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Shear Bond Strength of Two Chemically Different Denture Base Polymers to Reline Materials

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Keywords
Shear bond; urethane dimethacrylate; methyl methacrylate; reline; light polymerization.

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This study was supported by Vote F funding, University of Malaya, Kuala Lumpur, Malaysia.

Previously presented at the 21st International Association of Dental Research, Southeast Asia Division & Exhibition, Bali, Indonesia.

Accepted August 26, 2008

doi: 10.1111/j.1532-849X.2009.00481.x

Abstract

Purpose: This study evaluated the shear bond strengths of light-polymerized urethane dimethacrylate (Eclipse) and heat-polymerized polymethylmethacrylate (Meliodent) denture base polymers to intraoral and laboratory-processed reline materials.
Materials and Methods: Thirty disks measuring 15 mm diameter and 2 mm thick were prepared for each denture base material following the manufacturers' recommendation. They were relined with Meliodent RR, Kooliner, and Secure reline materials after 1 month of water immersion. Ten additional Eclipse specimens were relined using the same Eclipse resin. A shear bond test was carried out on an Instron machine at a crosshead speed of 1.0 mm/min 24 hours after relining. Data were analyzed using two-way and one-way ANOVAs and post hoc Dunnett's T3 test ($p = 0.05$). The nature of failure was analyzed under a stereomicroscope. The effect of dichloromethane adhesive on the two denture polymer surfaces and the failed interfaces of mixed and adhesive failures were analyzed under a SEM (scanning electron microscope).
Results: Two-way ANOVA showed significant differences in the shear bond strength values as a function of the denture base polymers, reline materials, and their interaction ($p < 0.05$). One-way ANOVA showed significant differences in shear bond strength values among denture base-reline combinations ($p < 0.05$) except for Meliodent-Kooliner and Eclipse-Meliodent RR relines. Meliodent showed the highest shear bond strength value when relined with Meliodent RR (14.5 ± 0.5 MPa), and Eclipse showed the highest value with Eclipse relining (11.4 ± 0.6 MPa). Meliodent denture base showed adhesive, cohesive, and mixed failure, while all Eclipse showed adhesive failure with various reline materials.
Conclusion: The two chemically different denture base polymers showed different shear bond strength values to corresponding reline materials.

The most commonly used denture base resin is heat-polymerized polymethyl methacrylate (PMMA). Visible light-polymerized urethane dimethacrylate (UDMA) was introduced in the 1980s, and Triad (Dentsply Int., York, PA) was the first light-polymerized denture base polymer available in the market. This system eliminates flasking, boiling out, packing, and the water-bath polymerization normally required in conventional denture processing. The latest addition to UDMA denture base polymer is known as Eclipse Prosthetic Resin System (Dentsply Int.) where three types of UDMA resins are supplied for the construction of the denture.[1,2] Studies show that Eclipse exhibits significantly higher surface hardness, flexural strength, and flexural modulus than PMMA denture base polymers.[1,3]

Regardless of the materials used for denture construction, the denture needs to be regularly modified as the alveolar ridge progressively resorbs. There are two main treatment options to manage the denture with poor adaptation to the oral tissue: either construct a new denture, or restore the fit of the existing denture by relining.

Reline is defined as the procedure used to resurface the tissue side of a denture; it produces an accurate foundation for the denture-bearing area.[4] This eliminates making a new denture, and some clinicians believe that this is more time saving and economical. Many hard autopolymerizing reline resins have been investigated for their properties,[5-7] including bonding to denture base polymers.[8-10] Bond strength has been shown to be dependent on the chemical compositions of both the reline materials and denture base polymers.[6,10-12] A weak bond has been associated with reduction in the mechanical strength of the relined denture, bacteria accumulation, and staining.[8,11]

The exact chemistry of Eclipse is not revealed by the manufacturer, and its ability to bond to reline materials is also not

