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Effect of Surface Preparation on the Failure Load of a Highly Filled Composite Resin Bonded to a Denture Base Resin

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Abstract

Purpose: The purpose of this study was to evaluate the effect of the surface preparation on the maximum fracture load value of a highly filled gingival shade composite resin bonded to a denture base resin.
Materials and Methods: Block specimens were prepared from a heat-processed denture base resin and divided into five groups. The flat surfaces of the specimens were abraded with 400-grit silicon carbide paper, then prepared in one of the following ways: (1) without preparation (group 1); (2) application of silane coupling agent (group 2); (3) application of dichloromethane (group 3); (4) application of dichloromethane following the silane coupling agent (group 4); or (5) tribochemical silica coating (group 5). A highly filled gingival shade composite resin was applied (area Ø = 5 mm) and polymerized with a light polymerizing unit. Specimens made entirely of heat-processed denture base resin were also fabricated as references (group 6). The halves of the specimens of groups 4, 5, and 6 were thermocycled up to 10,000 times in water between 5°C and 55°C with a 1-minute dwell time at each temperature. Shear testing was performed in a universal testing machine at a crosshead speed of 1 mm/min, and the maximum fracture load values were determined ($n = 10$).
Results: The maximum fracture load values of the highly filled gingival shade composite resin bonded to the denture base resin for all preparation groups were significantly enhanced before thermocycling ($p < 0.05$). Group 5 exhibited the greatest fracture load value, followed by group 4, compared to the other groups ($p < 0.05$), however, the fracture load values significantly decreased for these groups after thermocycling ($p < 0.05$), whereas the fracture load value of group 6 did not decrease ($p > 0.05$).
Conclusions: Tribochemical silica coating and the application of dichloromethane after the silane coupling agent were effective surface preparations for the bonding of a highly filled gingival shade composite resin to a denture base resin, however, the bond durability of these treatments may be insufficient.

Dental magnetic attachments have recently been used in removable prostheses.[1-5] When the yoke of a magnetic attachment is embedded in the denture base, autopolymerizing acrylic resin has generally been used with the brush-dip technique,[2] chiefly because of its easy handling. Because it is not always easy to achieve high bond strength of an autopolymerizing acrylic resin to a denture base resin, surface preparations for denture base resins using methyl methacrylate,[6,7] dichloromethane,[8-11] and ethyl acetate[12,13] have been studied; however, the bond strength provided by the surface treatments was always lower than that of the intact denture base resin. The chemical bonding between the denture base resin and the autopolymerizing acrylic resin still may be insufficient, resulting in the potential for microleakage and bond failure at the resin/resin interface. Furthermore, there is porosity within the matrix and cracks between the matrix and poly(methyl methacrylate) (PMMA) particles in the autopolymerizing acrylic resin.[14,15] Consequently, long-term reliability based on the criterion of sufficient bond strength between these two resins and a sound autopolymerizing acrylic resin has not yet been achieved.

In recent years, a new highly filled gingival shade composite resin, specially designed to provide the shades seen in natural gingiva, has been commercialized. This material was originally indicated for implant superstructures, crowns, and fixed and removable partial dentures (RPDs). It was assumed that highly filled composite resin bonded to a denture base resin by means of an appropriate surface preparation could be used as a substitute for an autopolymerizing acrylic resin. The purpose of this study was to evaluate the effect of surface preparation on the bonding of a highly filled gingival shade composite resin to a denture base resin.

