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## Tensile Bond Strength of a Soft Liner to an Acrylic Resin after Primer Application and Thermocycling

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**Purpose:** The aim of this study was to evaluate the effect of a surface treatment with Sofreliner primer on tensile bond strength of a resin-based soft liner (Coe-Soft) bonded to a polymethylmethacrylate (PMMA) resin before and after accelerated aging simulated by a thermocycling machine. **Material and Methods:** One hundred and twenty rectangular specimens of PMMA resin were fabricated with  $10 \times 10 \text{ mm}^2$  of cross-section and 40 mm of height. The resin-based soft liner was positioned between two PMMA resin specimens. The specimens were divided randomly into 6 groups ( $n = 10$ ) according to the surface treatment and accelerated aging cycles (0, 500 and 1000). After accelerated aging, specimens were submitted to tensile bond strength test. The fractures were classified as adhesive, cohesive or both. **Results:** Most failures were classified as cohesive ( $p = 0.025$ ) and there was an increase on the mean values of tensile bond strength for group with primer application regardless the cycles of accelerated aging, and also an increase along thermocycling periods. The scanning electronic microscopy was used to illustrate the types of failures and the effect of primer application on PMMA acrylic resin surface. **Conclusion:** The primer application associated with resin-based soft liners is clinically viable, once this treatment strategy provides a reliable adhesion.

**Keywords:** primers and coupling agents, surface treatment, denture liners, acrylic resins, adhesion

### 1. Introduction

The resilient denture liners are indicated for the restoration of inflamed support tissues, severe bone resorptions and low resilience mucosa because of their damping effect and by providing increased prosthesis stability<sup>1-4</sup>.

These materials are classified as resin-based soft liner and silicone-based soft liner<sup>3</sup> and are composed by monomers and polymers associated with plasticizers<sup>5,6</sup> which provide softness to the relining material and comfort to the patient<sup>7</sup>. Despite this advantage, the failure on adhesion of resilient liners to denture bases is one of the most common problems in clinical practice<sup>1,8,9</sup>.

This failure is related to several factors: the non-use of primer, the mechanical properties of acrylic resin<sup>9,10</sup> and relining materials, and the aging of relining materials, which are in a constant contact with saliva, water, and changes in temperature due to feeding<sup>8</sup>. The aging may change the relining materials properties, providing loss of plasticizers and resilience reduction, thus affecting the adhesion<sup>11</sup>.

Some union agents, i.e. primers, are developed to prevent these failures because of their interaction with the surface of

acrylic resin and the resilient liners<sup>1,2</sup>. It has been speculated that, although fabricated for silicone-based soft liner, the organic solvents present in primers composition may improve the adhesion between these materials<sup>12-14</sup>.

This study aimed to evaluate the effect of Sofreliner primer on tensile bond strength of a resin-based liner before and after accelerated aging simulated by a thermocycling machine. The first hypothesis of the study was that the Sofreliner primer should have a positive effect, significantly increasing the bond strength, and the second hypothesis was that the application of Sofreliner primer promotes more cohesive failures.

### 2. Material and Methods

The Table 1 presents the materials used in this in vitro study. For tensile test, 120 rectangular specimens of PMMA resin were fabricated from solid metallic matrices with dimensions of  $10 \times 10 \times 40 \text{ mm}$ . The resin excesses were removed with a maxicut bur and the specimens were sequentially polished with 200- and 400-grit sandpapers.

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The solid metallic matrices were placed inside a metallic flask, and a metallic bar with 3 mm in thickness was positioned between them. An impression of this conjunct was performed with laboratorial silicone (Zetalabor, Zhermack, Badia Polesine, Rovigo, Italy). After silicone polymerization, metallic matrices were substituted by PMMA specimens and the metallic bar was substituted by resin-based soft liner. Sofreliner primer was applied on the half of PMMA specimens at the surface that was in contact with the resin-based soft liner. These specimens were storage in distilled water at 37 °C for 24 h before the tensile test<sup>15</sup>. All the materials were prepared according to manufacturer’s instructions and by the same operator.

The specimens were randomly divided into 6 groups (n = 10) according to the surface treatment and to accelerated aging cycles (0, 500 and 1000). The number of cycles simulated a short or prolonged clinical use. The thermocycling was performed for 30 s with alternated baths in distilled water at 5 °C or 55 °C<sup>16</sup>.

The tensile bond strength test was performed in a universal testing machine (EMIC DL-3000, EMIC, São José dos Pinhais, PR, Brazil) with a constant velocity of 5 mm/min until failure was detected<sup>5</sup>. The tensile bond strength (MPa) was calculated dividing the maximum tensile strength value by the specimen cross-sectional area. The failure analysis was performed by a single operator through a stereomicroscope (Carl Zeiss 40096) under 32 × magnification, after the rupture of the specimen and failure was classified as cohesive, adhesive or both. The failures classification was performed according to the quantity of resin-based soft liner displaced. The total tearing of relining material was classified as cohesive, the total displacement of the material was classified as adhesive

while the partial displacement between specimens and partial tiring of the relining material was classified as both.

