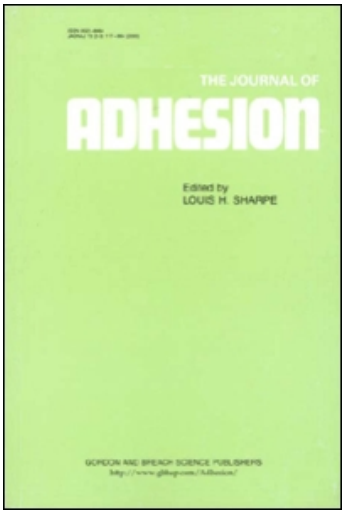

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Effect of Disinfection on Adhesion of Reline Polymers

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Cylinders (3.5 × 5.0 mm) of the reline resins Kooliner (K), New Truliner (N), Tokuso Rebase Fast (T), and Ufi Gel Hard (U) were bonded to cylinders (20 × 20 mm) of the denture base resin Lucitone 550 (L), and samples were divided into two controls and four test groups (n = 8). Shear tests (0.5 mm/min) were performed after polymerization or immersion in water (37°C) for 7 days (controls); two or seven cycles of disinfection by immersion in sodium perborate (50°C/10 min) or microwave irradiation (650 W/6 min). Statistical analyses (α = 0.05) revealed that two cycles of microwave and chemical disinfection increased the mean bond strengths of materials T (9.08 to 12.93 MPa) and L (18.89 to 23.02 MPa). For resin L, seven cycles of chemical (15.72 MPa) and microwave (17.82 MPa) disinfection decreased the shear bond strength compared with the respective control (21.74 MPa). Resins U (13.12 MPa), K (8.44 MPa), and N (7.98 MPa) remained unaffected.

Keywords: Bond strength; Dental polymers; Microwave irradiation

INTRODUCTION

Hard chairside reline resins have been reported to be clinically useful to improve denture retention and patient comfort [1,2]. After relining,

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adjustments of the denture bases with rotary instruments are often required, producing rough surfaces that can lead to mucosal irritation and promote microbial adherence and colonization [3]. Therefore, denture polishing is needed to achieve smooth surfaces. During polishing procedures, opportunistic pathogenic microorganisms [4] can be transferred from dentures to pumice, thus resulting in cross-contamination between patients and dental personnel [5]. Hence, dentures should be disinfected before being sent to the laboratory and before insertion [4,5]. A previous clinical study has demonstrated that an infection-control protocol, which included scrubbing the dentures with 4% chlorhexidine combined with immersing in sodium perborate solution at 50°C for 10 min, was effective in reducing the microbial growth on dental prostheses [6]. Similarly, microwave irradiation for 6 min in water at 650 W performed on contaminated hard reline specimens also proved to eliminate pathogenic microorganisms [7].

The disinfection method should inactivate the microorganisms without compromising the denture base materials. Previous studies investigated the effects of microwave and chemical disinfection on properties such as hardness [8], surface roughness [9], flexural strength [10], and dimensional stability [11]. Another concern when hard chairside reline resins are used is the strength of the adhesion between the reline and the denture base material [12–15]. A strong bond is needed to prevent delamination of the two materials, harboring of bacteria, and staining [1,2,16]. The adhesion can be improved by the application of bonding agents [12,14,16,17], methyl methacrylate monomer [14,17,18], or other organic solvents [14,15,18] on the denture base resin surface. These treatments cause the polymer surface to swell, and the adhesion is obtained by interdiffusion of monomers of the reline resin into the swollen denture base polymer structure, polymerization, and formation of an interpenetrating polymer network (IPN) [12–14]. Increased depth of the swollen layer on the denture base surface and high bond strength have been observed for reline bonding agents having 2-hydroxyethyl methacrylate (HEMA) [12].

Disinfection by immersion in disinfectant solutions was found to adversely affect the strength of repairs made with autopolymerizing resins, causing adhesive failures at the repair joint. This suggested that the penetration of the components of the disinfectant solutions may occur more readily at the interface [19]. In contrast, microwave irradiation in a dry condition reduced the residual monomer content [20,21] and improved the flexural strength of heat-polymerizing specimens repaired with autopolymerizing acrylic resin [21]. A study on the effect of thermal cycling on the bond strength of autopolymerizing

acrylic resin repairs revealed a significant decrease in the fracture load, probably due to water sorption and thermal stress [22]. Conversely, an increase in bond strength between composite resins after thermoclycing has been observed when a HEMA-containing adhesive was used. This was attributed to the plasticizing effect of the water absorbed by the adhesive layer, which acted as a stress breaker at the bonding interface [23]. During the infection control protocol or microwave disinfection methods, the materials are exposed to chemical solution, microwave irradiation, and water at increased temperature. Therefore, the bond strength between the reline and denture base resins may be affected.

