

Effect of Accelerated Aging on Permanent Deformation and Tensile Bond Strength of Autopolymerizing Soft Denture Liners

Jessica Mie Ferreira Koyama Takahashi, DDS, MS, Rafael Leonardo Xediek Consani, DDS, MS, PhD, Guilherme Elias Pessanha Henriques, DDS, MS, PhD, Mauro Antônio de Arruda Nóbilo, DDS, MS, PhD, & Marcelo Ferraz Mesquita, DDS, MS, PhD

Department of Prosthodontics and Periodontology, Piracicaba Dental School, State University of Campinas, Piracicaba, São Paulo, Brazil

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Correspondence

Jessica Mie F. K. Takahashi, Avenida Limeira, 901, CEP 13414-903, Piracicaba-SP, Brazil. E-mail: jemfkt@yahoo.com.br

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Abstract

Purpose: The aim of this study was to evaluate the effect of different accelerated aging times on permanent deformation and tensile bond strength of two soft chairside liners, acrylic resin (T) and silicone (MS) based.

Materials and Methods: Different specimens were made for each test of each reliner. The specimens ($n = 10$) were submitted to accelerated aging for 2, 4, 8, 16, 32, and 64 cycles. Tensile bond strength testing was performed at a crosshead speed of 5 mm/min and permanent deformation with a compressive load of 750 gf. Data were submitted to Mann-Whitney test to compare the materials at different times, and Kruskal-Wallis and Dunn tests were used for comparing aging intervals within a given reliner.

Results: MS presented a lower percentage of permanent deformation ($p < 0.0001$) and higher tensile bond strength ($p < 0.0001$) than T in all time intervals and was not affected by the accelerated aging process, which reduced the permanent deformation and increased tensile bond strength of T ($p < 0.05$).

Conclusion: MS presented lower permanent deformation and higher tensile bond strength than T. Although T presented changes in those properties after accelerated aging, both materials might be suited for long-term use.

The main characteristic of soft denture liners is viscoelasticity, which is directly related to material deformation and is also responsible for cushioning masticatory forces. This behavior is due to the association between elastic ability and material viscosity when the material is submitted to compressive, stretching, and shearing forces.

The most frequent problem related to prosthesis relining is a flaw in the bond between the soft liner and the denture base.¹ Detachment of the soft liner from the denture base compromises the durability of the relining procedure and may occur due to an inefficient bond to the denture, or low cohesive strength of the material. In addition, irregularities formed at the soft liner and denture base interface increase the adhesion and growth of microorganisms.²

Several mechanical tests have been applied in an attempt to evaluate the quality of the bond between soft liners and acrylic denture bases, such as tensile, shear, and peel tests.²⁻⁵ Nevertheless, Kutay⁶ maintains that the use of different techniques to evaluate bonding may generate different results depending on the characteristics of the applied force and its distribution at the interface.

Peeling tests simulate the horizontal components of the masticatory forces responsible for soft liner detachment at the edge of the prosthesis.⁷ However, to perform the test, specimens must be fixed directly onto the soft liner, which concentrates the peeling strength on the material rather than on the bond interface, so that the results depend on the cohesive resistance of the soft liner, not the bond strength to the acrylic resin.^{4,8} In shear tests, the forces applied on the soft liner are similar to those seen clinically; however, these forces are concentrated on the edges of the reliner material and are therefore not distributed throughout the entire relined area. Nevertheless, stress concentrated mainly on the edges compromises the results obtained with shear tests, influencing the amount of elastic deformation of the reliner.⁹ The tensile test, on the other hand, is better suited to evaluating the bond strength of soft liners and acrylic resin denture bases, because it verifies the strength required to rupture the bond or the reliner, allowing for the comparison of different materials and polymerization methods.¹⁰

Relined prostheses are also constantly affected by the presence of saliva, which is absorbed by the material, and acts to solubilize some of the plasticizer content of the soft liners.

