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**Adhesion potential of relining materials to polyamide and PMMA-based denture  
base materials: effect of surface conditioning methods**

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DOI: <https://doi.org/10.1080/01694243.2019.1617935>

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ZORA URL: <https://doi.org/10.5167/uzh-183145>

Journal Article

Accepted Version

Originally published at:

Kümbüloğlu, Övül; Yildirim, Bengisu; Al-Haj Husain, Nadin; Özcan, Mutlu (2019). Adhesion potential of relining materials to polyamide and PMMA-based denture base materials: effect of surface conditioning methods. *Journal of Adhesion Science and Technology*, 33(17):1939-1947.

DOI: <https://doi.org/10.1080/01694243.2019.1617935>

**Adhesion potential of relining materials to polyamide and PMMA-based denture base materials:**

**Effect of surface conditioning methods**

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**Short title:** *Adhesion of relining materials to polyamide and PMMA*

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**Abstract:** This study evaluated the bond strength of relining materials to different denture base materials polyamide and polymethylmethacrylate denture base materials after various surface conditioning methods. Denture base resin specimens (N=128; n=8 per group) (10x10x2.5 mm<sup>3</sup>) were fabricated out of injection-moulded thermoplastic polyamide resin (POL) (Deflex) and heat-polymerized polymethylmethacrylate (PMMA, Dura Dent) (HC). The specimens were randomly divided into 4 main groups according to different surface conditioning methods: a) No conditioning, control (C), b) grinding with green stone (G), c) application of primer (V), d) silica coating with Al<sub>2</sub>O<sub>3</sub> particles coated with SiO<sub>2</sub> (Rocatec) (R). Half of the specimens in each group received auto-polymerized hard relining resin (GC, GC Reline Hard), and the other half PMMA based relining resin (SC, Dura Dent). After thermocycling (x5000), the bonded specimens were tested under tensile forces (0.5 mm/min). Data (MPa) were analyzed using Mann Whitney U and Kruskal Wallis tests (alpha=0.05). Bond strength of relining resins were significantly higher to PMMA than to POL, regardless of the conditioning method (p<0.05). While R positively affected the bond strength results (p<0.05) (4.99±1.65-3.27±1.31), application V or G did not show significant effect to POL-relining resin adhesion. After R conditioning, bond strength values were significantly higher in HC-GC group (7.48±2.32) than POL-GC group (3.27±1.31) (p<0.05). Adhesion of auto-polymerized relining materials to thermoplastic polyamide or polymethylmethacrylate denture resins could be improved after surface conditioning with silica-coating.

**Keywords:** Adhesion; Air-abrasion; Denture base material; Polyamide; Polymethylmethacrylate; Silica-coating; Surface conditioning; Relining resin

## **Introduction**

In partial removable dentures, the metal denture bases and clasps are typically made of metals and compromise aesthetic expectations in visible areas of the mouth. Such metallic denture bases or clasps are replaced with thermoplastic polyamide resin since 1950 [1-4].

The advantages of polyamide resins are higher elasticity and toxicological safety for patients with resin monomer and metal allergy [5-10]. In addition, polyamide is an injection-moulded thermoplastic resin that do not yield to polymerization shrinkage and formation of non-polymerized residual monomers when compared to chemical polymerization [11]. Also, flexible and almost translucent clasps provide denture retention by utilizing undercuts of abutment teeth [12]. However, polyamide resins are prone to high water sorption and thereby decreased compressive strength, bacterial contamination, discoloration, and bonding difficulties with auto-polymerized resins [12-14]. Insufficient bond strength between polyamide or polymethylmethacrylate resins and relining resins [15], require initial surface conditioning of the denture resin in order to increase physico-chemical interactions with the relining resin that could be applied either at the laboratory or chairside where the latter is easier and faster than laboratory procedures [16].

After polymerization of chairside auto-polymerized relining resins, higher amounts of residual monomer are usually present and that allow for chemical reaction with the relining resin materials [12] but few studies evaluated the adhesion of relining materials to polyamide denture base materials using auto-polymerized relining resins in the literature [11,12,16].

The objective of this present study therefore, was to evaluate the bond strength between auto-polymerized relining resins and denture base resins after different surface conditioning methods. The null hypothesis tested was that surface conditioning methods would not show significant effect on the bond strength results.