The aim of this study was to evaluate the effect of chemical and microwave disinfection on the bond strength between a heat-polymerized denture base resin and four autopolymerizing reline resins. The bond strength of the denture base acrylic resin relined with the same material was also evaluated for comparison. The null hypothesis was that both disinfection methods could be used without adverse effects on the bond strength between the denture base resin and the reline materials.

MATERIALS AND METHODS

The product names, batch numbers, manufacturers, compositions, powder/liquid proportions, and polymerization cycles of the materials studied are listed in Table 1.

In preparing the specimens, PVC tubes (Tigre S/A, Tubos e Conexões, Joinville, SC, Brazil) were used for fabrication of wax (Wilson, Polidental Indústria e Comércio Ltda, São Paulo, SP, Brazil) cylinders (20 mm × 10 mm), which were then invested in flasks (OGP 3.0, OGP Produtos Odontológicos, São Paulo, Brazil) using Type IV dental stone (Troquel Quatro, Polidental Indústria e Comércio Ltda, São Paulo, SP, Brazil). After elimination of the wax, the denture base resin Lucitone 550 was mixed and packed into the PVC tubes using a hydraulic press (Vipi Dental, Pirassununga, São Paulo, SP, Brazil). A total of 240 denture base resin cylinders, 48 for each denture base/reline material combination, were polymerized in a water bath (P-100, Termotron equipamentos, Piracicaba, SP, Brazil) using the short cycle recommended by the manufacturer (Table 1). After polymerization, the flask was left to cool at room temperature for 30 min, followed by 15 min under running water. Specimens were removed from the flasks and stored in distilled water at $37 \pm 1^{\circ}C$ for 50 ± 2 h [24].

After water storage, the denture base resin surfaces to be bonded were smoothed with 240-grit silicone carbide paper (3 M do Brazil,

TABLE 1 Materials Used in This Study

Material	Batch	Manufacturers	Powder composition	Liquid		Powder/ liquid ratio (g/mL)	Polymerization cycles
				Composition	Molecular weight		
Kooliner	0201102	GC America Inc, Alsip, IL, USA	PEMA	IBMA	142	2.1 g/1.0 mL	10 min at room temperature
New Truliner	0310-528	The Bosworth Co., Skokie, IL, USA	PEMA	IBMA DBP	142 278	2.01 g/1.5 mL	20 min at room temperature
Tokuso Rebase Fast	U570612	Tokuyama Dental Corp., Tokyo, Japan	PEMA	MAOP 1,6-HDMA	186 254	2.056 g/1.0 mL	5.5 min at room temperature
Ufi Gel Hard	025292	Voco, Cuxhaven, Germany	PEMA	1,6-HDMA	254	2.12 g/1.2 mL	7 min at room temperature
Lucitone 550	P-65173 L-37375	Dentsply Indústria e Comércio Ltda., Petrópolis, RJ, Brazil	PMMA	MMA EDGMA	100 198	2.1 g/1.0 mL	90 min at 73°C and 30 min at 100°C

Notes. PEMA, poly (ethyl methacrylate); PMMA, poly (methyl methacrylate); IBMA, isobutyl methacrylate; DBP, di-*n*-butyl phthalate; MAOP, *β*-methacryloyl oxyethyl propionate; 1,6-HDMA, 1,6-hexanediol dimethacrylate; MMA, methyl methacrylate; and EDGMA, ethylene glycol dimethacrylate.

Ribeirão Preto, SP, Brazil) in an automatic grinding and polishing unit (Metaserv 2000, model 95–2829, Buehler UK Ltd., Coventry, England) at 350 r.p.m. for 40 s to simulate clinical relief of the denture base for bonding of the reline resins. The 240-grit paper has been used for surface preparation in investigations on the bond strength between hard reline and denture base acrylic resins [14,17]. The surfaces were washed with liquid detergent (Limpol, Bombril-Cirio, São Paulo, SP, Brazil) for 20 s, rinsed in distilled water, and blot dried. The surfaces were then treated according to the manufacturer's instructions for each hard reline material, with the exception of Kooliner resin, for which the bonding sites were prepared by painting the surfaces with Lucitone monomer for 180 s. This procedure was based on the results of a previous study [18], which demonstrated that wetting the denture base resin surface with Lucitone 550 monomer improved the sites for bonding and promoted the highest bond strength for material Kooliner. A masking tape with a 3.5-mm-diameter circular opening was placed on the treated denture base surfaces to provide a uniform bonding area (9.62 mm^2).