In this environment, the prosthesis is also subjected to several temperature changes as a consequence of the patient's eating habits.^{11,12}

When in use, most materials present compromised mechanical properties.^{3,11-15} Therefore, in an attempt to simulate this aging process, several artificial methods are used, such as thermocycling and storage in distilled water. Exposure of materials to UV light, heat, and water spray has been widely used in dentistry research^{3,14,16} to evaluate the effects of dental material aging. Nevertheless, considering the greater complexity of the oral environment, accelerated aging has proven to be an effective method to compare the behavior of soft denture liners.¹⁶

Chairside soft relining materials are used extensively in prosthetic dentistry, making it necessary to conduct research evaluating their mechanical and physical properties. Thus, the aim of this study is to evaluate the permanent deformation and tensile bond strength of two chairside soft liners with different chemical compositions, and the effect of accelerated aging times on these relining materials.

Materials and methods

Two autopolymerizing soft liners and one heat-polymerizing acrylic resin were selected for this study. The materials selected were an acrylic resin-based soft liner (T) (Trusoft, Bosworth, Skokie, IL, batch 0510-551), a silicone-based soft liner (MS) (Mucopren Soft, Kettenbach, Germany, batch 60331), and an acrylic resin (QC-20, Dentsply, York, PA, batch 666552-powder, 580313-liquid).

Cylindrical specimens of each reliner were made for the permanent deformation test in accordance with ADA/ANSI specification 18.¹⁷ Specimens were made using a manufactured metal cast with five molds measuring 12.7 mm in diameter and 19 mm high. These were filled with the reliner materials and submitted to the polymerization process recommended by the manufacturers. After the polymerization process, the metal cast was completely dismounted, and the specimens were removed without any strain applied to them. Excessive material was removed with a sharpened blade.¹⁸ Poly(vinyl siloxane) specimens received an additional treatment with a surface sealer and were later immersed in 50°C water for 15 minutes, as recommended by the manufacturer.

Acrylic resin blocks for the tensile bond strength test were prepared using silicone rubber molds (Zetalabor, Zhermack, Madia Polesine, Rovigo, Italy) obtained by investing brass dies with 3-mm thick spacers in a denture flask.¹⁸ Specimens were made by placing the acrylic resin inside the silicone molds and processing the resin for 20 minutes while immersed in water at 100°C. After the polymerization process, the acrylic resin blocks were ground with 120-, 200-, and 400-grit silicon carbide paper until they were 40-mm long and had a cross section of 9 × 9 mm² gauged with a digital caliper (Starrett, Itu, Brazil) (0.01-mm precision). All acrylic resin blocks received an adhesive layer prior to reliner processing and finishing. The adhesives were provided by the manufacturers and were different for each material. Two adhesive layers were applied over the 9 × 9 mm² surface of the acrylic resin blocks prior to the relining, as recommended by the manufacturers of both materials. The

Table 1 Mean (SD) permanent deformation (%)

Aging cycles	MS	T
Control	0 (0) ^{Ba}	20.23 (3.82) ^{Aa}
2	0 (0) ^{Ba}	4.85 (1.41) ^{Ab}
4	0.04 (0.12) ^{Ba}	4.55 (1.59) ^{Ab}
8	0.01 (0.03) ^{Ba}	4.85 (2.35) ^{Ab}
16	0 (0) ^{Ba}	4.89 (1.56) ^{Ab}
32	0.03 (0.07) ^{Ba}	4.37 (1.16) ^{Ab}
64	0.02 (0.03) ^{Ba}	3.96 (2.19) ^{Ab}

Means followed by different uppercase letters in a row differ statistically according to Mann-Whitney test ($p < 0.0001$). Means followed by different lowercase letters in a column differ statistically according to Kruskal-Wallis and Dunn tests ($p < 0.0002$).

acrylic resin blocks were randomly relined, two by two, using a manufactured metal mold to provide appropriate alignment of the blocks, and a standardized 3-mm reliner thickness.^{6,18,19}