## **Materials and Methods**

The materials used in this study are listed in Table 1.

### **Specimen preparation**

Denture base resin specimens (N=128; n=8 per group) (10x10x2.5 mm<sup>3</sup>) (Fig. 1) were fabricated out of injection-moulded thermoplastic polyamide resin (POL) (Deflex, Nuxen SRL, Argentina) and heat-polymerized polymethylmethacrylate (PMMA) (Dura Dent, Izmir, Turkey) (HC).

The specimens were randomly divided into 4 main groups according to different surface conditioning methods:

### **Surface conditioning methods**

No conditioning, control (C): In this group, denture resins were only polished with silicone carbide papers in the order of (1000, 1500, 2000 grit, for 20 s each, 1500 rpm) (SiC paper, Piramit, Istanbul, Turkey) and did not receive any conditioning.

Grinding with green stone (G): Denture resins were grinded with green stone (Stone Point Blaudent, Efes Dental, Bursa, Turkey) for 15 s at 500 rpm.

Application of primer (V): Denture resins were coated with one layer of primer using microbrush (V Primer, Sun Medical, Moriyama, Japan) and waited for its reaction for 5 minutes.

Silica coating (R): In this group, the specimens were air-abraded first with 110 µm Al<sub>2</sub>O<sub>3</sub> particles (Rocatec Pre, 3M ESPE, St. Paul, USA) and then with 110 µm Al<sub>2</sub>O<sub>3</sub> particles coated with SiO<sub>2</sub> (Rocatec Plus, 3M ESPE) at a pressure of 2.8 bars from a distance of 10 mm for 15 s. Conditioned surfaces were silanated with a silane coupling agent (ESPE-Sil, 3M ESPE) and waited 5 minutes for its reaction.

The conditioned specimens in each group were embedded in silicone moulds with the adhesion surfaces exposed and randomly divided into two groups. While one half received auto-polymerized hard relining resin (GC) (GC Reline Hard, Tokyo, Japan), the other half received PMMA-based relining resin (SC) (Dura Dent). Relining materials were placed onto the conditioned specimens and the polymerized according to each

manufacturer's recommendation. Table 2 presents the classification of specimens according to surface conditioning method.

The bonded specimens were then subjected to thermocycling (x5500, 5-55°C, dwell time: 20 s) (Esetron, Smart Robototechnologies, Ankara, Turkey).

### **Tensile bond strength test**

The bonded specimens were mounted in the jig of the Universal Testing Machine (Shimadzu, Kyoto, Japan) (Fig. 2) and then subjected to the tensile bond strength (TBS) test at a crosshead speed of 0.5 mm/min. TBS values were recorded in MPa.

### **Statistical analysis**

A sample size of 8 in each group was calculated to have more than 80% power to detect a difference with an effect size of  $f=0.364$  using a two-group Satterthwaite t-test (G\*Power software version 3.1.10, Heinrich Heine University, Düsseldorf, Germany) with a 0.05 two-sided significance level. Statistical analysis was performed using SPSS 18.0 software (SPSS Windows, Chicago, IL, USA). Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test normal distribution of the data. As the data were not homogeneously distributed, TBS data were analyzed using Mann-Whitney U and Kruskal Wallis tests where the bond strength was the dependent variable and conditioning methods (7 levels: C, G, V, R), liner resin (2 levels: GC and SC) and denture resins (2 levels: POL and PMMA) as independent variables. P values less than 0.05 were considered to be statistically significant in all tests.

### **Results**

Bond strength of relining resins to denture resins were significantly affected by the denture resin type ( $p<0.05$ ), surface conditioning method ( $p<0.05$ ) but not with the liner resin material. Interaction terms were significant ( $p<0.05$ ).

Regardless of the conditioning method, mean bond strengths of relining resins were significantly higher to PMMA than to POL ( $p < 0.05$ ).

While R positively affected the bond strength results ( $p < 0.05$ ), application V or G did not show significant effect to POL-relining resin adhesion (Table 2). After R conditioning, bond strength values were significantly higher in HC-GC group ( $7.48 \pm 2.32$ ) than POL-GC group ( $3.27 \pm 1.31$ ) ( $p < 0.05$ ).

