Effect of Surface Treatment on the Bonding of an Autopolymerizing Soft Denture Liner to a Denture Base Resin

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> **Purpose:** This in vitro study evaluated the effects of surface treatments and thermocycling on the bonding of autopolymerizing silicone soft denture liner (Sofreliner) to denture base resin. *Materials and Methods:* The bonding surfaces of denture base cylinders were polished with 600-grit silicon carbide paper and pretreated with applications of Sofreliner Primer, Sofreliner Primer after air abrasion, Reline Primer, or Reline Primer after air abrasion. Failure loads and elongation at failure were measured after subjecting specimens to 0, 10,000, 20,000, and 30,000 thermocycles. Failure modes were assessed for all specimens. Seven specimens were fabricated for each of 16 groups, including four pretreatments and four thermocycle groups. Results: Failure loads of the Sofreliner Primer group were significantly higher than those of the air-abrasion group up to 20,000 thermocycles; both groups showed cohesive failures of the soft denture liner. Failure loads of the Reline Primer group were significantly higher than with Reline Primer after air abrasion up to 10,000 thermocycles. Failure mode after 10,000 thermocycles was cohesive for the Reline Primer group and mixed cohesive/adhesive for Reline Primer after air abrasion. Failure loads of the Sofreliner Primer group were significantly higher than those of the Reline Primer group at each thermocycling interval. Elongation values decreased after 10,000 thermocycles for all groups. Conclusion: Air abrasion on the denture base resin surface was not effective in enhancing failure load. Cyclic thermal stress is one factor degrading the bond between soft denture liner and acrylic resin denture base. Int J Prosthodont 2004;17:297-301.

Soft denture liners have been widely used for cases with irritation of the denture-supporting mucosa. They act as a cushion and provide an even distribution of functional load onto the stress-bearing mucosa. Problems with the clinical use of soft denture liners include loss of softness, colonization by *Candida albicans*,

plaque and calculus accumulation, porosity, and poor tear strength.^{1,2} However, the main problem with silicone soft denture liners is the loss of adhesion at the interface with the denture base resin, which exacerbates these other problems. The bonding of soft denture liners has been evaluated by means of tensile,^{1–13} shear,^{3,4,8,9,14–16} and peel tests.^{8,9,11,17,18} Their longevity was evaluated by immersion in water,^{3,6,7,10,13,15,16,19} accelerated weather testing,^{12,20,21} and thermocycling.^{1,5,17}

Silicone-based soft denture liners have little or no chemical adhesion to denture base resin; therefore, an adhesive primer is supplied to aid bonding to the denture base resin. Thus, the bonding of silicone soft denture liners depends on the tensile strength of the liners and the adhesive primers used.^{1,2,15} The effect of roughening the bonding surface by air-particle abrasion has also been reported.^{6,18} Previous studies report that bond strengths to the roughened surface are higher compared to the smooth surface

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Material	Brand name	Lot No.
Polyvinyl siloxane soft denture liner	Sofreliner (medium soft), Tokuyama	690842
Fluid denture base resin	Pour Resin, Shofu	Powder: 048583, liquid: 069633
Adhesive primer	Sofreliner Primer, Tokuyama	815
Adhesive primer	Reline Primer (for resin), GC	0105081





Fig 1 Tensile test specimen.

because of the irregularity of the surface, which provides mechanical retention for the soft material.^{22,23} Other studies report that mechanical surface preparation of acrylic resin denture base surfaces has an adverse effect on bonding of the lining material^{4,16} and is not warranted.¹⁸ Therefore, it was hypothesized that the priming procedures may affect the bonding longevity of a lined denture.

