

Effect of surface preparations on the repair strength of denture base resin

Hiroshi Shimizu, DDS, PhD,^a Kenneth S. Kurtz, DDS,^b Masaharu Yoshinaga, DDS,^a Yutaka Takahashi, DDS, PhD,^a and Tetsuya Habu, DDS, PhD^a

^aDivision of Removable Prosthodontics, Department of Oral Rehabilitation, Fukuoka Dental College, Fukuoka, Japan, and ^bMontefiore Medical Center/Albert Einstein College of Medicine, Bronx, NY, USA

Purpose: The objective of this study was to evaluate the effect of surface preparations on the repair bond strength of an acrylic denture base resin material.

Materials and Methods: Rectangular specimens (n = 80) were prepared with a heat processed denture base resin (Lucitone 199) and divided into five groups. Four sets of 16 specimens were cut in half, and the remaining one set was not cut and served as the reference group. The surface preparations for the cut specimens were; 1) without preparation, 2) application of dichloromethane, 3) air-abrasion with 50 µm alumina, and 4) alumina air-abrasion followed by application of dichloromethane. These four groups were repaired with an autopolymerizing acrylic repair resin (Repair Material). The specimens were immersed in 37°C water for either one-day or 30 days. Transverse strengths were determined with a three-point bending test.

Results: Repair bond strengths in MPa of the denture base material after one-day and 30 days were; 73.0 and 52.0 for group 1, 74.8 and 57.3 for group 2, 76.7 and 54.7 for group 3, and 76.3 and 64.9 for group 4, whereas transverse strengths of uncut group were 85.5 and 85.0. Of the four surface preparations assessed, group 4 showed the greatest 30-day bond strength, although reduction in bond strength after aging was remarkable and the value was inferior to that of uncut group (p<0.05).

Conclusion: Within the limitation of the current *in vitro* study, air-abrasion with 50 µm alumina followed by the application of dichloromethane was the most effective surface preparation for the denture base resin. (Int Chin J Dent 2002; 2: 126-133.)

Clinical Significance: Alumina air-abrasion combined with application of dichloromethane facilitates bond strength to a heat processed denture base resin material when the fracture site is repaired with an autopolymerizing acrylic repair resin.

Key words: acrylic resin, alumina, denture base, dichloromethane, repair.

INTRODUCTION

Acrylic resin currently is the most dominant denture base material utilized in clinical prosthodontic

practice.¹ Acrylic denture base fracture is an infrequent, yet sometimes unavoidable complication of denture care. Repair of this fracture must be easily performed and have adequate repair strength. Autopolymerizing acrylic repair resin is often utilized as a repair material because of its easy handling and rapid polymerization properties. However, high bond strength between heat processed denture base resin and autopolymerizing acrylic repair resin is not always predictable.^{2,3} To gain optimum strength for repairs, it is essential that a good bond exists between these two types of acrylic resin.

The fundamental difference between the heat processed denture base resin and autopolymerizing acrylic resin is the method by which benzoyl peroxide is decomposed to yield free radicals. Generally, the conversion achieved using chemically autopolymerizing acrylic repair resin is not as complete as that achieved using heat activated systems. Chemically activated resin exhibits physical properties that are somewhat inferior to those of heat processed denture base resin⁴ and displays lower transverse strength.^{1,5}

To improve bond strength, several researchers have reported preparations to improve adhesion between broken denture base segments or for attachment of denture base resin to denture teeth. These include; 1) application of a solution of equal volumetric parts of dichloromethane (methylene chloride) and autopolymerizing (cold-curing) monomer,⁶ 2) application of chloroform,⁷ 3) application of a 1:1 mixture of dichloromethane and heat-polymerization monomer,⁸ 4) application of autopolymerizing monomer,⁹ 5) application of bonding agent of visible light-cured resin and heat-polymerization monomer,¹⁰ and 6) application of dichloromethane.¹¹ Limited information, however, is available about combined effect of mechanical and chemical retentions for the denture base repair. The purpose of the present study was to examine the transverse strengths of repaired denture base resin specimens, in which the repair surfaces had been modified using three surface preparations. Another purpose of this study was to investigate the durability of the bond of the repaired specimens after water storage.