Table 1 Denture base polymers and relining materials used

Brand name	Material type	Main composition	Processing method	Manufacturer	Batch number
Eclipse	Heat- and light-polymerized UDMA denture base polymer	Single paste; Matrix: UDMA; Filler: Silica and PMMA beads	Heat- and light-polymerized for 10 minutes (using Menu 1)	Dentsply Int., York, PA	Lot 030909
Meliodent	Heat-polymerized PMMA denture base polymer	Powder: PMMA; Liquid: MMA	Heat-polymerized at 70°C for 7 hours and 100°C for 1 hour	Heraeus Kulzer, Hanau, Germany	Powder: Lot A1397B-2; Liquid: Lot 012155
Meliodent RR	Laboratory, autopolymerized PMMA reline	Powder: PMMA; Liquid: MMA, EGDMA, EGPT	Autopolymerized at 55°C and 2 bars pressure for 10 minutes	Heraeus Kulzer, Hanau, Germany	Powder: Lot RB136B-14; Liquid: Lot 013029
Secure	Intraoral autopolymerized reline	Two paste; Base: HEMA; Catalyst: BP	Autopolymerized at 37°C for 10 minutes	Imtec Corp., Ardmore, OK	Lot 137872019
Kooliner	Intraoral autopolymerized reline	Powder: PEMA; Liquid: IBMA, DMPT	Autopolymerized at 37°C for 10 minutes	GC America, Alsip, IL	Lot 0406021
Eclipse	Heat- and light-polymerized UDMA denture base polymer	Single paste; Matrix: UDMA; Filler: silica and PMMA beads	Heat- and light-polymerized for 6 minutes (using Menu 4)	Dentsply Int., York, PA	Lot 030909
Secure adhesive	Bonding agent for Secure	DM	–	Imtec Corp., Ardmore, OK	Lot 137872019

UDMA = urethane dimethacrylate; PMMA = polymethyl methacrylate; MMA = methyl methacrylate; EGDMA = ethylene glycol dimethacrylate; EGPT = ethylene glycol dimethacrylate; EGPT = ethylene glycol *p*-toluidine; HEMA = hydroxyethyl methacrylate; BP = benzoyl peroxide; PEMA = polyethyl methacrylate; IBMA = iso-butyl methacrylate, dimethyl-*p*-toluidine; DM = dichloromethane.

well documented. It is therefore the purpose of this in vitro study to compare the shear bond strengths of UDMA (Eclipse) and PMMA (Meliodent) denture base polymers when they were relined with representatives of intraoral and laboratory reline materials.

Materials and methods

The brand names, manufacturer information, and processing methods of the materials used in this study are listed in Table 1.

Preparation of denture base specimen

Specimens of both Eclipse and Meliodent denture base polymers were prepared by investing brass columns measuring 15 mm in diameter and 4 mm in height in dental stone. A powder:liquid ratio of 100 g of stone to 30 ml of water was used to prepare gypsum molds. Eclipse baseplate resin and the stone mold were preheated in a conditioning oven (Dentsply Int.) to 55°C for 2 minutes to allow easier adaptation of the material. Separating agent (Dentsply Int.) was applied onto the mold, and the warmed resin was adapted into the mold using finger pressure. Air barrier coating (Dentsply Int.) was applied onto the exposed surface to prevent inhibition of polymerization by oxygen. Polymerization was carried out following the manufacturer's instruction in the light-curing unit (Dentsply Int.) where six halogen lamps of 41 V each were used. According to the manufacturer, a maximum temperature of 129°C will be reached during the polymerization. A total of 40 specimens were prepared. Thirty Meliodent denture base specimens were prepared using compression molding technique and polymerized in a water bath (Menfredi, Turino, Italy).

All Meliodent and Eclipse denture base specimens were left in water at 37°C for 30 days. They were then mounted in clear self-cure epoxy resin to allow them to be assembled on the testing machine with one of the specimen surfaces exposed for relining. The specimen surface was wet ground using a grinding and polishing machine (Metaserv® 2000, Buehler, Lake Bluff, IL) on 600-grit silicone carbide paper.

Relining procedure

A brass ring of 6 mm internal diameter and 2.5 mm height was placed at the center of the exposed surface to confine the reline materials. Prior to relining, the specimen surface was cleaned with alcohol and rinsed with distilled water. Surface moisture was removed with clean gauze, and the specimen was left to air dry for 30 seconds.