Excessive water absorption in autopolymerizing silicone materials compared to heat-polymerizing silicones has also been reported.²⁴ However, physical properties of autopolymerizing silicones, including water absorption, low solubility, and surface roughness,^{14,19} have been improved. The autopolymerizing soft lining materials are usually used for direct lining, as they are easy to manipulate and require no laboratory procedures. However, longevity of the adhesive bonding of autopolymerizing soft denture liners to denture base resin has not been clarified. The purpose of the present in vitro study was to evaluate the effect of surface treatments on bonding of autopolymerizing soft denture liner to denture base resin after thermocycling.

Materials and Methods

Materials used in this study are presented in Table 1. A denture base resin made for use with the fluid resin

technique was mixed with a powder:liquid ratio of 18 g:10 mL. The mixture was poured into silicone rubber molds and left at room temperature for 2 minutes, then polymerized in 50°C water for 10 minutes under a pressure of 0.4 MPa with a polymerizing unit (SSKJ-50, Shofu). A total of 224 denture base specimens were fabricated into cylinders of 8-mm diameter and 4.2-mm height. The bonding surface of each specimen was polished with 600-grit silicon carbide paper under water irrigation, and finally prepared to 4-mm height. Denture base resin specimens were stored in 37°C distilled water for 21 days to allow saturation to occur. Saturation was determined by weighing the denture base cylinders every 24 hours after removing surface moisture with clean gauze and leaving specimens in air for 30 seconds. Measurement was completed with an electronic scale (FR-300, A&D) with 0.1-mg accuracy. The weight reached a plateau after 21 days of storage.

Denture base specimens were arbitrarily divided into four groups of 56 specimens each. The bonding surfaces of the denture base specimens were pretreated with one of four procedures:

- Application of Sofreliner Primer (SR)
- Air abrasion, followed by application of Sofreliner Primer (ASR)
- Application of Reline Primer (RL)
- Air abrasion, followed by application of Reline Primer (ARL)

Tensile test specimens were fabricated by polymerizing the soft denture liner in a 2-mm thickness between a pair of pretreated denture base cylinders (Fig 1). The soft denture liner was polymerized in a Teflon (DuPont) mold at 37°C inside an incubation chamber (MIR-162, Sanyo Electric) for 10 minutes. Twenty-eight tensile test specimens were fabricated for each pretreatment group at a time and equally divided into four groups. One group was stored in 37°C distilled water for 24 hours, then subjected to tensile testing. This group was used as a control. The other three groups were subjected to 10,000, 20,000, and 30,000 thermocycles, respectively. Specimens were immersed alternately in 4 and 60°C water baths with a 1-minute dwell time at each temperature. A total of 112 specimens, 7 specimens each in 16 groups, were fabricated. **Fig 2** Tensile testing; a = mounting jig; b = specimen holder; c = tensile specimen.



Table 2 Mean (Standard Deviation) Tensile Failure Loads (N)*

Specimen [†]	No thermocycling	10,000 thermocycles	20,000 thermocycles	30,000 thermocycles
SR	108 (7)	107 (7)	104 (6)	71 (12) ^d
ASR	89 (7) ^a	87 (6) ^b	85 (5)	61 (7) ^d
RL	98 (6) ^a	87 (8) ^b	50 (6) ^c	20 (6) ^e
ARL	74 (6)	65 (8)	41 (7) ^c	18 (5) ^e

*Values connected by horizontal lines were not significantly different within the same surface treatment (*P* > .050); values with the same letter were not significantly different within the same No. of thermocycles (*P* > .050). [†]See Materials and Methods for abbreviations.

A specimen holder consisting of an acrylic resin rod and a screwed metal hook was attached to both the top and bottom surfaces of each tensile test specimen with cyanoacrylate adhesive (Zerotime, Cemedine). The tensile specimen assembly was mounted onto a universal testing machine (Instron model 1114) with a mounting apparatus to ensure proper alignment (Fig 2). The tensile test was carried out at a 25.4 mm/min cross-head speed until failure. Data collection was managed through a scanner (model 5100 scanner, Vishay Measurements Group) and recorded into data system software (Strain Smart version 3.1, Vishay Measurements Group) 10 times per second throughout the tensile testing; thus, the load-displacement relationship for each specimen was obtained. The maximum tensile load was recorded, and elongation was calculated. The maximum stress during failure was described as the failure load (N), as specimens did not always completely separate cohesively.

