the interaction effects between aging and materials. The Shapiro-Wilk test, Kruskal-Wallis test, and Duncan multiple range test were used to assess the mean values of the TBS. The Spearman correlation was used to correlate between the values of durometer SH and TBS.

IBM SPSS for Windows (v20.0; IBM Corp) was used to assess the statistical analyses.

RESULTS

The SH values at 24 hours and 28 days for soft lining materials were significantly different with the variation in products ($P<.05$; Table II). In a repeated-measures ANOVA, the repeated test time was 2 (24 hours and 28 days), and the sphericity according to the Mauchly test was not obtained. Thus, the Huynh-Feldt correction ($\varepsilon=1.000$) was used for sphericity and for testing the durometer SH differences among the materials. Except for GC Reline UltraSoft (extra soft type; durometer SH, 24 hours = 21.30 ±0.29), all the tested materials showed durometer SH (24 hour) values within the limit of the soft type (25 < durometer SH, 24 hour ≤ 50) specified by ISO 10139-2:2009. After the specimens were aged for 28 days, all the tested materials showed a significant increase in the durometer SH value ($P<.001$). The main effects within materials were significant ($P<.001$), and the interaction effect (aging × materials) was also significant at $P<.001$ (Fig. 3).

Table III shows the TBS values. Although the normality assumption was accepted by the Shapiro-Wilk test ($P>.05$), the equal variance assumption was not satisfied with the Levene test ($P=.005$). After detection of a

![Durometer Shore A hardness changes of long-term soft lining materials.](image-url)
significant difference with the Kruskal-Wallis test (P < .001), the Duncan multiple range test was performed. The TBS of GC Reline Ultrasoft was 0.82 ± 0.32 MPa and that of Mucopren Soft was 0.96 ± 0.46 MPa, which was significantly lower than the TBS values of the other products (P < .05). Because GC Reline Ultrasoft was the only material to be classified as an ‘extra soft’ based on the result of the durometer SH test, its TBS of 0.82 ± 0.32 MPa satisfied the requirement of the ISO standard, which specifies that the value should be above 0.50 MPa in 8 of the 10 specimens of ‘extra soft’ materials. Among the ‘soft’ materials, Durabase, the only acrylic resin-based liner in this study, showed a significantly lower TBS compared with the silicone-based ‘soft’ materials, except for Mucopren Soft, which did not satisfy the lower limit (1.0 MPa) of TBS specified in the ISO standard for ‘soft’ liners. Among the tested materials, GC Reline Soft showed the highest durometer SH (28 day) value of 57.20 ± 0.28 and the highest TBS value of 2.99 ± 0.43 MPa (P < .05). Adhesive failure was observed for all materials except GC Reline Ultrasoft and Mucopren Soft. For those 2 materials, the mode of failure was adhesive, cohesive, and mixed. The TBS and SH showed an insignificant moderate positive correlation (r = 0.571, P = .180; durometer 24 hour SH = 8.01x + 18.503, r = 0.607, P = .148; durometer 28 day SH = 5.13x + 33.75, where x = TBS) with the Spearman correlation analysis.

DISCUSSION

According to the ISO standard for long-term soft lining materials, these materials are categorized into type A (soft type) for 25 < durometer SH ≤ 50 and type B (extra soft type) for durometer SH ≤ 25 when measured at 24 hours after specimen preparation. The standard specifies that the specimens that are aged for 28 days should exhibit durometer SH ≤ 55 for type A and durometer SH ≤ 35 for type B materials, because the lining materials should not harden excessively after an extended period of use. The first null hypothesis was tested by a repeated-measures ANOVA and was rejected because a significant difference was found for the materials (P < .001) and a significant interaction effect (aging × materials) was found (P < .001). For long-term soft lining material, a smaller increase in the durometer SH is considered desirable. However, the 28-day durometer SH value increased significantly compared with the 24-hour durometer SH value for both acrylic resin-based and silicone-based materials, and this result is in agreement with those of Mese and Guzel and Iwaki et al. The difference in the durometer SH value between the 24-hour test and the 28-day test was
the largest for Dentusil at 13.90 and the smallest for Mucosoft at 2.00 in the ‘soft’ material groups.