One specimen with primer application and one with no surface treatment were submitted to the scanning electronic microscopy (SEM) (JSM 610LA; JEOL) under 140× and 5,000× magnifications in order to illustrate the type of failures and the surface.

The averages and standard deviations were calculated for each group, results were submitted to the two-way analysis of variance (ANOVA) and significant differences were compared by Tukey test ( $\alpha = 0.05$ ). Failures were analyzed by Fisher test ( $\alpha = 0.05$ ).

### 3. Results

The tensile bond strength means are presented in Table 2. The analysis of variance shows no statistically significant difference between the factors primer and thermocycling. However, tensile bond strength values increased for the group with Sofreliner primer, regardless accelerated aging cycles. Increased tensile bond strength results were also observed along the thermocycling periods for both groups (with or without primer), regardless surface treatment.

Table 3 shows that cohesive failure was significantly more prevalent in groups with Sofreliner primer application in comparison with the group without primer. Thus, it can be assumed that the distribution of types of failure depended on the primer application. The SEM analysis illustrates the types of failures (Figure 1) and the effect of primer on PMMA acrylic resin surface (Figure 2). This surface treatment provided a more homogenous surface with a layer that is probably the agent necessary for bonding the PMMA acrylic resin to the resin-based soft liner.

Table 1. Materials used in this study.

Material		Composition	Manufacturer
QC-20		Heat-cured acrylic resin	Dentsply International INC, New York, EUA
Sofreliner Primer		Methylene chloride, polymethylmethacrylate with polyorganosiloxane	Tokuyama Dental Corp., Tokyo, Japan
Coe-soft		Powder: polyethyl methacrylate, zinc undecylenate, and pigments. Liquid: benzyl salicylate, dibutyl phthalate ethyl alcohol, methylsalicylate, oil mint.	Coe Laboratories Inc, Chicago, EUA.

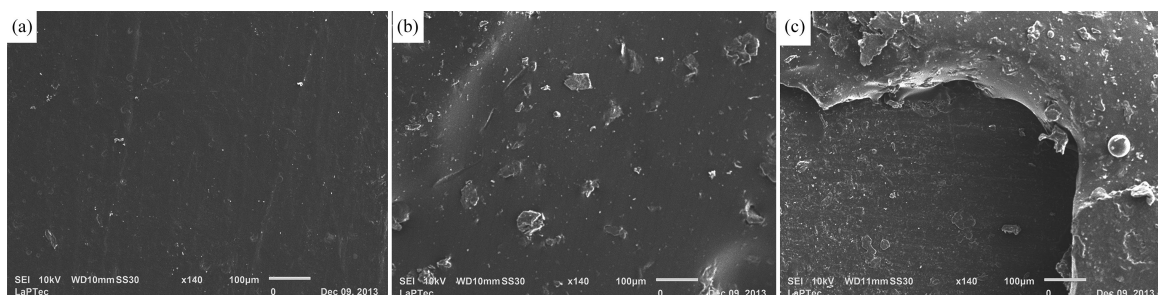
Table 2. Tensile bond strength results (MPa) according to the surface treatment (Sofreliner) and cycles of accelerated aging.

	Baseline	500 cycles	1000 cycles
Non- primer application	0.43 (0.12) Aa	0.46 (0.09) Aa	0.44 (0.04) Aa
Primer application	0.45 (0.11) Aa	0.46 (0.16) Aa	0.51 (0.14) Aa

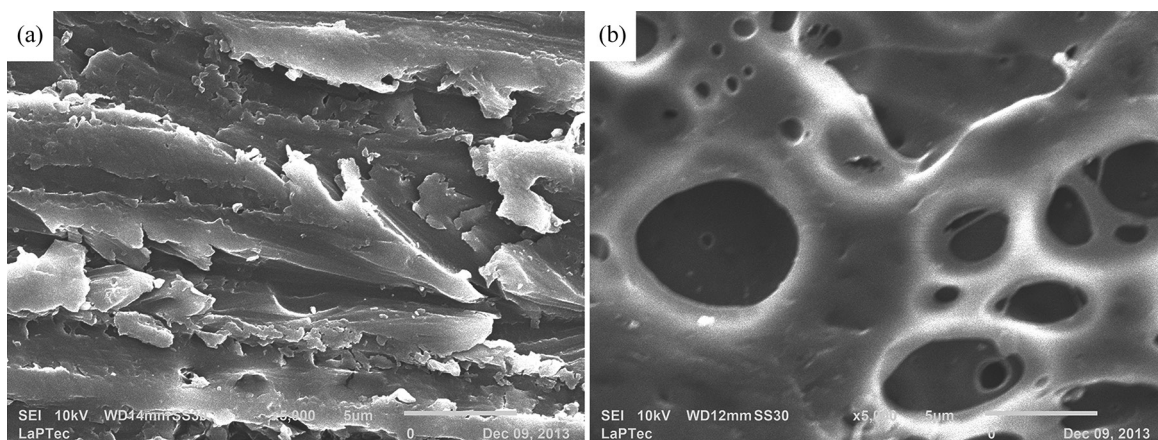
Means followed by different uppercase letter (line) and lowercase letters (column) differ statistically according to Tukey test ( $p \leq 0.05$ )

Table 3. Type of adhesive failures according to the application or not of Sofreliner primer.