MATERIALS AND METHODS

A heat processed denture base resin (Lucitone 199, Dentsply International Inc., York, PA, USA) and an autopolymerizing acrylic repair resin (Repair Material, Dentsply International Inc.) were selected for the study (Table 1). A total of 80 rectangular specimens of the heat processed denture base resin, 3.0 x 10.5 x 68.5 mm in dimension, were prepared according to the manufacturer's instructions. The poly(methyl methacrylate) (PMMA) powder (21 g) and the liquid (10 mL) were mixed for 15 s. The dough time to reach packing plasticity was 9 minutes. Procedures for mixing and packing the resin in stone molds followed conventional laboratory procedure for denture processing, as powder/liquid ratio can contribute to distortion.¹² After processing, the specimens were abraded under running water with silicone carbide paper to 600 grit, to a final dimension of 2.5 x 10.0 x 68.0 mm, after which the specimens were stored in 37°C distilled water for 30 days. The intact 16 specimens were used as references, and the remaining 64 specimens were cut in half with a band saw under running water guided by a standardized positional jig. The resulting rectangular shape would not contribute mechanical advantage to the repair site,¹³ and was easiest to duplicate. Cut surfaces were made parallel to each other and perpendicular to the long axis of the

specimens by abrading under water with 100 grit silicone carbide paper to simulate roughening of the repair surface of the denture with laboratory burs.

Table 1. Materials used.

Materials	Manufacturer	Batch number
<u>Denture base resin</u>		
Lucitone 199	Dentsply International Inc., York, PA, USA	Powder 991129, Liquid 9911014
<u>Denture repair resin</u>		
Repair Material	Dentsply International Inc.	Powder 000118, Liquid 000404

The abraded surfaces were divided into four groups; 1) untreated, 2) application of dichloromethane with preparation duration of 5 s,¹⁴ 3) 50 μm alumina air-abrasion at a right angle to the surface from 5 mm distance for 10 s at an emission pressure of 0.48 MPa using a grit blaster (Microblaster, Comco Inc., Burbank, CA, USA), and 4) 50 μm alumina air-abrasion followed by the application of dichloromethane with preparation duration of 5 s. All specimens were air-blasted to remove volatile component of dichloromethane or excess alumina particles on the test surfaces.

The parallel halves of the specimens were placed in open-ended stone molds of the same dimensions as the original intact specimens for the transverse strength test. The open-ended stone molds were used so that the ends to be repaired could be moved apart to leave a 3 mm rectangular butt joint gap. The repair surfaces were wetted with the MMA monomer liquid of the repair material before autopolymerizing acrylic repair resin was applied to the joint space. The duration of MMA wetting for the repair was 180 s.⁹ This gap was then filled with the autopolymerizing acrylic repair resin. The mixing ratio (powder to liquid) was 2:1 (w/w) and the free-flowing mix was poured into the joint space to allow for a slight excess to ensure a complete joint. After the polymerization process was completed at 23°C to mimic the most basic clinical circumstances, the repaired specimens were then trimmed to their original dimensions with 600 grit silicone carbide paper, eliminating the unpolymerizing surface layer.¹⁵ Half of the specimens were kept in distilled water at 37°C for one-day before testing was performed. The remainder of the specimens was kept in distilled water at 37°C for 30 days to evaluate the influence of aging in water on bond strength. This would simulate immediate and aged clinical repair circumstances.

A three-point bending test was performed with the universal testing machine (TCM-200, Minebea Co. Ltd., Tokyo, Japan). The distance of 60 mm between the supports and a crosshead speed of 10 mm/minute was used during loading. The load at fracture was converted to transverse strength (S) by the formula $S = 3PL/2bd^2$, where P is the breaking load, L is the length between the jig supports (60 mm), b is the width of the specimen and d is the thickness of the specimen. All tests were performed under uniform atmospheric

conditions of 23°C and 50% relative humidity. The data were analyzed statistically using two-way analysis of variance (ANOVA). The variables were surface preparation and the period of water immersion. Newman-Keuls *post-hoc* comparison was applied when appropriate (95% confidence level).

To determine the morphologic changes to the surface after preparations, a few extra specimens were prepared according to the original protocol. They were sputtered with gold, and observed with a scanning electron microscope (SEM, JSM-T 330, JEOL Inc., Tokyo, Japan) operated at 5 kV. For all specimens, the interface where failure occurred was observed through an optical microscope (92052, Nikon, Tokyo, Japan) at x30 magnification.

RESULTS

Two-way ANOVA and Newman-Keuls *post-hoc* comparison revealed that there were significant differences in transverse strength due to the variables surface preparation and period of water immersion, whereas there were no significant differences in their interaction ($p < 0.05$). There were no significant differences in transverse strength due to the surface preparations after one-day water immersion (73.0 to 76.7 MPa) whereas after 30 days water immersion, 50 μm alumina air-abrasion followed by dichloromethane application showed higher strength (64.9 MPa) than the other methods and the control (52.0 to 57.3 MPa). The transverse strengths of the original intact specimens of the Lucitone 199 material, which was not included in the statistical analysis, were 85.5 MPa for one-day water immersion and 85.0 MPa for 30 days water immersion. Contrast evaluation revealed that there was no significant difference between these strengths. The average strengths, standard deviations, statistical categories, and the percentages (%) of the transverse strengths to that of the original intact specimens are summarized in Table 2. Modes of failure of all specimens except for the original intact specimens are presented in Table 3.