The difference in the durometer SH of GC Reline Ultrasoft, the only ‘extra soft’ liner in this study, was also high at 13.43. The increase in durometer SH with aging is thought to be caused by the gradual leaching of the plasticizers or other soluble contents from the soft lining materials. Therefore, an optimal fit between the soft lining materials and the denture base and long-term soft lining materials is needed. TBS values were compared using the TBS method for evaluating the bonding of the lining materials. Hence, careful application of the primer or adhesive and meticulous application of the liner over the denture base should be achieved to reduce bubble inclusion. Also, when performing the TBS test, the perpendicular alignment of the tensile load axis, which passes through the center of the specimen to the adherent surface, is important. Otherwise, a twisting or shearing force will be exerted on the bonded surface, causing inaccurate TBS values, especially when bubbles appear at the edge of the specimens.

McCabe et al19 carried out the TBS test with Lucitone 199 at a crosshead speed of 10 mm/min, and the same denture base resin and crosshead speed were used in this study. The reported TBS values were 3.11 ±0.39 MPa for GC Reline Soft and 0.79 ±0.16 MPa for GC Reline Ultrasoft, which were similar to the TBS values of 2.99 ±0.43 MPa for GC Reline Soft and 0.82 ±0.32 MPa for GC Reline Ultrasoft in this study. However, in the study by Bayati et al,29 GC Reline Soft showed a TBS value of 1.94 ±0.26 MPa at a crosshead speed of 5 mm/min, which was somewhat different from the present study. In other previous studies assessing the TBS, various crosshead speeds ranging from 2 mm/min to 60 mm/min were used.23-25,27,28 The difference in the crosshead speed can produce different results. Generally, a faster crosshead speed results in a higher TBS and lower elongation.

For the alignment of the adherent surface perpendicular to the loading axis, Mutluay and Ruytera27 used an alignment device. A screw head was inserted into the denture base polymer and a tapped hole in a brass bar was attached to the screw. The tapped brass bar was aligned by using a specially designed alignment device. In the present study, the authors established the perpendicular alignment of the adherent surface to the loading axis and the passive specimen positioning before loading by using a specially designed loading assembly and fixture as shown in Figure 2. By using this TBS testing assembly, accurate measurement as well as the ease in positioning, alignment, and removal of the test specimens could be accomplished. Thus, a more reliable value of TBS could be determined.

When clinicians apply the lining materials over a denture base resin, the inclusion of small bubbles in the bonded interface is generally inevitable. Therefore, a larger standard deviation of the TBS values is observed compared with that of the durometer SH values. Hence, careful application of the primer or adhesive and meticulous application of the lining materials during long-term use would affect the softness and bond strength of the lining materials. Further studies on the effects of those parameters are required.

**CONCLUSIONS**

Within the limitations of this in vitro study, the following conclusions can be drawn. Except for GC Reline Ultrasoft, all the materials showed 24-hour durometer SH values within the ISO range (25 < durometer Shore A, 24 hour ≤ 50) specified for the ‘soft’ long-term lining materials. After the specimens were aged for 28 days, all the tested materials showed a significant
increase in durometer SH (P < .001). The GC Reline Ultrasoft (which was the only material to be classified as ‘extra soft’ based on the durometer SH test) and Mucopren Soft showed significantly lower TBS values (P < .05). Among the tested materials, GC Reline Soft showed the highest durometer SH (28 day) value (57.20) and the highest TBS value (2.99 MPa) (P < .001). Adhesive failure is moderately correlated with durometer SH, especially after 28 days, but was not significant (r = 0.571 for durometer SH 24 hour versus TBS; r = 0.607 for durometer SH 28 day versus TBS).

REFERENCES


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