After all specimens for both tests were finished, they were randomly distributed into seven groups: G1 (control), G2-G7 [submitted to the accelerated aging (AA) process]. G2-G7 were inserted into an EQ-UV chamber (Equilam, Diadema, Brazil). Specimens were exposed to UV light irradiation from four 40 W UV-B fluorescent tubes with an irradiation peak of 1.8 W/m²/mm, at a temperature of 43.3°C and distilled water spray for 2, 4, 8, 16, 32, and 64 cycles. Each cycle represented 2 hours of exposure to UV light followed by 18 minutes of distilled water spray.^{3,14,16}

The permanent deformation test was performed using a mechanical device described in ADA/ANSI specification 18;¹⁷ however, testing was performed with a compressive load of 750 gf^{5,18} instead of the 80% standard compression mentioned in the ADA/ANSI specification. The load was applied to the specimen for 30 seconds, generating a compression value (A); the load was then removed, and after another 30 seconds, a value for elastic recovery was taken (B). The percentage of permanent deformation was determined by the formula $(A-B/19 \times 100)$.

The tensile bond strength test was performed using an EMIC DL-500MF testing machine (EMIC, Paraná, Brazil). Specimens were positioned in the testing machine and gripped within the acrylic resin block portion. Testing was performed at a crosshead speed of 5 mm/min.^{5,15,18} Bond strength values were calculated as stress at failure divided by the cross-sectional area. After the test, the type of failure was observed and classified as adhesive, cohesive, or both.

Means and standard deviations were calculated for each group and were submitted to non-parametric tests. The Mann-Whitney test was applied to compare the soft liners at each aging interval, and Kruskal-Wallis and Dunn tests were used to compare all the aging intervals of each reliner ($p = 0.05$).

Results

Table 1 presents the means and standard deviations (SD) of the permanent deformation test and statistical analysis results. Statistical analysis revealed a significant difference between the values obtained for each material ($p < 0.05$) and, irrespective of the aging interval, MS specimens presented lower permanent

Table 2 Mean (SD) tensile bond strength (MPa)

Aging cycles	MS	T
Control	3.15 (1.04) ^{Aa}	0.17 (0.03) ^{Bd}
2	2.79 (0.54) ^{Aa}	0.29 (0.13) ^{Bcd}
4	2.78 (0.55) ^{Aa}	0.37 (0.18) ^{Bbcd}
8	3.34 (1.44) ^{Aa}	0.64 (0.32) ^{Babc}
16	3.41 (0.76) ^{Aa}	0.35 (0.06) ^{Bbcd}
32	3.11 (0.97) ^{Aa}	0.66 (0.15) ^{Bab}
64	3.61 (0.75) ^{Aa}	0.89 (0.27) ^{Ba}

Means followed by different uppercase letters in a row differ statistically according to Mann-Whitney test ($p < 0.0001$). Means followed by different lowercase letters in a column differ statistically according to Kruskal-Wallis and Dunn tests ($p < 0.0002$).

deformation than did the T specimens ($p < 0.0001$). When the influence of different numbers of aging cycles for each reliner was considered, no statistical difference was observed in the MS groups ($p = 0.90$); however, the T groups presented a significant reduction of permanent deformation when the control group was compared with the other groups ($p < 0.05$), but there was no statistical difference between the accelerated aging groups ($p > 0.05$).

Tensile bond strength means (SD) are presented in Table 2, along with a statistical analysis revealing a significant difference between the relining materials ($p < 0.0001$). In this test, MS presented higher tensile bond strength values than did T, irrespective of the aging interval. MS groups also presented no statistical difference when compared with each other ($p = 0.15$), while T groups presented changes following accelerated aging treatment with an increase in tensile bond strength as the number of cycles increased ($p < 0.05$); however, there was no difference between the groups of four and 16 aging cycles. The highest and lowest tensile bond strength means were presented by the groups submitted to 64 cycles and the control group, respectively.

After the tensile bond strength test, specimens were evaluated regarding the type of failure that occurred during testing (Table 3). The MS groups initially presented only adhesive failures, but an increased percentage of mixed failures occurred with aging, but no cohesive failures. T groups presented both cohesive and mixed failures.