Fractured surfaces were observed under an optical microscope (SMZ-10, Nikon) at a magnification of $10 \times$ to assess the failure mode. Failure modes were categorized as cohesive failure of the soft denture liner, adhesive failure at the soft denture liner–denture base resin interface, or a combination.

The mean values of each group were statistically analyzed by two-way analysis of variance (ANOVA), with pretreatment procedures and thermocycling numbers as independent factors. Differences among groups were analyzed by a Bonferroni-Dunn test at a 95% confidence level.

Results

Two-way ANOVA indicated significant differences between priming procedure (P < .001) and thermocycling (P < .001) for failure load. In addition, significant interaction between priming procedure and thermocycling (P< .001) indicated that some pretreatment procedures were more affected by thermocycling.

Failure loads of group SR were significantly higher than those of all the other groups, both before and after thermocycling (Table 2). For both groups SR and ASR, failure loads were maintained up to 20,000 thermocycles, after which they decreased (P < .001); failure load varied more for groups RL and ARL (Table 2).

All specimens showed cohesive failure of the soft denture liner before thermocycling, whereas a varying pattern of cohesive and adhesive failure occurred after an increasing number of thermocycles (Table 3).

Elongation values in groups SR and RL were higher than those in groups ASR and ARL (Table 4). In all groups, elongation decreased with increasing number of thermocycles.

	Table 3	Mode	of Failure	of Each	Group
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Specimen*	No thermocycling	10,000 thermocycles	20,000 thermocycles	30,000 thermocycles	
SR	С	С	С	C+A	
ASR	С	С	C+A	C+A	
RL	С	С	C+A	А	
ARL	С	C+A	А	А	

*See Materials and Methods for abbreviations.

C = cohesive failure of soft denture liner; A = adhesive failure at soft denture liner–denture base resin interface; C+A = cohesive and adhesive failure.

Table 4	Mean	(Standard	Deviation)	Elongation	of S	pecimens	Until Fa	ilure*
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Specimen [†]	No thermocycling	10,000 thermocycles	20,000 thermocycles	30,000 thermocycles
SR	4.5 (0.3) ^a	3.8 (0.3)	3.3 (0.2)	2.0 (0.5) ^d
ASR	3.6 (0.2)	2.8 (0.3) ^{b,c}	2.7 (0.2)	2.0 (0.2) ^d
RL	4.3 (0.2) ^a	3.0 (0.2) ^b	1.5 (0.1)	1.2 (0.1) ^e
ARL	3.2 (0.2)	2.5 (0.2) ^c	1.8 (0.2)	1.1 (0.1) ^e

*Values connected by horizontal lines were not significantly different within the same surface treatment (*P* > .050); values with the same letter were not significantly different within the same No. of thermocycles (*P* > .050). [†]See Materials and Methods for abbreviations.

Discussion

The bonding interface between soft denture liner and denture base resin is mainly subjected to shear and tear stresses in clinical use.⁹ During tensile testing, shear stress is generated at the periphery of the bonding interface, as the bonding areas stay the same while the soft denture liner stretches.²⁵ Although the tensile test used in the present study is an accepted method, the test conditions may not simulate the clinical situation, as the test specimens had double adhesive surfaces and clinical cases have a single adhesive surface.