The relining procedure using each reline material was performed following the manufacturer's instruction (Table 1). As recommended by the manufacturer, two layers of dichloromethane adhesive were applied on the denture specimen surface when relined with Secure resin. No surface treatment on the denture surface was advocated by the manufacturer of Kooliner resin. With intraoral relining materials, polymerization was carried out at 37°C to simulate the temperature of the oral cavity. Relining using Eclipse material was carried out by finger adaptation of the softened material to the Eclipse denture base specimen within the brass ring. The manufacturer, however, does not recommend the PMMA denture base to be

relined using Eclipse material, as the temperature required for polymerization is too high for PMMA.

Shear bond strength test

All bonded specimens were stored in distilled water at 37°C for 24 hours as described in ISO specification 11405:2003[12] for short-term water storage. The shear bond test was carried out using an Instron Universal Testing machine (Instron Inc., High Wycombe, UK) at a crosshead speed of 1.0 mm/min. Compressive load was applied using a knife-edged blade, which was placed parallel to the material interface. The test was performed under uniform, dry atmospheric conditions at a temperature of 23°C:

$$F = \frac{N}{A},$$

where F is the shear bond strength (MPa), N is the maximum force exerted on the specimen (in Newtons), and A is the size of the bonding area (mm^2).

The debonded surface was inspected using a stereomicroscope (Kyowa SD-2PL, Tokyo, Japan) at a magnification of 10× to determine the amount of reline material left on the denture base. The nature of failure was categorized as cohesive (more than 50% trace of reline material on the denture base specimen or vice versa), adhesive (no traces of reline material on the denture base or vice versa), or mixed (less than 50% trace of reline material on the denture base or vice versa). One examiner was involved in the recording.

SEM evaluation

The surface of one specimen for each denture base polymer was evaluated under a scanning electron microscope (SEM) (Quanta 200, FEI, Hillsboro, OR) before and after dichloromethane adhesive application using low-vacuum imaging modes. Additionally, the failed interfaces of Meliodent base-Secure reline and Eclipse base-Eclipse reline specimens were also examined.

Statistical analysis

A two-way analysis was used to determine the effect of denture base polymer, reline materials, and their interaction. For this analysis, the value of Eclipse base-Eclipse reline specimen was excluded. One-way ANOVA and post hoc Dunnett T3 tests were used for comparison of shear bond strength values among seven denture base-reline polymer combinations.

Results

Two-way ANOVA showed significant differences in the shear bond strength values as a function of the denture base polymers, reline materials, and their interaction ($p < 0.05$). One-way ANOVA showed that there were significant differences in the shear bond strength values ($p < 0.05$). The mean shear bond strength values for the seven denture base-reline combinations are presented in Table 2. The shear bond strength values ranged from 2.4 ± 0.5 to 14.5 ± 0.5 MPa. Within PMMA denture base groups (Meliodent), the highest shear bond strength value was achieved when relined with Meliodent RR materials (14.5 ± 0.5 MPa), while in UDMA groups (Eclipse), the highest shear bond strength value was achieved with Eclipse reline (11.4 ± 0.6 MPa). The numbers of cohesive, mixed, and adhesive failures for various denture base-reline combinations are also shown in Table 2.

Table 2 Shear bond strength of PMMA and UDMA denture base polymers to various reline materials (mean values and SDs) and number of bonding failures

Denture base polymer	Reline material	Bond strength Mean ± SD (MPa)	Failure type (n) C/M/A
PMMA-Meliodent	Meliodent RR	14.5 ± 0.5[a]	10/0/0
	Secure	9.9 ± 1.0[c]	0/10/0
	Kooliner	4.5 ± 0.5[e]	0/0/10
	Eclipse*	NA	NA
UDMA-Eclipse	Meliodent RR	4.6 ± 0.7[e]	0/0/10
	Secure	8.1 ± 0.7[d]	0/0/10
	Kooliner	2.4 ± 0.5[f]	0/0/10
	Eclipse	11.4 ± 0.6[b]	0/0/10

*Not possible to reline Meliodent polymer with Eclipse.
n = 10.
One-way ANOVA within column showed a significant difference in shear bond strength of the denture base-reline polymer combinations ($p < 0.05$). Same letter indicates values that were not statistically different ($p > 0.05$).
C = cohesive failure; M = mixed failure; A = adhesive failure; SD = standard deviation.

