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Shear Bond Strength of Denture Teeth to Heat- and Light-Polymerized Denture Base Resin

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Keywords
Diatoric; bonding agent; bonding strength; denture base resin; urethane dimethacrylate; poly(methyl methacrylate).

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Supported in part by an ACPEF Research Fellowship.

Accepted 2 December 2009

doi: 10.1111/j.1532-849X.2010.00639.x

Abstract

Purpose: To evaluate the shear bond strengths of highly cross-linked denture teeth bonded to heat-polymerized poly(methyl methacrylate) (PMMA) or a light-polymerized urethane dimethacrylate (UDMA) denture base resin with or without a diatoric and with or without an acrylate bonding agent.
Materials and Methods: The denture base resins tested were Lucitone 199 (heat-polymerized PMMA) and Eclipse (light-polymerized UDMA). One hundred sixty mandibular central incisor denture teeth were divided into four groups ($n = 40$): group 1: ground surface as control; group 2: ground surface with diatoric; group 3: ground surface with bonding agent; group 4: ground surface with bonding agent and diatoric. Half of each group ($n = 20$) was processed with either heat- or light-polymerized resin. All specimens were treated with thermocycling for 1000 cycles, alternating between 5 and 55°C with a dwell time of 30 seconds. Half the specimens in each group were treated with cyclic loading at 22 N for 14,400 cycles at 1.5 Hz. All specimens were tested with shear load to failure. Data were analyzed with student's *t*-test, 2- and 3-way ANOVA, and Dunnett's T3 method ($p < 0.05$).
Results: Statistical analysis demonstrated no significant effect on shear bond strength from cyclic loading. For the Lucitone 199 (L) specimens, mean shear bond strengths and standard deviations were (N) 66.5 ± 28.4, 72.7 ± 31.5, 80.6 ± 17.1, and 76.9 ± 21.9 for groups 1L, 2L, 3L, and 4L, respectively. For the Eclipse (E) specimens, mean shear bond strengths and standard deviations were (N) 3.7 ± 1.2, 7.3 ± 3.3, 90.0 ± 20.7, and 94.2 ± 17.8 for groups 1E, 2E, 3E, and 4E, respectively. No statistically significant differences in shear bond strengths were noted for the Lucitone 199 groups ($p = 0.11$). Eclipse shear bond strengths were significantly higher in groups 3E and 4E than in groups 1E and 2E ($p \leq 0.05$). In a 3-way ANOVA for groups 3 and 4, the shear bond strengths for the Eclipse specimens were significantly higher than the Lucitone 199 specimens ($p = 0.01$).
Conclusions: When evaluating the shear bond strength of IPN denture teeth to denture base resins, specimens using an acrylate bonding agent with the Eclipse (light-polymerized) resin yielded significantly higher shear bond strengths than all of the Lucitone 199 groups and the Eclipse resin groups without a bonding agent.

Denture tooth debonding is an ongoing concern exacerbated with the introduction of complex implant-supported prostheses.[1,2] Dismantling and repairing highly sophisticated acrylic-veneered implant prostheses can inconvenience the patient and challenge the clinician. To minimize the need for repairs, many authors have investigated the most likely union of materials and methods to provide optimal bond strength of the denture tooth to the denture base.

Attempts to improve bond strength between denture teeth and the denture base by mechanical preparation of the denture tooth ridge-lap demonstrated mixed results.[3-7] Additionally, most applied chemicals etch the surface of the denture tooth by changing the morphology and the chemical properties of the materials.[8] Priming the denture tooth surface with methyl methacrylate monomer liquid during processing yielded significantly higher bond strength values than other surface treatments.[8-10] Generally, this change is obtained by wetting the ridge-lap surfaces with methyl methacrylate monomer.[11] Organic solvents such as chloroform,[12] acetone,[8,13] and methylene chloride[14,15] have also been used for this process. Many studies have reported that these organic solvents increase the bond strength of the denture tooth to the denture base.[12-15] In some studies the type of denture tooth demonstrated a significant difference in the bond strength. Takahashi et al found highly cross-linked denture teeth to have significantly lower bond strengths than other acrylic denture teeth.[5]