A specially designed metal split mold having a circular opening (3.5 mm diameter×5.0 mm length) was used for the relining procedures (Figure 1). The denture base cylinder was placed in the mold and secured *via* screws, so that the metal mold opening position coincided with the masking tape opening position. The autopolymerizing reline materials were then mixed according to the manufacturers'

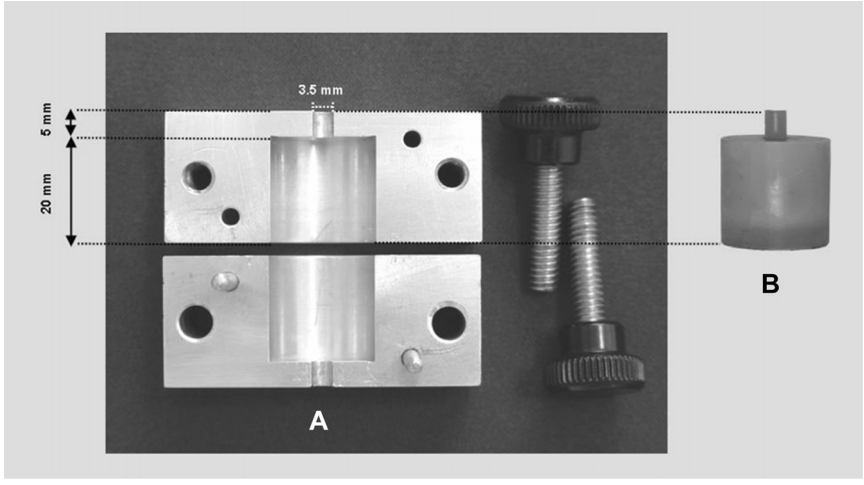

FIGURE 1 Metal split mold (A) and specimen (B).

instructions and inserted into the 3.5 mm×5.0 mm split mold opening. An acetate sheet was placed over the material, and pressure was applied until polymerization was completed. The screws were loosened, the two parts of the mold were separated, and the relined specimen was removed.

When the specimens were relined using the heat-polymerizing acrylic resin Lucitone 550, initially a metal die (3.5 mm in diameter and 5.0 mm long) was directly glued to the center of the bonding surface with a small drop of cyanoacrylate glue (Super Bonder, Henkel Loctite Products, Rocky Hill, CT, USA). The denture base cylinders were then invested in silicone (Zetaplus, Zhermack, Badia Polesine, Rovigo, Italy), further supported by type IV dental stone within the flasks. After the stone was set, the flask was opened, the metal die was removed, and the bond surface was prepared as described and treated with Lucitone 550 monomer for 180 s [25]. The masking tape was positioned on the bonding surface, and the denture base acrylic resin Lucitone 550 was mixed, inserted into the silicone mold, and polymerized (Table 1). After polymerization, the flask was cooled to room temperature, and the specimens were deflasked and stored in distilled water at 37 ± 1°C for 50 ± 2 h [24].

The 48 specimens of each reline material were divided into two control groups and four test groups of eight specimens each (Table 2). Specimens from ICP 2 and MW 2 test groups were disinfected twice to simulate disinfection when contaminated dentures come from the patient and before being returned to the patient. For test groups 2 (ICP 7) and 4 (MW 7), the specimens were submitted to a total of seven cycles of disinfection using the infection control protocol or microwave irradiation. The specimens were disinfected daily for 7 days and were stored in water at 37°C between disinfection cycles [10]. Daily

TABLE 2 Groups and the Disinfection Methods Used in the Study

Group	Disinfection method
C1 (control 1)	No submission to any disinfection method
C2 (control 2)	Immersed in distilled water at 37°C for 7 days
ICP 2	Disinfected twice using a disinfection control protocol (scrubbing with 4% chlorhexidine for 1 min, immersing in 3.8% sodium perborate solution at 50°C for 10 min, and immersing in water for 3 min)
ICP 7	Disinfected seven times using the disinfection control protocol
MW 2	Disinfected twice using microwave disinfection (immersed in 200 ml of water and irradiated with 650 W for 6 min)
MW 7	Disinfected seven times using microwave disinfection

disinfection was chosen because a number of follow-up visits for denture base adjustments may be required after relining. Thus, dentures can be exposed to repeated disinfections during this period. Considering that the number of recall appointments may vary among patients, seven disinfection cycles were chosen randomly and intended to detect any possible cumulative effect of the disinfection methods on the bond strength of the materials evaluated.