Discussion

The longevity of soft liners is directly related to their viscoelastic behavior. Changes in composition and the aging of the materials can reduce resilience and compromise the stress absorption ability of the material. At the same time, soft liner detachment from the prosthesis surface is the main cause of failure in the prosthesis relining procedure,^{1,15} which may occur due to inadequate cohesive strength or a deficiency of the bond to the acrylic resin denture base.⁴

Pinto *et al*^{5,18} stated that 1000 cycles of thermocycling aging would correspond to 1 year of prosthesis clinical wear and could therefore be used as a simulation; however, no correlation has been made between the clinical realm and UV accelerated aging. Due to a lack of this correlation, UV-accelerated ag-

ing intervals were determined by an association of the time consumed by the thermocycling procedure. It was observed that it took 86 hours to perform 2000 thermocycles. Based on this information, the UV chamber performed an equivalent of 64 cycles in this interval. The values in Table 1 show that the silicone-based material (MS) presented a lower percentage of permanent deformation than T in all evaluated intervals, and that MS was not affected by the accelerated-aging process, maintaining permanent deformation values between 0 and 0.04%, while T presented reduced permanent deformation after only two aging cycles. When submitted to compression, soft liners are expected to absorb the load and suffer deformation; when the compression ceases, they should return to their original form, resulting in complete elastic recovery. In this study, the silicone-based material presented a more satisfactory elastic behavior in the permanent deformation test than did the acrylic resin, confirming the previous statement that silicone reliners have better viscoelastic properties than acrylic resin reliners.⁴

The reduction in the percentage of permanent deformation presented by T, with an initial mean value of 20.23% and a mean of 4.59% in all other intervals, could be related to the particularities of the reliner composition. Acrylic resin reliners are frequently affected by water sorption and a loss of chemical components, which can alter their stiffness. The plasticizer content is responsible for the resiliency of acrylic-based reliners, acting on the reduction of their glass transition temperature to a value similar to that of the oral cavity.^{18,20} As the reliner ages, these compounds become soluble in an aqueous environment, which can compromise the viscoelasticity of the materials⁸ and increase stiffness. During clinical use, the reliners are also exposed to water sorption; water acts as a plasticizing agent on polymers by separating the polymer chains, thereby reducing the reliners' stiffness. As a result of the simulated aging, T presented increased stiffness, and consequently, a decreased percentage of permanent deformation.²¹ This effect may have been caused by an excessive loss of plasticizer not compensated by the water sorption process, hence the increased stiffness of the T reliner.

A material's percentage of deformation is important for evaluating the amount of elastic recovery it presents after compression. With regards to this characteristic, MS had a better performance. It can therefore be implied that the reliner suffered no permanent deformation even after aging; however, this behavior might clinically compromise the ability of the reliner to absorb masticatory loads and transfer them to the supporting tissues.²² Nevertheless, when the soft liner presents a low percentage of permanent deformation, it benefits prosthesis adaptation.³ Considering this argument, the reduction in T permanent deformation after aging may not be entirely harmful to the material, because it initially presented excessive permanent deformation and, after aging, became less elastic and more appropriate for use.

The tensile bond strength of a relined acrylic resin is best used to evaluate the bond strength between the two materials; strengths are distributed between the relining material and the relined surface, enabling the adhesive and cohesive strengths to be evaluated.^{9,23} Several crosshead speed values are reported in the literature for tensile tests,^{6,8,12,19,24} thus, a 5 mm/min speed was selected so the stress would build up at the bond

Table 3 Type of failure (%) (n = 10)

Material	Failure type	Aging cycles						
		Control	2	4	8	16	32	64
MS	Adhesive	100%	90%	70%	60%	60%	90%	80%
	Mixed	—	10%	30%	40%	40%	10%	20%
	Cohesive	—	—	—	—	—	—	—
T	Adhesive	—	—	—	—	—	—	—
	Mixed	—	40%	20%	50%	70%	10%	70%
	Cohesive	100%	40%	70%	50%	70%	10%	70%
	No failure	—	20%	10%	—	—	—	—

interface and the relining material, exposing the reliner to elastic and plastic deformation until failure occurred.⁶ When the test is performed at a high crosshead speed, the stress builds up too quickly, and is concentrated on the reliner rather than being transferred to the interface, which would make the results dependent on the cohesive strength of the reliner, not on the bond strength to the acrylic resin.