Figures 1 and 2 are SEM views of Meliodent and Eclipse denture base surfaces before and after dichloromethane adhesive application, respectively. A SEM view at a lower magnification of 400× was used for the Meliodent specimen as compared to a 3000× magnification for Eclipse, because at higher magnification it was not possible to demonstrate the change in the surface pattern of the Meliodent specimen. At a 3000× magnification, the Meliodent specimen surface appeared homogenous, and no obvious scattered depression was portrayed.

Figure 3 is the SEM view of mixed failure at the Meliodent base-Secure reline bonding interface, and Figure 4 shows adhesive failure at the bonding interface of Eclipse base-Eclipse reline specimens after the shear bond test.

Discussion

In this study, the shear bond strengths of relined heat-polymerized PMMA were compared to those of a UDMA-based (Eclipse) denture base polymer. According to the manufacturer, Eclipse requires both heat and light for polymerization, and a high temperature of 129°C is required for complete conversion of the polymer. It was also stated that Eclipse can be relined using the same Eclipse material as well as with other conventional autopolymerized materials. So far, no study has reported on the bond strength of Eclipse as compared to many reports available with PMMA denture base polymer.[8,13,14-17] This study showed that for all reline materials excluding Eclipse reline, the shear bond strengths to PMMA were higher than the

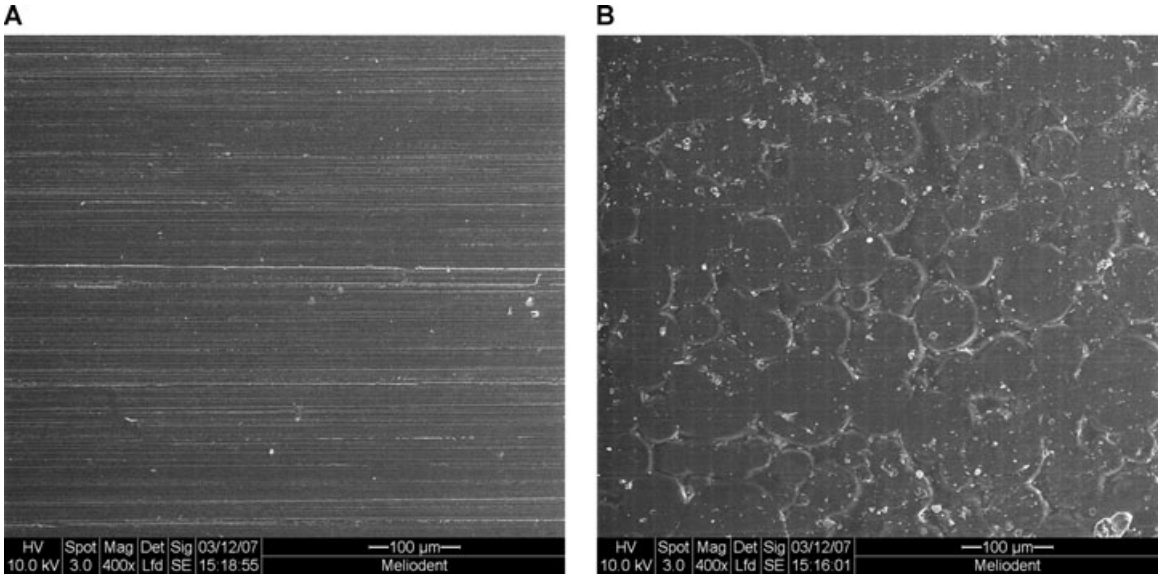

Figure 1 (A) SEM view of the Meliodent denture base surface before treatment with dichloromethane at 400× magnification. (B) SEM view of the Meliodent denture base surface after treatment with dichloromethane at 400× magnification.

strength of corresponding reline materials to UDMA polymer. The reline materials chosen in this study may not cover all products available in the market, but they are representative of intraoral and laboratory-processed reline materials.

The results of this study showed that higher bond strengths were obtained when reline materials chemically similar to the denture base were used for relining. A similar observation has been reported where Triad VLC reline material produced higher bond strength to Triad VLC than to PMMA denture base polymers.[18] Similarly for PMMA denture polymer, higher bond strength was reported with MMA-based resin as compared to non-MMA-based material,[14] suggesting that greater cross-linking occurred between similar base materials.