For shear bond tests, each specimen was mounted in a metal holder on the universal testing machine (MTS-810, Material Test System, Éden Prairie, MN, USA) and loaded in air at room temperature ($23 \pm 2^{\circ}C$) with a knife-edged blade positioned parallel to the material interface at 0.5 mm/min [26]. The shear bond strengths (MPa) were calculated by dividing the force required to break the specimen by surface area of adhesion (9.62 mm^2).

Data from shear tests were analyzed using two-way analysis of variance (ANOVA), followed by the Tukey honestly significant difference (HSD) *post hoc* test. Significance level was set at $P = 0.05$.

RESULTS

The two-way ANOVA revealed significant ($P < 0.001$) differences in the shear bond strength for the variables material and group, and

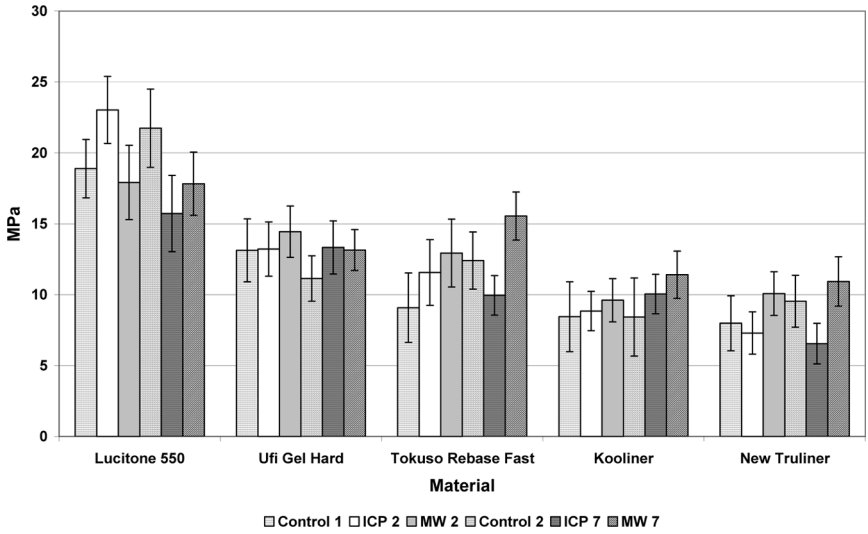

FIGURE 2 Mean shear bond strength of controls and experimental groups.

their interaction. The means and standard deviations for shear bond strength are shown in Figure 2. For Lucitone 550 material, ICP 2 specimens exhibited significantly higher mean shear bond strength than control C1 specimens ($P = 0.014$). In both disinfection methods, the mean bond strength values of Lucitone 550 specimens submitted to seven cycles of disinfection were significantly lower (ICP 7: $P < 0.001$; MW 7: $P = 0.030$) than those of the specimens immersed in water for 7 days (C2). After two cycles of microwave disinfection (MW 2), Tokuso Rebase Fast material showed a significantly higher mean bond strength value than that of control group C1 ($P = 0.039$). For New Truliner, no significant differences in shear bond strength were found between the experimental groups (ICP 2 and MW 2, ICP 7 and MW 7) and their respective controls (C1 and C2), regardless of the disinfection method used. No significant changes were observed for materials Ufi Gel Hard and Kooliner in all groups evaluated ($P > 0.05$).

DISCUSSION

Different test methods have been proposed for evaluating the bond strength between hard reline and denture base acrylic resins. The shear bond test used in the present study applies a shear load directly to the reline–denture base junction and represents better than tensile load the stresses that the interface between the polymers are subjected to clinically [13]. This test has been used by several investigators to determine the bond strength between denture polymers [13–15,17,27].