Poly(methyl methacrylate) (PMMA) is the most commonly used material for denture base fabrication; however, there have been reports of allergic response in patients sensitive to methyl methacrylate monomer.[16] While researchers have turned to the visible light-polymerized resins as an alternative to PMMA, the light-polymerized denture base products demonstrated a reduction in the bond strength of the denture tooth to the base material.[17-19] The shear bond strength between light-polymerized denture base resin and denture teeth was increased with the use of a bonding agent.[19-21]

A new light-polymerized urethane dimethylacrylate (UDMA) denture resin system (Eclipse, Dentsply International, York, PA) claimed to have improved bond strength with the use of a bonding agent. Recent publications demonstrate mechanical properties of the material comparable with traditional PMMA denture base resins.[22-24] The Eclipse resin system combines three resins that handle similar to wax and eliminate the need for flasking, boil-out, and long processing times: (1) base, (2) set-up, and (3) contour resin. First, a processed base is light polymerized, and then the teeth are added with the set-up resin. Subsequently, the contour resin is used to simulate the gingival tissues. Finally, the denture is light-polymerized and polished with traditional techniques for conventional denture bases.

Traditionally, most published data assessing the bond strength of denture teeth to denture base resins were recorded with static loads in either compression or tension.[5,25,26] These methods do not take into account the dynamic forces associated with oral function. Thermocycling and cyclic loading have been used to simulate the oral environment;[27,28] however, there is limited information on the strength of the denture tooth bond to the acrylic denture base resin systems after being exposed to thermocycling and cyclic loading.

The purpose of this investigation was to evaluate the shear bond strengths of highly cross-linked denture teeth with or without a diatoric bonded to heat-polymerized PMMA and a light-polymerized UDMA denture base resin, with or without the application of a bonding agent. Thermocycling and cyclic loading were used during testing to more closely simulate the oral environment. The null hypothesis was based on the assumption that there will be no significant difference in shear bond strength of denture teeth to the two denture base resins in the four surface treatment groups with or without cyclic loading.

Materials and methods

This investigation evaluated the shear bond strengths of a heat-polymerized PMMA (Lucitone 199, Dentsply International) and a light-polymerized UDMA (Eclipse) to a highly cross-linked acrylic denture tooth (Portrait IPN, Dentsply International). The effect of an acrylate bonding agent (Eclipse Bonding Agent, Dentsply International) was tested and analyzed. A total of 160 mandibular central incisor denture teeth were divided into four groups (n = 40): group 1: ground surface; group 2: ground surface with a diatoric; group 3: ground surface with a bonding agent; group 4: ground surface with a bonding agent and a diatoric (Table 1).

Table 1 Surface treatment groups used in the Lucitone 199 (L) and Eclipse (E) specimens (n = 20)

Product	Group	Surface treatment
Lucitone 199	Group 1L	Ground surface with monomer
–	Group 2L	Ground surface with monomer and a diatoric
–	Group 3L	Ground surface with bonding agent
–	Group 4L	Ground surface with bonding agent and a diatoric
Eclipse	Group 1E	Ground surface
–	Group 2E	Ground surface with a diatoric
–	Group 3E	Ground surface with bonding agent
–	Group 4E	Ground surface with bonding agent and a diatoric

To provide a uniform surface for bonding, all the dentures were ground on the ridge-lap area using a standard silicone mold (Sil-Tech, Ivoclar Vivadent, Amherst, NY) with an open window. The ridge-lap was ground with a carbide bur (Tungsten carbide cutter, H251GE, Brasseler USA, Savannah, GA) using a low-speed handpiece (K9, KaVoEWL, Leutkirch, Germany) at a speed of 15,000 rpm using magnification loupes (Optical Loupes 2.5×, Orascopic, Middleton, WI). A new bur was used after preparation of each group of 20 teeth to ensure similar grinding efficiency for all specimens. After grinding, the teeth were placed in distilled water at room temperature in an ultrasonic unit for 10 minutes and rinsed twice with distilled water. Two additional teeth were attached with wax to a plastic replica of the base ($36 \times 12 \times 6$ mm^3) and pressed into soft silicone. This generated a standard silicone mold that was used to fabricate the specimens for the study (Fig 1).