Materials and methods

A total of 70 block specimens ($10 \times 10 \times 3.0$ mm^3) of heat-processed denture base resin (Acron clear, Powder 0702281, Liquid 0704171, GC Corp., Tokyo, Japan) were prepared in accordance with the manufacturer's instructions. The PMMA powder (24 g) and liquid (12 ml) were mixed, and the dough was allowed to reach packing plasticity after approximately 15 minutes at room temperature. A conventional laboratory procedure was used to mix and pack the resin in stone molds for denture processing. After processing, each block was embedded in an autopolymerizing resin with an acryl ring, and the surfaces of the denture base resin were abraded under running water with up to 400-grit silicon carbide paper. The abraded flat surfaces of the specimens were prepared in one of the five ways (Table 1). In groups 3 and 4, the preparation duration of dichloromethane (Wako Pure Chemical Industries Ltd., KLR7743, Osaka, Japan) was 5 seconds.[11] In groups 2 and 4, the surfaces of the specimens coated with the silane coupling agent (Clearfil ceramic primer, 0001AA, Kuraray Medical Inc., Tokyo, Japan) were kept wet, and the subsequent procedure was done as soon as possible. In group 5, the tribochemical silica coating was performed using the Rocatec system (3M ESPE AG, Dental Products, Seefeld, Germany) with 110-μm grain-sized alumina (Rocatec Pre, 3M ESPE AG, Dental Products) coated with silicon dioxide (Rocatec Plus, 3M ESPE AG, Dental Products) and a silane coupling agent (Rocatec ESPE-Sil, 252565, 3M ESPE AG, Dental Products) according to the manufacturer's specifications.

Sticky masking tape with a 6.0 mm diameter hole was placed on the bonding surface of each specimen, and a Teflon ring with a circular hole (5.0 mm inner diameter, 6.0 mm outer diameter) was placed in the hole on the masking tape to hold it in place and define the bonding area. A highly filled gingival shade composite resin (Gradia Gum, 704261, GC Corp.) was applied inside the Teflon ring and then polymerized with a light polymerizing unit (UniXS II, Heraeus Kulzer GmbH, Wehrheim, Germany) for 3 minutes. After the polymerization process was completed, the masking tape and Teflon ring were removed as gently as possible. Twenty test specimens made entirely with the heat-processed denture base resin were also fabricated as references for a one-piece block and cylinder using a stone mold, specially made for the current experiment (group 6).

Table 1 Surface preparations

Group	Preparation
1	Without preparation
2	Application of silane coupling agent
3	Application of dichloromethane
4	Application of dichloromethane following application of silane coupling agent
5	Tribochemical silica coating
6	Made entirely with the heat-processed denture base resin as a control

All specimens were immersed in distilled water at 37°C for 24 hours. Sixty specimens (six sets of ten specimens) were tested for a 24-hour maximum fracture load without thermocycling. Ten specimens each from groups 4, 5, and 6 (three sets of ten specimens) were placed in a thermocycling apparatus (Thermal Shock Tester TTS 1, Thomas Kagaku Co. Ltd., Tokyo, Japan) and cycled in water between 5°C and 55°C with a dwell time of 1 minute at each temperature for 10,000 cycles.

Shear testing was performed in a universal testing machine (Autograph AGS-J, Shimadzu Corp., Kyoto, Japan) to determine the maximum fracture loads at a crosshead speed of 1 mm/min. The means and standard deviations (SD) for the maximum fracture loads ($n = 10$) were calculated separately and statistically analyzed with one-way ANOVA both before and after thermocycling. After one-way ANOVA, the Newman-Keuls post-hoc comparisons test was performed at a significance level of $\alpha = 0.05$. In groups 4 to 6, the respective mean post-thermocycled fracture loads were compared to the pre-thermocycled fracture loads using Student's *t* test. All statistical analyses were performed using analytical software (STATISTICA Standard 03J, StatSoft Inc., Tulsa, OK).

The type of bond failure was determined after shear testing when the fracture surfaces of the specimens were examined using an optical microscope (Nikon 92052, Nikon Corp., Tokyo, Japan) at 30× magnification. Failure was evaluated in this study as A (adhesive failure at the heat-processed denture base resin/highly filled composite resin interface), C (cohesive failure within the heat-processed denture base resin without interface separation), or M (mixture of cohesive failure and adhesive failure).

Results

One-way ANOVA and Newman-Keuls post-hoc comparisons tests revealed that there were significant differences in maximum fracture load due to preparation both before and after thermocycling ($p < 0.05$). Before thermocycling, the mean fracture load of group 5 (tribochemical silica coating) was the greatest, followed by group 4 (dichloromethane after the silane coupling agent) ($p < 0.05$). Group 1 (without preparation) yielded the lowest fracture load ($p < 0.05$). After thermocycling, group 5 (tribochemical silica coating) exhibited greater mean fracture load than group 4 (dichloromethane after the silane coupling agent) ($p < 0.05$). Student's *t* test indicated that the mean fracture loads in groups 4 and 5 were significantly reduced by thermocycling ($p < 0.05$), whereas there was no significant difference between the pre- and post-thermocycled fracture loads