Failure loads of baseline specimens can be calculated in the range of 1.5 to 2.1 MPa by dividing failure load by adhesive area. These values were higher than those of autopolymerizing silicone denture liner (0.7 to 0.9 MPa),^{1,7} and were close to the values of heat-polymerizing silicone denture liner (1.6 to 2.0 MPa).^{2-4,7,9} The variation in the values might have been caused by differences in specimen size, specimen configuration, thickness of soft lining material, cross-head speed, type of denture base resin, type of soft lining material, surface preparation, and processing techniques.^{9,11}

Some studies report that bond strengths to roughened acrylic resin surfaces are approximately double those to the smooth surface because of the increase of adhesive area and mechanical interlocking.^{22,23} However, the results of the present study agree with others reporting opposite results.^{4,16,18} The groups bonded to a smooth surface maintained a cohesive failure mode longer compared to the groups bonded to an air-abraded surface. Airabraded resin surfaces may have pits, cracks, crevices,

discontinuities with sharp corners, and projections. These surface irregularities may not allow complete flow of the soft denture liner and may result in the formation of small voids by air entrapment. Therefore, stress concentrations^{1,4,5} may be developed in the vicinity of the bonding interface and initiate failure during tensile testing.

Cyclic thermal stress causes shear stress at the bonding interface, as it provokes repetitive shrinkage and expansion, and results in a difference of thermal volumetric change between denture base and soft denture liner. However, some materials show an increase, and others a decrease, in bond strength after 3,000⁵ or 5,000^{1,17} thermocycles. Increased bond strength is probably a result of further polymerization in a 60°C water bath. Therefore, a greater number of thermocycles was chosen for the evaluation of failure load to more closely simulate the clinical situation. As a result, debonding occurred concentrically, and the bonding areas were reduced. This reduction considerably affected both failure loads and elongation values. Another effect of thermocycling may be alteration of the elasticity of the soft lining material. Group SR exhibited decreased elongation values, while failure loads and modes at the baseline were the same after 10,000 thermocycles.

During thermocycling, the soft denture liner absorbs a large amount of water.^{14,24} This water absorption may lead to a considerable amount of dimensional change and result in shear stress at the bonding interface. Furthermore, hydrolytic degradation of the bond occurs when water diffuses into the interface and contacts the adhesive primers. Similar degradation occurs by long-term immersion in distilled water.^{3,6,7,10,13,15,16} However, the tested condition may be milder compared to the clinical

condition, as only one side of the specimen disk faced the aqueous environment.

Since silicone soft denture liner does not adhere chemically to denture base resin, a proprietary bonding agent is supplied to achieve adhesion.^{1,5,18} Therefore, the bonding of these materials also depends on the adhesive primer used.^{1,2,5} To evaluate the possibility of an alternative adhesive for denture base acrylic, Reline Primer was evaluated in addition to the proprietary primer. As the chemical components of both primers were not published, it is impossible to determine the cause of the different results. However, it is speculated that adhesive primers may consist of an organic solvent and adhesive monomer, which react with both silicone and resin materials. Differences in organic solvent may affect the penetration of the adhesive monomer into the denture base resin, and a difference in adhesive monomers may affect the reactivity of the added soft denture liner. Although Sofreliner Primer worked better for the bonding of Sofreliner, it may not work well for the bonding of other soft lining materials.

Although heat-polymerizing denture base resin is an ideal material to simulate the clinical situation, a fluid resin was chosen because of ease of specimen fabrication. Moreover, as lined dentures are exposed to repetitive mechanical stress during mastication, the present laboratory study did not simulate the clinical condition sufficiently. Therefore, further investigations including a cyclic loading test should be employed to evaluate bonding under conditions more closely approximated to clinical reality.

Conclusions

The following conclusions were drawn within the limitations of this in vitro study:

- Roughening of the denture resin surface with air-particle abrasion was not effective for enhancing failure load and maintaining longevity of a soft denture liner bonded to the denture base resin tested.
- 2. Cyclic thermal stress is one factor that degrades the bond between soft denture liner and acrylic resin denture base.
- 3. Application of Sofreliner Primer to the denture base resin was most suitable in obtaining durable bonding of Sofreliner to the denture base resin.

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