Half the teeth from each group (n = 20) were processed with Lucitone 199 denture base resin. Two teeth were placed in the silicone mold, and it was filled with warmed base-plate wax (TruWax, Dentsply International). Using magnification loupes (2.5×), the excess wax was removed, and the wax patterns were flasked with plaster (Mounting Plaster, WhipMix, Louisville, KY) in the base of the flask and dental stone (Microstone, WhipMix) over the specimens. Tinfoil substitute (Separating Fluid, Ivoclar Vivadent, Schaan, Liechtenstein) was used as a separating medium. The flasks were warmed in the dewaxing unit (Boil-Out Unit-KaVo TYP 5522, KaVoEWL) at 90°C, and the stone and teeth were cleaned with hot soapy water (Tide Detergent Powder, Proctor and Gamble, Cincinnati, OH) to remove the wax. Tinfoil substitute was applied with care to avoid coating the teeth (Fig 2). Before processing, the teeth were treated according to their assigned groups (Table 1). Lucitone 199 resin

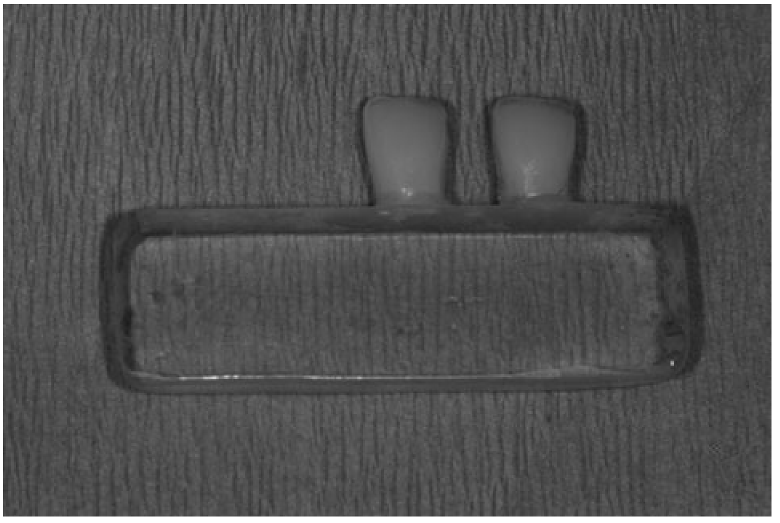


Figure 1 The example pattern used to fabricate the silicone mold used to form the specimens.

was prepared according to the manufacturer's instructions, trial packed to 3000 psi, and processed in the polymerization unit (Polymerization Unit-KaVo TYP 5506, KaVoEWL) for 9 hours. After processing, the specimens were placed in stone remover solution (Plaster and Stone Remover, Heraeus Kulzer, Armonk, NY) for 2 hours, and any "flash" was removed with a carbide bur (Tungsten Carbide Cutter, H78E, Brasseler USA) using magnification loupes (2.5×).

Half the teeth from each group (n = 20) were processed with Eclipse denture base resin. The Eclipse-processed bases were fabricated by placing base material into preformed silicone molds ($36 \times 12 \times 6$ mm^3) and polymerized in the light-processing unit (Eclipse Light Processing Unit, Dentsply International) for 10 minutes. Two teeth were treated with their respective surface treatments according to their group (Table 1) and placed in the silicone mold with the Eclipse resin base. Set-up resin was flowed between the teeth and the base with a hot instrument (Electric Waxer, Dentsply International). After cooling, the specimens were removed from the silicone mold, and any excess from around the teeth was removed using magnification loupes (2.5×). These specimens were warmed in a 55°C oven (Eclipse Oven, Dentsply International) for 1 hour, and coated with air barrier coating (Triad Air Barrier Coating, Dentsply International). The specimens were processed in the light-processing unit for 10 minutes.