In this study, heat-polymerized PMMA relined with laboratory-processed autopolymerized PMMA reline material exhibited the highest bond strength (14.5 ± 0.5 MPa) among all denture base-reline combinations. Sarac et al[15] obtained a slightly higher shear bond strength value of 16.7 ± 0.5 MPa for PMMA and autopolymerized PMMA reline. The observation of 100% cohesive failures for this denture base and reline

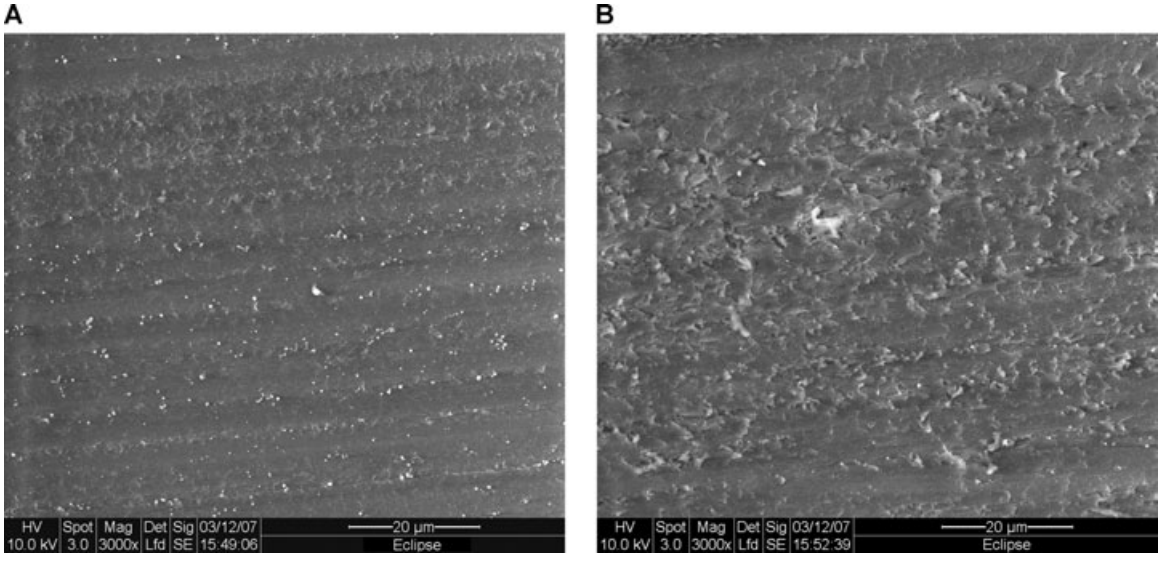

Figure 2 (A) SEM view of the Eclipse denture base surface before treatment with dichloromethane at 3000× magnification. (B) SEM view of the Eclipse denture base surface after treatment with dichloromethane at 3000× magnification.