	Type of failures			Fisher’s exact test: p-value (Two-tailed) alpha	0.025
	Adhesive	Cohesive	Both		
Non-primer application	1	19	10		0.05
Primer application	0	27	3		0.025



**Figure 1.** Types of failures after rupture on tensile bond strength test. (a) Adhesive failure with the complete rupture of the PMMA acrylic resin to the resin-based soft liner; (b) Cohesive failure with the displacement of the resin-based soft liner; (c) Image of both types of failures listed above.



**Figure 2.** (a) PMMA acrylic resin with non-application of Sofreliner primer and (b) after Sofreliner application.

#### 4. Discussion

The first hypothesis was rejected because the results did not show statistically significant difference regarding the tensile bond strength between the resin-based soft liner and the PMMA resin with primer application. The second hypothesis was accepted because the results showed a significant difference regarding the types of failures with predominant cohesive failure after primer application.

The primer used in the present study is fabricated for silicone-based soft liner and solvents (99.5%) and agents of union (0.5%) in its composition. This primer was tested because there is no primer available on dentistry market for resin-based soft liner. The main function of these agents of union is to unite the PMMA resin with the silicone<sup>12</sup>. According to SEM images, the authors assume that a layer of these agents of union was created after the primer application. Regarding the solvent, the authors believe it was responsible for the slight increase on tensile bond strength mean results and for the majority of cohesive failures after application of the evaluated primer, because of the conditioning effect on the PMMA acrylic resin surface.

The increasing of cohesive failures after primer application suggests an improvement on the tensile bond strength of the resin-based soft liner to the PMMA acrylic resin because the adhesion between these materials might be stronger than the intermolecular forces of the relining material.

This fact probably occurred because of the conditioning effect promoted by the solvents, increasing the physical contact of these materials.

Regarding the predominance of cohesive failures, it can be observed a slight increasing in tensile bond strength results after primer application, regardless thermocycling. However, this increasing did not lead to statistical differences between groups. The authors assume that this fact probably occurred because of the conditioning effect on PMMA acrylic resin surface promoted by the solvents present in the primer composition, as Can et al.<sup>8</sup> affirmed, facilitating the bonding of the resin-based soft liner to the PMMA acrylic resin. According to Mutluay et al.<sup>13</sup>, the solvents increase the surface wettability, promote the cleaning of the surface, and dissolve unattached particles of PMMA acrylic resin surface. The authors believe that significant differences could be observed on mean tensile bond strength results if the primers presented more effective solvents on their composition.

In literature there are several surface treatments for improving the bond strength of resin based soft liners with PMMA. Philip et al.<sup>17</sup> found that treatment with alumina particles for 5 minutes abrasion followed by monomer immersion exhibited higher bond strength. However, Kulkarni et al.<sup>18</sup> concluded that only the use of the monomer is effective method to increased bond strength between PMMA resin and soft liner without the use of blasting. Monomer may be



effective in increase the dissolution of the PMMA surface, providing greater bond strength<sup>18</sup>. Gundogdu et al.<sup>19</sup> reported that the pretreatment with 36% phosphoric acid may result in an increased surface area, consequently increasing the tensile bond strength between the resilient lining and denture base material.

Regardless primer application, no statistically significant difference was observed in tensile bond strength when the specimens were submitted to thermocycling. According to Minami et al.<sup>12</sup>, the increase in tensile bond strength is probably due to the extra polymerization provided by the warm bath in water, at a temperature of around 60°C.

Although accelerated aging increased the tensile bond strength in this study, this test generally negatively affects some mechanical properties of relining materials<sup>16</sup>. According to Goiato et al.<sup>15</sup>, thermocycling may lead to materials degradation by producing successive dilatations and contractions. Mancuso et al.<sup>10,20</sup> affirm that resin-based soft liners have high absorption, solubility and color change after accelerated aging. According to Hermann et al.<sup>6</sup> and Pinto JR et al.<sup>21</sup>, the clinical use simulated by thermocycling promotes loss of the plasticizers present in relining materials composition, increasing their hardness. These findings

corroborate with Goiato et al.<sup>22,23</sup> who found greater hardness and color change in other studies.

This in vitro study has some limitations, i.e. the use of one relining material and a primer fabricated for silicone-based soft liner. This primer was selected because there is not a universal primer available on dentistry market that can be used for the surface treatment of PMMA resin and allow the use of any relining material. Additional studies should be performed using primers with more effective solvents and which can be applied on resin-based soft liners, in order to improve the tensile bond strength of these materials to the PMMA acrylic resin. Besides, the thermocycling simulates just the temperature changes, not considering some factors, i.e. the influence of mastication.

## 5. Conclusion

The primer application on the PMMA acrylic resin surface improved the tensile bond strength results, promoted a majority of cohesive failures and increased the tensile bond strength values of the resin-based soft liner and the PMMA acrylic resin. The clinical use of the evaluated primer is viable for resin-based soft liners.

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