In Groups 2 and 4, the diatoric was prepared by grinding into the ridge-lap with a #4 round bur (Carbide Cutter H1.31.014, Brasseler USA) with dimensions of 1×2 mm^2 using magnification loupes (2.5×) (Fig 3). After grinding the diatoric, the teeth were placed in distilled water in the ultrasonic unit for 10 minutes and rinsed twice with distilled water. In groups 3 and 4, the teeth were treated with Eclipse Bonding Agent following the manufacturer's instructions.

After the specimens were fabricated, they were stored in distilled water at 37°C for 7 days. Then all the specimens were treated with thermocycling; 1000 cycles alternated from 5 to

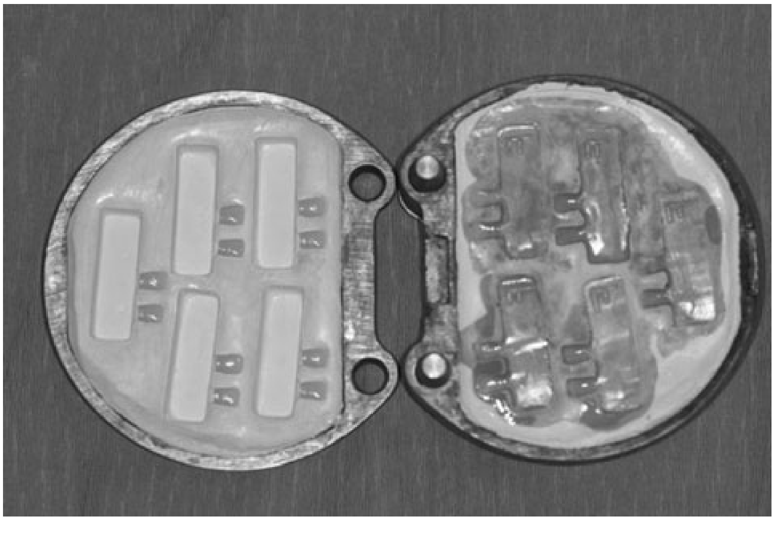


Figure 2 Denture flask prepared for processing the Lucitone specimens.

Figure 3 Cross-section of the tooth specimen with a prepared diatoric.

55°C with a dwell time of 30 seconds. After thermocycling, half the teeth were stored for an additional 28 days and then treated with cyclic loading at 22 N for 14,400 cycles at 1.5 Hz in a fatigue machine (Coil Cycler, Proto-Tech, Portland, OR), which allowed for constant submersion of the specimens in 37°C distilled water during treatment. The load was applied to the incisal edge at a 135° angle to the long axis. The specimens were tested for shear bond strength in a force gauge (DART Series Digital Force Gauge, Shimpo, Itasca, IL) on a motorized stand (Programmable Motorized Test Stand, Shimpo). The load was applied to the incisal edge at a 135° angle to the long axis with a crosshead speed of 5 mm/min until failure.

The debonded surfaces of the teeth and the bases were observed under a light microscope (Carolina Biological Supply Company, Burlington, NC) to record the mode of failure. Representative samples were selected randomly and observed after sputtering with a platinum conductive layer of approximately 30 nm in a scanning electron microscope (FE-SEM, JSM 7000F, JEOL Ltd., Tokyo, Japan) with an acceleration voltage of 10 kV and a working distance of 25 mm. The mode of failure was classified as (1) adhesive failure (clean break at the bond), (2) cohesive failure (full break in the base material or tooth), and (3) mixed failure (a combination of adhesive and cohesive failure).