There was no statistically significant difference between POL-SC ( $1.22 \pm 1.09$ ) and POL-GC ( $2.16 \pm 1.16$ ) ( $p > 0.05$ ).

## Discussion

This study evaluated the tensile bond strength between auto-polymerized relining resins and denture base resins after different surface conditioning methods. Since bond strength of relining resins to denture resins were significantly affected by the denture resin type, surface conditioning method, the null hypothesis tested could be rejected.

Polyamides are produced after a condensation reaction between the diamine  $\text{NH}_2\text{-(CH}_2\text{)}_6\text{-NH}_2$  and dibasic acid  $\text{CO}_2\text{H-(CH}_2\text{)}_4\text{-COOH}_3$  [3,14]. Polyamide is a crystalline polymer, and therefore highly chemical and heat resistant [12,14]. In the present study, as an accelerated aging test method, thermocycling was practiced for 5500 cycles between 5 and 55°C. However, thermocycling does not represent the oral environment completely since saliva, pH changes and dynamic fatigue loading are not simulated [17]. Thermocycling typically causes a significant reduction in bond strength by weakening the resin structure [16]. When considering the water sorption of polyamides, it is reasonable explain the lower bond strength obtained in conjunction with POL.

In the present study, the bond strength between relining resins and denture base resins was measured using tensile test. This type of test method is commonly used to measure the bond strength between lining materials and denture resins but in order to obtain accurate results it is necessary to be sensitive to the force applied to the specimens [2,12].

Different surface treatments are required to increase the insufficient bond strength between polyamide resins and auto-polymerized relining resins [9,16,18]. In the present study, the most effective surface treatment was the tribochemical silica coating for group POL-GC. This may be due to mechanical interlocking and chemical bonding between the relining resin and polyamide denture base resin. In tribochemical silica coating, silica-coated aluminum oxides are deposited onto the surface and their kinetic energy transforms the thermal energy. The silica particles remaining on the surface after the coating forms a chemical bond with the silane coupling agent [11]. It has been previously reported that silica coating can improve the bond strength of polyamide resin to a chemically polymerized resin [11,16,17]. In the present study, first Rocatec Pre (particle size of 110  $\mu\text{m}$ ) and then Rocatec Plus (particle size of 110  $\mu\text{m}$  coated with silica) were blasted onto the polyamide surface. Air-abrasion could be performed using different particle size. The reason for choosing this particle size was due to favourable results reported with this particle size [16,19]. Katsumata et al [11] also suggested the use of smaller particle size (Rocatec soft) of 30  $\mu\text{m}$  alumina coated with silica for tribochemical silica coating of polyamide resins. Although it is claimed that polyamide surface Rocatec Plus removes more material from the surface, this was not noted in the present study.

After the application of Rocatec system followed by silane coupling agent treatment, Katsumata et al [11] reported bond strengths of 16.41 MPa for specimens that were thermocycled and 13.18 MPa for those not thermocycled. On the other hand, Hamanaka et al [16] reported less bond strength (5.8-6.6 MPa) for polyamides after tribochemical silica bonding with Rocatec. In another study, Hamanaka et al [16] stated TBS values of (14.68 $\pm$ 2.71 MPa) between Lucitone FRS polyamide resin and chairside auto-polymerized relining resin and (13.41 $\pm$ 1.74 MPa) between Valplast polyamide resin- chairside auto-polymerized relining resin after tribochemical silica coating with Rocatec. In the present study, after tribochemical silica coating with Rocatec, in groups POL-SC (4.99 $\pm$ 1.65 MPa) and POL-GC (3.27 $\pm$ 1.31 MPa) lower TBS values were found. The difference could be attributed to thermocycling of the specimens. After thermocycling, similar results were obtained by Hamanaka et al [16] for polyamide (4.6 $\pm$ 1.6 MPa) and PMMA (5.7 $\pm$ 0.5 MPa) after tribochemical



silica coating. In the present study, without surface treatment for HC-SC group ( $10.03 \pm 1.11$  MPa) TBS value was found and after tribochemical silica coating ( $11.4 \pm 4.51$  MPa). The different test methods could be the reason of the difference. Katsumata et al [11] reported ( $3.18 \pm 1.3$  MPa) TBS for polyamide-relining resin and ( $12.59 \pm 2.12$  MPa) for heat polymerized denture resin-auto-polymerized relining resin. As a result, it is stated that Rocatec improves the adhesion results to polyamide resins. In this study, after surface conditioning with Rocatec, in POL-SC group ( $4.99 \pm 1.65$  MPa) TBS and in HC-SC group ( $11.4 \pm 4.51$  MPa) was noted found. The reason for the different results could be due to the selection of different brand of polyamides.