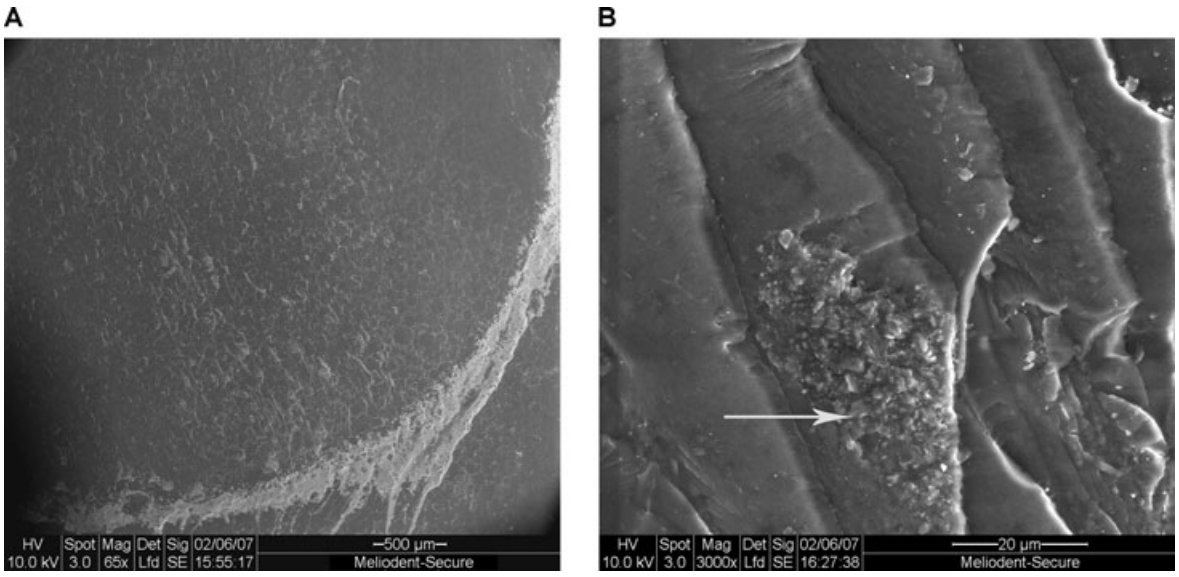

Figure 3 (A) SEM view at the bonding interface of Meliodent base-Secure reline with mixed failure at 65× magnification. (B) SEM view at the bonding interface of Meliodent base-Secure reline at a higher magnification of 3000× showing remnants of the Secure material attached to the Meliodent base surface.

combination in this study may also explain the improved bonding between them.

A comparison between the two intraoral reline materials (Secure and Kooliner) showed higher reline bond strength when the denture base was relined with the former. The use of dichloromethane adhesive prior to relining with Secure may explain the observation. The SEM view of PMMA denture base surface treated with dichloromethane revealed a smooth surface with scattered depressions (Fig 1). The effect of dichloromethane was to dissolve the surface layer, enlarge the surface area for bonding, and improve mechanical retention.[6,19,20] The debonded surface as observed under a SEM (Fig 3) showed Secure material remnants attached to PMMA denture base, indicating mixed cohesive and adhesive failure as opposed to Kooliner where the failure mode was of adhesive nature. Adhesive failure of Kooloner reline to PMMA denture base polymer observed in this study is in agreement with some previous studies.[16,21,22] As adhesion depends on the penetration of polymerizable monomer of reline material into the denture network,[6] higher molecular weight of IBMA (isobutyl