Two-way ANOVA was used for comparison of mean shear bond strength by group and cyclic loading condition for Lucitone 199 (L). For Eclipse (E), cyclic and noncyclic loading were compared separately, because groups 1E and 2E broke

Table 2 Lucitone 199 (L) mean shear bond strengths with standard deviations

	Cyclic loading			No cyclic loading		
	Mean (N)	SD	n	Mean (N)	SD	N
Group 1L	68.0	23.4	9	**66.5**	**28.4**	10
Group 2L	75.9	35.6	9	72.7	31.5	10
Group 3L	83.8	18.7	10	80.6	17.1	10
Group 4L	94.9	16.2	9	76.9	21.9	10

Groups: 1, ground surface with monomer; 2, ground surface with monomer and a diatoric; 3, ground surface with Eclipse bonding agent; 4, ground surface with Eclipse bonding agent and a diatoric. N = Newton; SD = standard deviation; n = number of samples.

before cyclic loading. A two-sided two-sample *t* test was used for cyclic loading and a one-way ANOVA for the noncyclic loading mean shear bond strengths. Posthoc test (Dunnett's T3 method) was used to determine the difference between the Eclipse groups without cyclic loading. Also, a 2-way ANOVA compared cyclic and noncyclic loading for the Eclipse specimens. A 3-way ANOVA compared the two denture base resins in groups 3L, 4L, 3E, and 4E using the parameters denture base, surface treatment, and cyclic loading condition. For all statistical analysis the significance level was set at $p < 0.05$. The frequency for each mode of failure (adhesive, mixed, cohesive) was reported for each group.

Results

The mean shear bond strengths between denture teeth and two denture base resins using four surface treatment groups with or without cyclic loading are listed in Tables 2 and 3. A 2-way ANOVA comparing the mean shear bond strengths by surface treatment group and cyclic loading condition in the Lucitone 199 specimens demonstrated no statistically significant difference ($p = 0.71$). Tests for main effects indicated that there were no statistically significant differences among the four surface treatment groups ($p = 0.11$) or between the two cyclic loading conditions ($p = 0.26$).

The two Eclipse resin groups, groups 1E and 2E, were not able to undergo the cyclic loading treatment because the

Table 3 Eclipse (E) mean shear bond strengths with standard deviations

	Cyclic loading			No cyclic loading		
	Mean (N)	SD	n	Mean (N)	SD	n
Group 1E	*	–	–	3.7	1.2	10
Group 2E	*	–	–	7.3	3.3	10
Group 3E	100.2	32.2	10	90.0	20.7	10
Group 4E	100.9	19.6	10	94.2	17.8	10

Groups: 1, ground surface; 2, ground surface with a diatoric; 3, ground surface with Eclipse bonding agent; 4, ground surface with Eclipse bonding agent and a diatoric. N = Newton; SD = standard deviation; n = number of samples.
*Eclipse specimens without a bonding agent did not undergo cyclic loading, as they broke with a force well below 22 N.

Table 4 Mode of failure with number of teeth in each category

Product	Group	Adhesive	Mixed	Cohesive
Lucitone 199	Group 1L	8 (40)	9 (45)	3 (15)
	Group 2L	6 (30)	10 (50)	4 (20)
	Group 3L	0	11 (55)	9 (45)
	Group 4L	0	9 (45)	11 (55)
Eclipse	Group 1E	20 (100)	0	0
	Group 2E	20 (100)	0	0
	Group 3E	0	11 (55)	9 (45)
	Group 4E	0	7 (35)	13 (65)

The mode of failure for each surface treatment group with the Lucitone 199 (L) and Eclipse (E) specimens are shown. Number of teeth and percentage (in parentheses) specimens are expressed.

specimens broke with minimal force (Table 3), so statistical analysis was performed separately for the two loading conditions. A two-sided 2-sample *t* test demonstrated no statistically significant difference in shear bond strengths between groups 3E and 4E under the cyclic loading conditions ($p = 0.96$). Statistically significant differences between the shear bond strengths of the four Eclipse groups that were not treated with cyclic loading were found using a 1-way ANOVA ($p < 0.001$). Posthoc pairwise comparisons using Dunnett's T3 method assuming unequal variances indicated that bond strengths were significantly higher for group 2E than for group 1E ($p \leq 0.05$). Also, groups 3E and 4E demonstrated statistically significantly greater bond strengths than groups 1E or 2E ($p \leq 0.05$). The mean shear bond strengths of groups 3E and 4E were not statistically significantly different from each other ($p > 0.05$). A 2-way ANOVA compared the two loading conditions in groups 3E and 4E and demonstrated no statistically significant differences in shear bond strengths ($p = 0.74$). Additionally, a 3-way ANOVA compared denture base resin (Lucitone 199 or Eclipse), surface treatment (groups 3 and 4), and loading condition and demonstrated that the mean shear bond strengths were significantly higher with the Eclipse resin than the Lucitone 199 denture base ($p = 0.010$).