MMA is frequently preferable in dentures that also has better adhesion potential [20]. Further, organic solvents, such as chloroform, acetone, and methylene chloride (dichloromethane) have been used to enhance the adhesion [14,21-23]. In this regard, application of ethyl acetate was also proposed to prepare the surfaces of heat-polymerized denture base resins [19]. Also, when ethyl acetate and MMA were compared pretreatment with MMA increased the flexural strength of the polyamide denture base resin [18]. Nevertheless, chemically activated 4-META/MMA-TBB resin bonds to both of the chairside auto-polymerized relining resin and the silica layer [16,20]. Moreover, 4-META/MMA-TBB resin has smaller polymer particles which facilitates flowing on the surface [16,24]. The chemically polymerized and photo polymerized chairside relining resins are typically based on either PMMA or its copolymer polyethyl methacrylate (PEMA) [12]. Chairside auto-polymerized relining resin used in this study (GC Reline Hard) includes PMMA and silicon dioxide. Since this material is free of MMA in its composition, it does not cause chemical irritations to the oral tissues [20]. Also considering the low mean values and high standard deviations, the presence of MMA however was not sufficient to obtain acceptable results in conjunction with green stone or vinyl primer application, indicating that additional roughening was essential.

In this study, without surface conditioning in POL-SC and POL-GC groups presented significantly lower TBS which is in accordance with a previous study [12]. On the contrary, hard relining materials presented higher bond strength results to PMMA [2] which needs to be further investigated in conjunction with the surface

conditioning methods applied in this study after thermocycling. Nevertheless, both polyamide and PMMA denture resins benefitted from tribochemical silica coating for better adhesion of relining resins tested in this study.

## **Conclusions**

From this study, the following could be concluded:

1. Adhesion of relining resins to polyamide denture resin was inferior compared to polymethylmethacrylate.
2. Auto and heat-polymerized relining materials tested resulted in similar bond strength.
3. Compared to control group results, surface conditioning methods were to increase the adhesion of relining resin to polyamide and polymethylmethacrylate denture resins.
4. Tribochemical silica coating resulted in the highest bond strength between denture base materials and relining materials tested.

## **Clinical Relevance**

In order to improve bond strength of auto-polymerized denture liners to denture base materials of polyamide resin or polymethylmethacrylate, denture resin base should be best conditioned using air-abrasion of 110  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles coated with  $\text{SiO}_2$ .

## **Conflict of interest**

The authors did not have any commercial interest in any of the materials used in this study.

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## **Captions to figures and tables:**

### **Tables:**

**Table 1** Brands, abbreviations, chemical compositions and manufacturers of the materials used in this study.

**Table 2** Mean tensile bond strength values (MPa±Standard Deviations) of experimental groups. \*Uppercase letters in one column represent statistically significant differences between groups and small letters within groups ( $p<0.05$ ). See Table 1 for group abbreviations.

### **Figures:**

**Fig. 1** Dimensions of the specimen with the relining resin material bonded to denture base resin.

**Fig. 2** Schematic drawing of the specimen in the jig for the universal testing machine subjected to tensile bond strength test.