When comparing tensile bond strength values for T and MS (Table 2), MS presented higher values than T in all aging intervals ($p < 0.0001$). Again, MS specimens were not affected by the accelerated aging process; its values were between 2.78 and 3.61 MPa.

Silicone-based reliners have little or no chemical interaction with acrylic resin, so the tensile bond strength values are related to the cohesive strength of the reliner or the efficacy of the adhesive used.² In this study, it is probable that an interaction between these two elements occurred because MS presented adequate resistance (which was observed in the previous test) and the adhesive was ethyl-acetate-based, which is known to be an effective acrylic resin solvent.⁴

Accelerated aging's lack of effect on MS may be directly related to its composition. Silicone reliners are generally composed of poly (dimethyl siloxane) molecules, and their resiliency is related to the amount of cross-linking.⁷ In contrast, acrylic resin soft liners owe their resiliency to the addition of plasticizers.

Increased bond strength was observed in T specimens as the number of aging cycles increased, with a mean of 0.17 MPa in the control group and 0.89 MPa after 64 aging cycles. As the aging period progressed, the specimens presented lower elongation during the tensile test, indicating increased stiffness and, consequently, higher tensile bond strength. This difference in reliner properties may be related to changes in its molecular structure due to the aging process. Accelerated aging may also act on different mechanisms, such as the continuation of the reliner polymerization process,³ UV light scission of the polymer chains, oxygen cross-linking, and leaching of plasticizers.¹⁶

During aging, the reliner absorbs water, becoming stiffer and compromising its load absorption ability,⁷ resulting in the transfer of tensile and shear strengths to the bond interface with the acrylic resin.^{19,25} When submitted to the tensile test, specimens are also exposed to shear strength, because once the reliner begins to stretch, it becomes thinner, while the bond area remains unaltered;⁹ shear forces develop and concentrate at the edge of the relined area,⁶ increasing the reliner's tendency to peel.

Reliner detachment can also occur in the presence of porosities and irregularities on the surface of the acrylic resin, both of which weaken the bond and increase stress concentration.¹⁰

With regard to failure type (Table 3), MS primarily presented adhesive failures; however, as aging increased, more mixed failures were observed, indicating few changes in the reliner. Thus, the cohesive strength of the material is higher or very similar to that of the tensile bond strength. Nevertheless, the trend towards cohesive failure indicates that tensile bond strength is higher than the reliner's cohesive strength.^{4,8,15}

T groups presented both cohesive and mixed failures. A cohesive failure occurs in the reliner, and the mixed failure occurs when both the adhesion and the reliner fail. The presence of this type of failure may suggest that a lower tensile strength of the specimens could be related to the high elasticity of the reliner, rather than deficiencies in the bond to acrylic resin; hence, the molecular interaction between two materials with similar chemical composition.⁷ The bond to the acrylic resin blocks was also improved with the use of a surface adhesive that increased the acrylic resin's wettability.¹⁰

The use of a surface sealer aids in maintaining reliner properties; therefore, its needs are more evident in acrylic resin reliners because of their higher susceptibility to water sorption and solubility¹³; however, Anil *et al*¹¹ demonstrated that the use of a surface sealer decreases microleakage in MS specimens, but this effect was not maintained after aging. To be considered satisfactory for clinical use, a soft liner must present a tensile bond strength of at least 0.45 MPa.^{2,25} The results obtained in this study indicate that MS presented satisfactory tensile bond strength, and that T only became satisfactory after the eighth aging cycle.

Conclusion

Based on the limitations in this study:

1. The silicone reliner presented lower permanent deformation and higher tensile bond strength than did the acrylic resin reliner.
2. The accelerated aging process did not affect the evaluated properties of the silicone reliner; however, it reduced permanent deformation and increased the tensile bond strength of the acrylic resin reliner.
3. The acrylic resin reliner is softer than the silicone reliner, but is less resilient and can be affected by aging.

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