The quality of the bond was evaluated for all the specimens (Table 4). No adhesive failures were observed for groups 3L and 4L. The mode of failure for groups 1E and 2E was 100% adhesive, and it was mixed for groups 3E and 4E. All specimens prepared with the bonding agent demonstrated mixed failure within the base material or the tooth (Fig 4). In the specimens with adhesive failure, the Lucitone 199 material remained in the diatoric (Fig 5), while the Eclipse material pulled away from the diatoric during shear load testing in group 2E (Fig 6).

Discussion

The null hypothesis was not rejected for the Lucitone 199 groups with or without cyclic loading, as no statistical differences were demonstrated in shear bond strengths ($p = 0.71$). While several studies demonstrate an increase in the strength of the bond between denture teeth and PMMA denture bases with the use of a bonding agent,[7,29,30] the present investigation found the application of methyl methacrylate monomer on the ground surface of the denture tooth to be just as effective as using the acrylate bonding agent. In this investigation, the size and design of the diatoric was limited in an attempt to maintain a uniform bonding surface, and no significant effect of the diatoric on the bonding strength was noted. Previous publications[4,5] demonstrated an increase in the strength of the bond by adding larger retention grooves to the denture tooth ridge-lap. Future studies may evaluate variations in mechanical ridge-lap alterations in conjunction with the light-polymerized UDMA.

While using Eclipse set-up resin with the proprietary bonding agent, the present investigation showed statistically

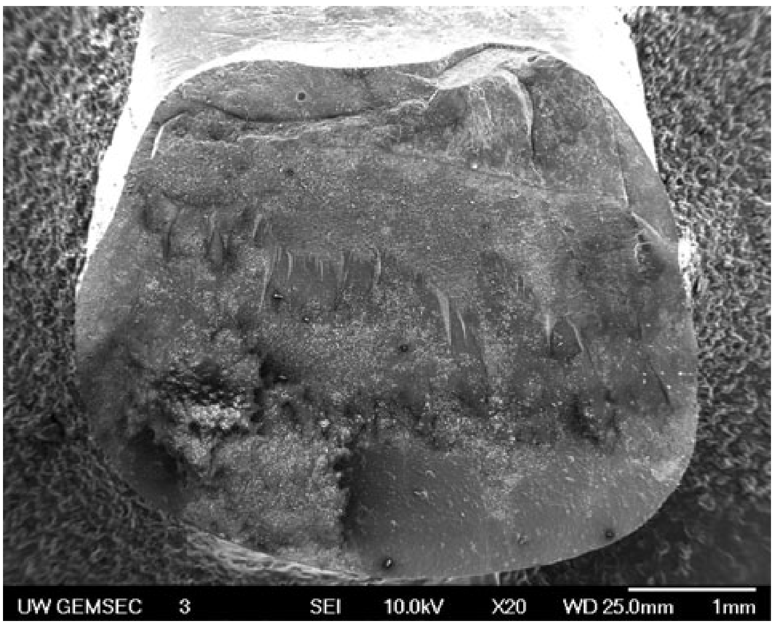


Figure 4 An example of a mixed failure demonstrated on the tooth surface of a broken Eclipse resin specimen (SEM 20× magnification).