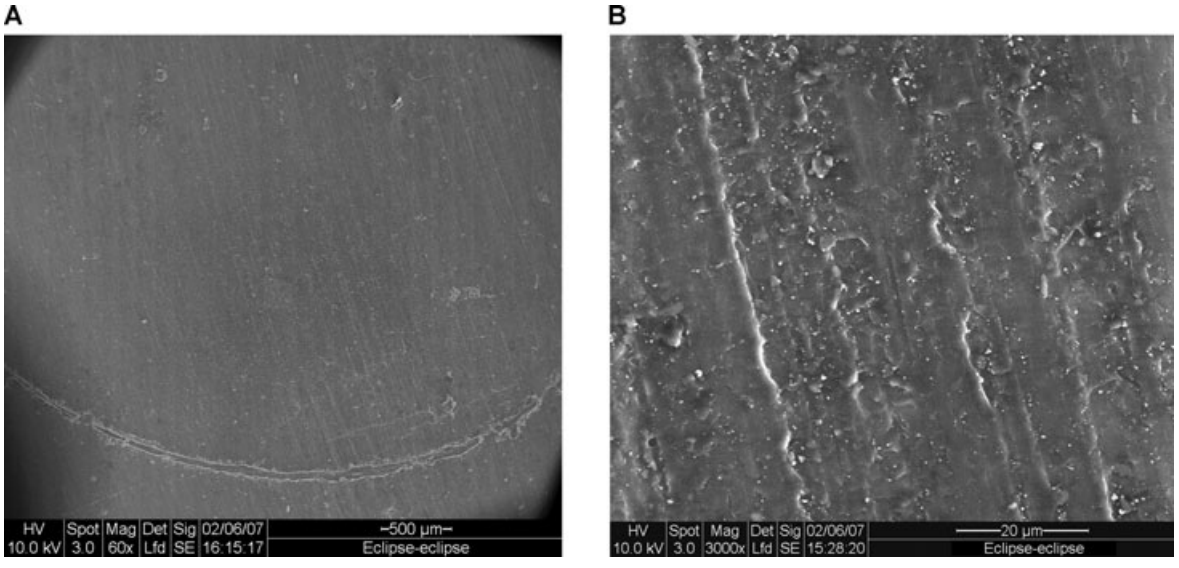

Figure 4 (A) SEM view at the bonding interface of Eclipse base-Eclipse reline showing adhesive failure at 60× magnification. (B) SEM view at the bonding interface of Eclipse base-Eclipse reline at a higher magnification of 3000×.

methacrylate) monomer available in Kooliner might have limited the monomer penetration.[6,9,14] In contrast, Neppelenbroek et al[17] found that Kooliner reline showed mixed modes of failure to PMMA base. The denture surface, however, was treated with MMA monomer, which might explain the improved bonding.[5]

The shear bond strength value for PMMA denture base to Kooliner reline material obtained in this study (4.5 ± 0.5 MPa) was significantly higher than that obtained by Takahashi and Chai[9] (2.21 ± 4 MPa). In their study, the relined specimens were thermocycled before testing, which could have resulted in a lower value, whereas the present study investigated the initial shear bond strength.

Within the UDMA group, a similar observation was found, where higher shear bond strength was obtained with Secure as compared to Kooliner relines. The effect of dichloromethane adhesive dissolving and roughening the Eclipse specimen surface is shown in Figure 2, and the effect of this was thought to improve its bonding to Secure reline material.

Adhesive failure was observed for all reline materials in the UDMA group, even for the Eclipse reline group. The most likely explanation for adhesive failures was the highly cross-linked nature of polymerized UDMA, which might have prevented adequate monomer to penetrate into the high-density polymer network.[20] A SEM view of one representative sample of Eclipse base and Eclipse reline combination with adhesive failure is shown in Figure 4. For this reline combination, inadequate adaptation of Eclipse reline material due to its high viscosity might have also contributed to the weak adhesion. The manufacturer advocates warming up the resin in the conditioning oven at a temperature of 50°C before relining to improve the flow. Air entrapment during manual manipulation was another possibility that could have caused weakening of the adhesion.

On the other hand, a comparison among all reline materials in the UDMA group showed Eclipse reline to have the highest bond strength. A study of Eclipse resin polymerization kinetics showed that the polymerization rate increased at higher temperatures, particularly across melting point transition and above the glass transition temperature.[23] This conversion character might explain the increased diffusion rate of the reline material into the base polymer where high temperature was instituted during reline polymerization. This might have compensated for the effect of the highly cross-linked nature of the base polymer, and hence resulted in the observation of higher bond strength with Eclipse reline. The lowest strength obtained for the Eclipse base-Meliodent RR reline combination could not be explained based on the present study.

This in vitro study evaluated the bonding of relined denture base specimens by shear test; however, the study design may not simulate the actual clinical condition ideally. With this test, there was only one type of force applied at a time, compared to the various masticatory forces that dentures are subjected to during mastication. In addition, this study only investigated the initial shear bond strength, and therefore the results could not be extrapolated for long-term performance. Further studies are required to evaluate the effect of aging or thermocycling on bond strength; however, the result of this study could provide some valuable information to clinicians when selecting a reline material that will provide adequate bond strength to the denture. This is particularly so for any new material introduced into the market, such as Eclipse denture base polymer, where not much evidence is available related to its properties. Dentures constructed from Eclipse material can be successfully relined only if a satisfactory bond exists with the relining material. Poor adhesion and the lower shear bond strength of Eclipse polymer, particularly to intraoral reline materials, compared to PMMA denture base polymer as observed in this study warrant further investigation.

Conclusion

1. The type of denture base polymer and reline material, and their interaction affected the bond strength of denture base polymers to reline materials.
2. There was a statistically significant difference in the shear bond strength values among denture base-reline material combinations ($p < 0.05$) except for Meliodent-Kooliner and Eclipse-Meliodent RR denture base-reline combinations ($p > 0.05$).
3. Eclipse specimens showed adhesive failures for all reline materials tested.

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