In a relining procedure, it is recommended that the denture base resin should be treated with monomer, solvent, or bonding agent [12,14–18] to achieve adequate adhesion between the two acrylic resins. Because of the diffusion of these chemicals into the polymer, the denture base surface swells [12,14], thus increasing the distance between the polymeric chains. During the contact time of the reline material to the bond surface, the monomers of the reline resin diffuse into the swelled phases of the denture base polymer and become interlocked, forming an interpenetrating polymer network (IPN) after polymerization [12–14,28].

Microwave and chemical disinfection did not adversely affect the shear bond strengths of the autopolymerizing reline resins to the denture base acrylic resin. In fact, two cycles of microwave disinfection significantly increased the mean bond strength for Tokuso Rebase Fast material. This finding could be related to the heating of the resins during microwave disinfection, because the water in which the

specimens were immersed started to boil after approximately 1 min and 30 s. The whole monomer is not always converted into polymer [29–33], and this residual monomer may adversely affect the properties of the polymerized resins [31]. The rise in temperature during microwave disinfection may have facilitated the residual monomer release [29,32] and further polymerization reaction [30], thereby reducing the monomer molecules, which may be present as a residue in the polymerized resin. Consequently, the strength of the interpenetrating polymer formed at the interfacial region and the cohesive strength of the relining material, which exists close to the interface, may have been improved. As a result, the bond strength between the reline material Tokuso Rebase Fast and the denture base resin Lucitone 550 was increased after two cycles of microwave disinfection [21]. The results also demonstrated that no further increase in shear bond strength occurred after seven cycles of microwave disinfection or 7 days of immersion in water at 37°C. These findings suggest that two cycles of microwave irradiation might have accelerated the reduction of residual monomer, and consequently, the increase in bond strength was achieved earlier.

The increased bond strength observed for Lucitone 550 after two cycles of chemical disinfection was not expected because the residual monomer in heat-polymerized acrylic resins is usually lower than that of autopolymerized acrylic resins [32]. One possible explanation could be the polymerization cycle used for processing the specimens. Although a terminal boil was included, a short period of 30 min at 100°C was used. This probably resulted in lower degree of conversion, which might have been improved after immersion of Lucitone 550 specimens in the disinfectant solution at 50°C [33].

After seven cycles of disinfection, Lucitone 550 specimens exhibited significantly lower mean bond strength than that of the specimens immersed in water for the same period of time, regardless the disinfection method used. Hence, the null hypothesis was rejected. These findings were probably due to the water uptake process. During repeated exposure to the disinfection procedures, the increased temperature may have enhanced the diffusion of the water within the polymeric chains. It is likely that the water molecules absorbed into the resin produced a plasticizing effect, thus weakening not only the Lucitone 550 material [34] but also the interface denture base/reline resin. Whether the changes in bond strength caused by additional disinfection cycles would be of clinical significance requires further investigation.

The resins Ufi Gel Hard, Kooliner, and New Truliner showed no measurable changes in the mean shear bond strength values after

both disinfection methods. The denture base surface treatment used for each material may partially explain these findings. The bonding agent for Ufi Gel Hard contains the nonpolymerizable solvent dichloromethane [14], which increases the ability of the reline material to intermix within the denture base swollen layers, allowing a strong bond to form [12]. Ufi Gel Hard bonding agent also contains the monomer 2-hydroxy ethyl methacrylate (HEMA), which has been considered a suitable adhesive for diffusing into the polymethyl methacrylate (PMMA) because its solubility parameter is close to that of PMMA substrate to be dissolved [28]. In addition, the low-molecular-weight HEMA can effectively penetrate into the PMMA [35] and polymerize along with the reline resin [16], thus enhancing the bonding. The bonding agent of New Truliner material contains methyl methacrylate (MMA) [16], which is also contained in the liquid of Lucitone 550 material used for wetting the bonding surface when Kooliner reline resin was used. Bonding agents containing MMA monomer have good swelling properties and also the ability to introduce the small MMA molecules into the denture base polymer for good bonding [12].

Although none of the autopolymerizing hard reline resins were detrimentally affected by the disinfection methods, some limitations should be kept in mind when these findings are interpreted. The specimens tested were different from actual denture configuration and were not subjected to cycling loading to simulate the repetitive mechanical stress that the dentures are exposed to during mastication. These aspects should be considered in further investigations.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following were concluded:

For the autopolymerizing reline resins, there were no apparent important differences in the shear bond strength that arose as a result of chemical and microwave disinfection. Conversely, the repeated exposure to both disinfection methods adversely affected the shear bond strength only when the specimens were relined with the heat-polymerizing denture base acrylic resin.

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