Table 2 Fracture load values (FLV) and failure modes of gingival shade composite bonded to denture base resin

Thermocycles Group	0 cycles			10,000 cycles		
	FLV (N) Mean ± SD	Significance	Failure type (n) C/M/A	FLV (N) Mean ± SD	Significance	Failure type (n) C/M/A
1	48 ± 42	a	0/0/10			
2	155 ± 45	b	0/0/10			
3	216 ± 79	c	0/0/10			
4	361 ± 26	d	0/5/5	261 ± 59	a	0/2/8
5	424 ± 27	e	0/8/2	339 ± 44	b	0/6/4
6	657 ± 55	f	10/0/0	594 ± 53	c	10/0/0

Different letters in a column indicate significant difference (one-way ANOVA, $p < 0.05$).
SD = standard deviation; C = cohesive failure; A = adhesive failure at the denture base resin/highly filled composite resin interface; M = mixture of cohesive failure and adhesive failure.

in group 6 (made entirely with the heat-processed denture base resin as a control) ($p > 0.05$). Table 2 summarizes the means and standard deviations of the fracture loads (N) before and after thermocycling, statistical significance, and numbers of cohesive, mixed, and adhesive failures. No cohesive failure occurred in this study except in group 6.

Discussion

It is well known that it is difficult to bond layers of a light-activated composite material together and that the use of an unfilled resin bonding agent after the application of a silane primer is an effective method of ensuring adequate bonding.[16] The finding of this study that the failure load for specimens without preparation was exceedingly low indicated that a highly filled gingival shade composite resin seldom bonds effectively to a denture base resin. Although the manufacturer has never officially announced its exact composition, the poor bonding capability of this material may be the low amount of the resin component. Another cause may be the small amount of residual monomer on the surface of the denture base resin. Some autopolymerizing acrylic reline resins that meet water sorption and solubility requirements demonstrated significantly lower bond strength to denture base acrylic resin.[17]

The failure loads for four of the surface preparations increased. Considering that adhesive failure exclusively occurred in all specimens in groups 2 and 3, the most effective preparations in this study may be the tribochemical silica coating (group 5) and second the application of dichloromethane following application of a silane coupling agent (group 4). Some investigators have evaluated surface preparations, including Rocatec, on removable dentures,[18-20] but they used it on RPD alloys, and their main purpose was to enhance the retention of the denture base resin to the metal framework. It appears that this study is the first to use the tribochemical silica coating using the Rocatec system as a surface preparation for denture base resin. Dichloromethane swells the surface and permits the diffusion of the denture base resin and creates a new surface texture with many pores.[11] Silanes are widely used to bond organic materials to inorganic materials. The combined use of dichloromethane and a silane coupling agent may have the synergistic effect of morphologically changing the denture base resin and coupling the highly filled gingival shade composite resin to the filler particles; however, the coefficients of the highly filled gingival shade composite resin to a denture base resin, even in group 5, are 64.5% for pre-thermocycling and 57.1% for post-thermocycling. Those in group 4 are 54.9% for pre-thermocycling and 43.9% for post-thermocycling. Furthermore, the failure load values decreased for the thermocycled specimens, including those in group 5. Therefore, the bond durability may be questionable even now. The mode of failure clearly supported the results of the fracture load values.

Through the use of these preparations, a further clinical application of a highly filled gingival shade composite resin will be the individualized coloring of the denture base to match the patient's own gingiva by adding it to the external denture surface made of PMMA denture base resin. Based on this study, it is not known whether tribochemical silica coating or the application of dichloromethane after the silane coupling agent are appropriate for enhancing the bonding of composite-based multilithic denture teeth to PMMA denture base resin. This study indicated that such preparations are effective only for application before the polymerization process of composite resin is completed. A further in vitro study and a long-term clinical follow-up are needed to evaluate the effect of the preparation methods, and new effective methods are strongly desirable.

Conclusions

Within the limitations of the current in vitro study, tribochemical silica coating and the application of dichloromethane after the silane coupling agent were effective surface preparations for the bonding of a highly filled gingival shade composite resin to a denture base resin, however, the bond durability of these treatments may be insufficient.

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