Table 2. Transverse strengths of the repaired denture base material.

Surface preparation	1 day immersion			30 days immersion		
	Mean	SD	Category	Mean	SD	Category
Untreated	73.0	2.9	a (85)	52.0	10.2	c (61)
Dichloromethane	74.8	4.8	a (88)	57.3	8.6	c (67)
Alumina air-abrasion	76.7	1.5	a (90)	54.7	8.7	c (64)
Alumina air-abrasion + dichloromethane	76.3	3.8	a (89)	64.9	7.3	b (76)
Uncut intact specimen	85.5	0.9	- (100)	85.0	3.5	- (100)

SD: standard deviation. Identical letters indicate that the values are not statistically different ($p > 0.05$).

Numbers in parentheses show the percentage of the strength to that of the corresponding intact specimen.

Fig. 1 depicts SEM views after surface preparations. SEM view of denture base resin surface by abrading with 100 grit silicone carbide paper showed lots of well-regulated scratches in one way direction.

Table 3. Mode of bond failure.

Surface preparation	1 day immersion	30 days immersion
Untreated	CCCCCCCC	MMAAAAAA
Dichloromethane	CCCCCCCA	CCMAAAAA
Alumina air-abrasion	CCCCCCCA	CCMAAAAA
Alumina air-abrasion + dichloromethane	CCCCCCCA	CCMMMMMA

A: Adhesive failure at the denture base resin-repair resin interface; C: Cohesive failure within the repair resin; M: Mixture of cohesive failure and adhesive failure. Each letter code corresponds to a separate specimen.

SEM view of denture base resin treated with alumina air-abrasion exhibited rougher surface compared with that by abrading with silicone carbide paper. SEM views of denture base resin treated with alumina air-abrasion followed by dichloromethane and that with dichloromethane showed an almost homogenous surface void of irregularities with pores and channels of less than 1 µm in diameter. One thing to be noticed was that the surface treated with alumina followed by dichloromethane looked a little bit uneven.

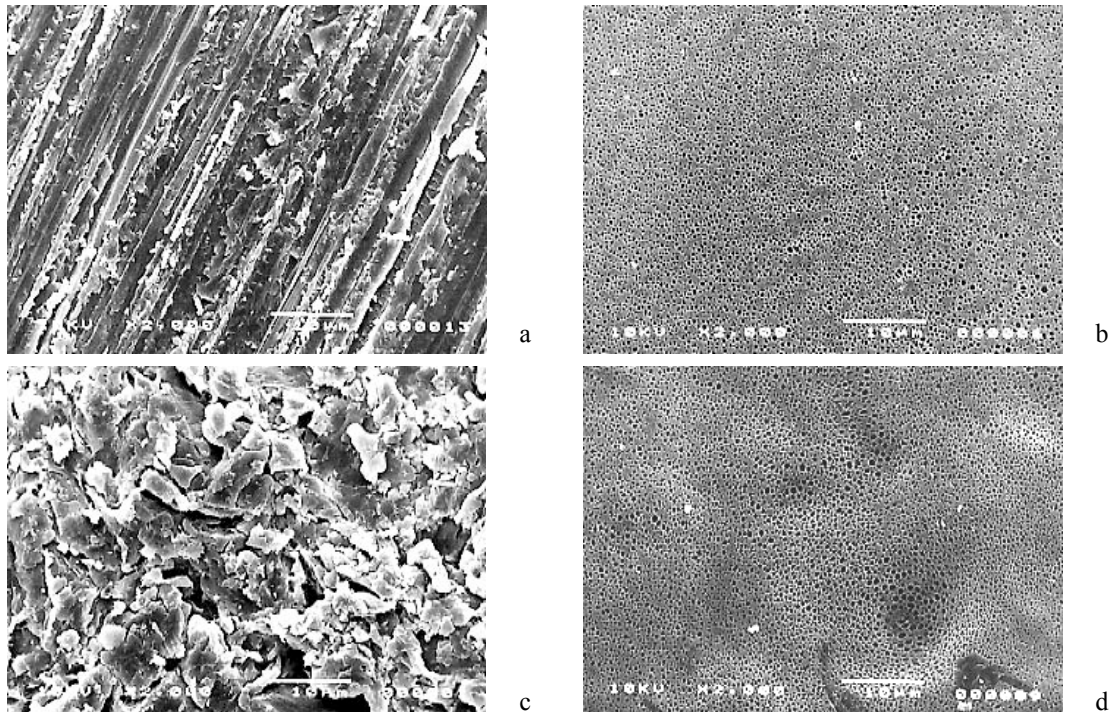


Fig. 1. Scanning electron micrographs of the denture base material (a: ground; b: ground + dichloromethane; c: alumina-blasted; d: alumina-blasted + dichloromethane).