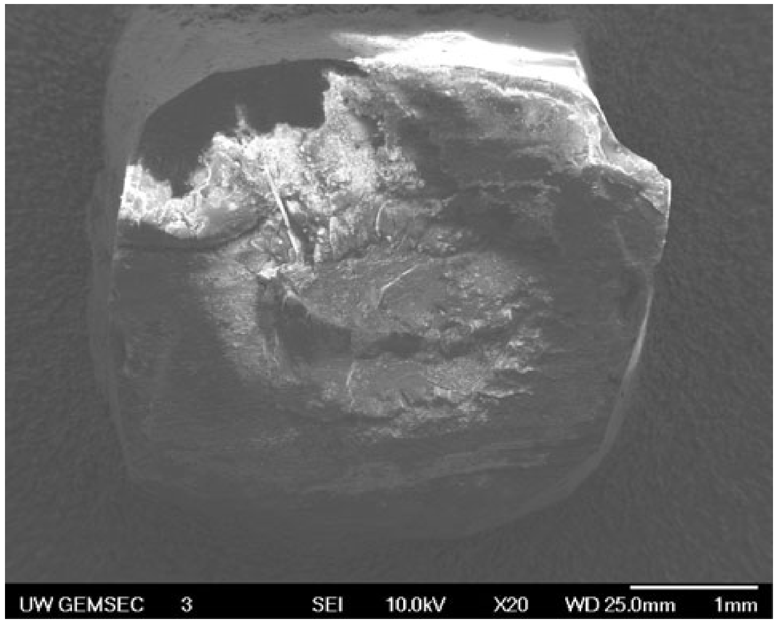
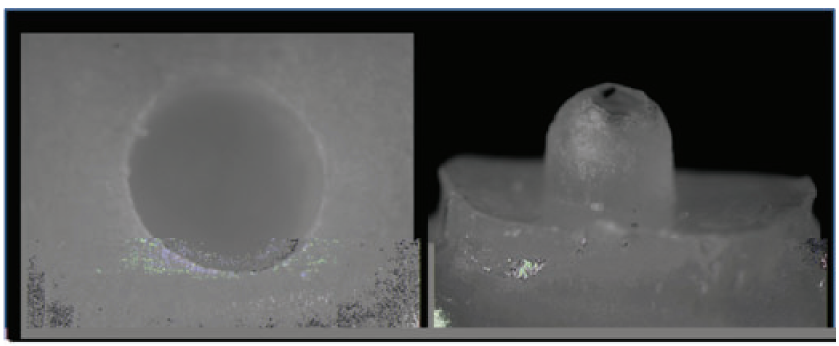


Figure 5 Example of tooth from a Lucitone specimen, the base material has remained inside the diatoric (SEM 20× magnification).

significantly greater shear bond strengths with light-polymerized UDMA than heat-polymerized PMMA ($p = 0.009$). Previous investigations demonstrated a decrease in bond strengths between denture teeth and visible light-polymerized resins compared to traditional PMMA denture base materials.[17-19] The improved shear bond strengths found in this study may be due to better compatibility between the bonding agent, the denture base, and the denture tooth. During processing of PMMA denture base resin, contamination with wax[31,32] and tinfoil substitute[33,34] are cited as the primary contributing factors for denture tooth debonding. The processing method of Eclipse specimens eliminates exposure of the teeth to these contaminants since the teeth are arranged with the set-up resin. A limitation of the present investigation was that the bond of the denture tooth to the Eclipse set-up resin was evaluated without the use of the contour resin. The highly cross-linked denture tooth was used, as it has been shown to decrease the bond strength in other studies[5,25,] and would provide a rigorous test. Further investigations could use additional types of denture teeth and evaluate the shear bond strength of the Eclipse contour resin to denture teeth.

The null hypothesis was rejected for the Eclipse resin specimens since a statistically significant difference was found in the shear bond strength between denture teeth and the Eclipse set-up resin in the four surface treatment groups. The Eclipse specimens without a bonding agent showed statistically lower mean shear bond strengths when compared to Eclipse specimens with a bonding agent ($p = 0.001$), and the mode of failure was completely adhesive without a bonding agent. These findings indicate that the Eclipse Bonding Agent should be used as a part of denture fabrication with the Eclipse Resin System. Other authors have concluded that a bonding agent should be applied when visible light-polymerized denture base resin is used with acrylic denture teeth.[19-21] The procedures for the Eclipse bonding agent are complicated, and the liquid is very volatile. Future research should be conducted to determine whether alternative bonding agents could be used to achieve comparable results. In the present investigation, the use of a diatoric without bonding