**Tables:**

<b>Material</b>	<b>Chemical Composition</b>	<b>Manufacturer</b>
Heat polymerized denture base resin (PMMA)	PMMA, MMA	Dura Dent, Erk Dental, Izmir, Turkey
Polyamide denture base resin (POL)	Polyamide	Deflex, Nuxen S.R.L., Buenos Aires, Argentina
Auto-polymerized relining resin (HC)	PMMA, MMA, N,N-Bis (2-hydroxyethyl)para toluidine	Dura Dent
Auto-polymerized relining resin (GC)	PMMA, Silicon dioxide	GC Reline Hard, GC, Tokyo, Japan
Stone Bur (G)	Silicon carbide	Stone point Blaudent, Efes Dental, Bursa, Turkey
Primer (V)	0.5% 6-(4-vinylbenzyl-n-propyl)amino-1,3,5-triazine-2,4-dithiol (VBATDT), 99.5% acetone	V-primer, Sun Medical, Moriyama, Japan
Rocatec Pre (R)	110 $\mu\text{m}$ $\text{Al}_2\text{O}_3$ particles	3M ESPE, St. Paul, USA
Rocatec Plus (R)	110 $\mu\text{m}$ $\text{Al}_2\text{O}_3$ particles coated with $\text{SiO}_2$	3M ESPE
ESPE-Sil	3-methacryloxypropyltrimethoxy silane in ethanol	3M ESPE

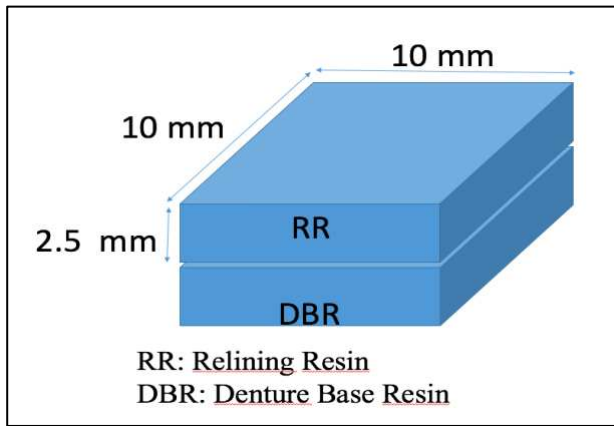
**Table 1** Brands, abbreviations, chemical compositions and manufacturers of the materials used in this study.

Surface Conditioning (n=8 per group)	Subgroups	Mean±Standard Deviation (MPa)
<b>C</b>	HC-GC	7.91±0.88 <sup>A,b</sup>
	POL-SC	2.61±2.68 <sup>B,b</sup>
	POL-GC	2.21±1.04 <sup>A,c</sup>
	HC-SC	10.03±1.11 <sup>A,a</sup>
<b>R</b>	HC-GC	7.48±2.32 <sup>A,b</sup>
	POL-SC	4.99±1.65 <sup>A,c</sup>
	POL-GC	3.27±1.31 <sup>A,c</sup>
	HC-SC	11.4±4.51 <sup>A,a</sup>
<b>V</b>	HC-GC	4.73±1.51 <sup>B,a</sup>
	POL-SC	1.10±0.83 <sup>B,b</sup>
	POL-GC	2.54±1.66 <sup>A,b</sup>
	HC-SC	5.97±0.68 <sup>B,a</sup>
<b>G</b>	HC-GC	4.34±1.44 <sup>B,b</sup>
	POL-SC	1.22±1.09 <sup>B,d</sup>
	POL-GC	2.16±1.17 <sup>A,c</sup>
	HC-SC	8.56±2.77 <sup>AB,a</sup>

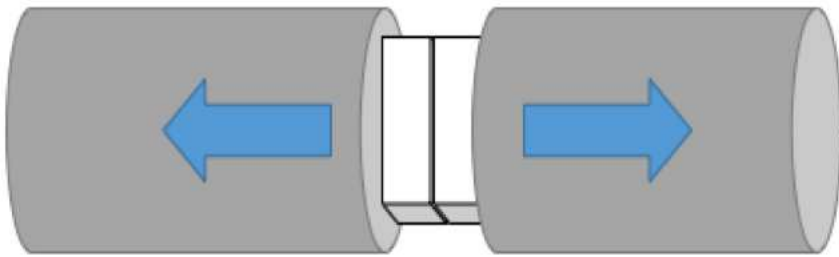
**Table 2** Mean tensile bond strength values (MPa±Standard Deviations) of experimental groups. \*Uppercase letters in one column represent statistically significant differences between groups and small letters within groups (p<0.05). See Table 1 for group abbreviations.



**Figures:**



**Fig. 1** Dimensions of the specimen with the relining resin material bonded to denture base resin.



**Fig. 2** Schematic drawing of the specimen in the jig for the universal testing machine subjected to tensile bond strength test.