DISCUSSION

Della Bona and van Noort¹⁶ described that the tensile strength test is more appropriate than shear bond strength test for evaluating the adhesive capabilities of resin composite to ceramics, because that shear bond strength test has as its inherent feature the measurement of the strength of the base material rather than the strength of the adhesive interface. This fact can be applied to the combination of a denture base resin and an autopolymerizing acrylic resin.¹⁷ The tensile strength test, however, needs exactly accurate load direction to the specimen and this factor, which is too difficult to control, considerably affects the results.¹⁸ The validity of three-point bending test was found to have close correlation with the failure mode of specimens.¹⁸ Therefore the three-point bending test was performed for the current study.

Tables 2 revealed that a heat processed denture base resin had a higher transverse strength to an autopolymerizing acrylic repair resin after one-day water immersion than that after 30 days water immersion and none of the surface preparations significantly improved the transverse strength of the repair specimens after one-day water immersion. A comparison in failure mode of all specimens after one-day water immersion did not show much difference. Cohesive failure of the repair material indicates that a sufficient bonding to the repair surface has been achieved.⁹ After 30 days water immersion, however, the transverse strengths of all groups significantly decreased and the highest transverse strengths corresponded to 50 μm alumina air-abrasion followed by dichloromethane application. The result demonstrates that the transverse strength between a heat processed denture base resin and an autopolymerizing acrylic repair resin have a tendency to decrease with the period of immersion in water. Surface preparation of 50 μm alumina air-abrasion followed by dichloromethane reduces the decline of the transverse strength between two types of resin.

Dichloromethane, is an organic and nonpolymerizable solvent, which swells the surface and permits a diffusion of the polymerizable material.⁶ The strength of the bond depends upon the degree of penetration of the solvent and the strength of the interwoven polymer network formed thereafter.⁶ Dichloromethane preparation can create surface pores and channels approximately 1 μm in diameter on a conventional acrylic resin tooth, and these channels tend to interconnect frequently.¹¹ This morphological change also occurs when dichloromethane is applied to heat processed denture base resin (Fig. 1). Prepolymerizing PMMA pearls present in the denture base resin should allow diffusion of the dichloromethane solvent. Vallittu previously described diffusion of MMA solvent into the matrix and interpenetrating polymer network of dental polymer.¹⁹ Since the prepolymerizing PMMA pearl are not crosslinked with the matrix, application of solvent can create crosslinking.

Alumina air-abrasion is utilized to roughen the repair surface of the denture base (Fig. 1) to increase the area of bond contributing to micromechanical retention and eliminating substances which adhere to the surface of the repair region, mechanically facilitating application of solvent or MMA monomer. The SEM view of denture base resin surface treated with 50 μm alumina air-abrasion followed by dichloromethane (Fig. 1) nearly looks like that of treated with dichloromethane. The base surface treated with dichloromethane is almost flat and smooth, whereas that treated with 50 μm alumina air-abrasion followed

by dichloromethane is a little bit uneven. This suggests that the preparation with 50 μm alumina air-abrasion followed by dichloromethane has a synergistic effect of the morphologic changes by means of dichloromethane and increasing of the surface area due to air-abrasion. However, the independent effect of dichloromethane and 50 μm alumina air-abrasion in improving the bond between a heat processed denture base resin and an autopolymerizing acrylic repair resin is not completely clear from the results of the current study. Remnants of the dichloromethane solvent may have a deleterious effect on bond strength.

This study concludes that the surface preparation of 50 μm alumina air-abrasion followed by the application of dichloromethane yields the highest bond strength after 30 days immersion in water among the three preparations in this study. This finding reveals a synergistic effect of dichloromethane and 50 μm alumina air-abrasion. This is explained in part by understanding the eliminative action of alumina air-abrasion which prepares the surface of the denture base resin for a roughened structure to apply dichloromethane. Furthermore, an analysis of failure mode demonstrated only one adhesive failure in this experimental group.

CONCLUSIONS

Alumina air-abrasion followed by application of dichloromethane facilitates bond strength to a heat processed denture base resin material when the fracture site is repaired with an autopolymerizing acrylic repair resin. Although reduction after aging is remarkable, this protocol may be considered for clinical use on repair sites of fractured acrylic resin denture bases.

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Reprint request to:

Dr. Hiroshi Shimizu

Division of Removable Prosthodontics, Department of Oral Rehabilitation, Fukuoka Dental College
2-15-1, Tamura Sawara-ku, Fukuoka 814-0193, Japan

FAX: +81-92-801-0513

E-mail: simizuhl@college.fdcnet.ac.jp

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