A B

Figure 6 (A): An example of the tooth surface with a diatoric in the Eclipse specimen without a bonding agent. Notice the clean adhesive failure (light microscope 30× magnification). (B) An example of the set-up resin on an Eclipse specimen without the bonding agent. Notice that the set-up resin has pulled away from the diatoric during the shear load testing (light microscope 20× magnification).

agent did statistically increase the mean shear bond strength of denture teeth to Eclipse without a bonding agent, but the mode of failure was still completely adhesive. In fact, the Eclipse resin pulled free of the diatoric space (Fig 6). Following ANSI Specification No. 15 guidelines, the failure of Eclipse specimens without a bonding treatment was unacceptable, since none of the specimens had at least a mixed failure.[35]

Thermocycling and cyclic loading were used to facilitate the simulation of the oral environment. The use of distilled water during these processes instead of artificial saliva is a limitation of this investigation. To simulate aging, all specimens were treated with 1000 thermocycles.[10] No studies have adequately shown a correlation of the number of thermocycles to months or years in the mouth;[36] however, some studies have shown a decrease in bond strength when 1000 cycles were used.[25] Future research should be conducted using thermocycling as a variable.

In the present investigation, cyclic loading (14,000 cycles at 1.5 Hz and 22 N) in the fatigue machine did not demonstrate a significant difference in shear bond strengths in the Lucitone 199 specimens ($p = 0.71$) or the Eclipse specimens ($p = 0.74$). Diaz-Arnold et al[23] found no significant difference in flexural strength in Eclipse resin with 10,000 cycles of cyclic loading. Some researchers have postulated that patients occlude their teeth 250,000 times per year.[37] Assuming these conditions, this investigation simulated approximately 3 weeks of function. In future studies, the effect of cyclic loading may be demonstrated by increasing the number of cycles. In addition, the load applied in cyclic loading was 22 N, which was determined from one half the mean shear bond strength of the Lucitone 199 ground surface specimens in a pilot investigation conducted by the authors. Furthermore, the angle of load in the fatigue machine and the force gauge was 135° to the long axis of the tooth[33,38] to simulate anterior occlusal loading forces.

Conclusions

Within the limitations of this investigation, the following conclusions can be made:

1. Heat-polymerized PMMA demonstrated no statistically significant differences in mean shear bond strength between the four surface treatment groups and loading conditions ($p = 0.71$). Thus, the use of a bonding agent did not enhance the shear bond strength of IPN denture teeth to the heat-polymerized PMMA. Also, the use of a monomer on the ground surface of the denture teeth was as effective as using the acrylate bonding agent.
2. Light-polymerized UDMA demonstrated statistically significant differences in mean shear bond strength between the four surface treatment groups without cyclic loading ($p < 0.001$). The specimens with a bonding agent demonstrated significantly higher bond strengths than the specimens without a bonding agent ($p < 0.05$). When no bonding agent was used, the specimens with a diatoric had significantly higher shear bond strengths than those without a diatoric ($p \leq 0.05$).
3. When a bonding agent was used, a comparison between the two denture base resins demonstrated significantly higher mean shear bond strengths with the light-polymerized UDMA ($p = 0.010$).
4. No statistically significant effects were noted from the cyclic loading treatment in the heat-polymerized PMMA ($p = 0.71$) or the light-polymerized UDMA ($p = 0.74$).
5. An evaluation of the mode of failure demonstrated adhesive debonding between the acrylic denture teeth and light-polymerized UDMA without a bonding agent. When a bonding agent was used with light-polymerized UDMA and the heat-polymerized PMMA denture base resins, mixed and cohesive failures were observed.

Acknowledgments

The authors would like to express their appreciation to Dentsply International for providing denture teeth, the Eclipse Prosthetic Resin System, and denture base resin. Correspondence with Dr. Stephen Wagner was helpful in understanding the clinical application of the Eclipse Resin System.

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