International Journal of Adhesion & Adhesives 31 (2011) 112–118

Contents lists available at ScienceDirect

International Journal of Adhesion & Adhesives

journal homepage: www.elsevier.com/locate/ijadhadh

The effect of mechanical roughening and chemical treatment on shear bond strength of urethane dimethacrylate denture base resin ☆

Fazrina T.M. Ariff [a], Norsiah Yunus [b,*], Mirza R. Baig [b]

[a] Department of Restorative Dentistry, Faculty of Dentistry, University of Technology MARA, Shah Alam, Selangor, Malaysia
[b] Department of Prosthetic Dentistry, Faculty of Dentistry, University of Malaya, Kuala Lumpur, Malaysia

ARTICLE INFO

Article history:
Accepted 2 November 2010
Available online 24 November 2010

Keywords:
Composite
Surface roughness
Surface treatment
Shear bond

ABSTRACT

Relining of ill-fitting denture is often required to establish the fit of denture base, hence better retention and stability of the prostheses. However clinical success depends on the ability of reline resin to bond with denture base. The effect of surface preparations of urethane dimethacrylate (UDMA) denture base resin (Eclipse) on the shear bond strength (SBS) to auto-polymerizing polyethyl methacrylate reline material was evaluated. Eclipse specimens were mechanically prepared using two different tungsten carbide burs and submitted to chemical treatments either with dichloromethane (Secure adhesive) or methyl acetate (Eclipse Bonding Agent). Reline resin was then applied to the prepared surface and shear bond strength was tested after 24 h. Data was analyzed using two-way ANOVA and post-hoc Tukey HSD test at $p=0.05$. The morphological changes of Eclipse surfaces after preparations were also observed under SEM. The results showed that SBS was significantly affected by mechanical roughening, chemical treatment and their interactions. Higher reline SBS values were observed for Eclipse specimens without mechanical roughening compared to those with roughening. Both chemical agents improved reline SBS with the highest bond strength shown when chemically treated using Secure adhesive. For mechanically roughened specimens, Eclipse Bonding Agent (BA) resulted in significantly higher reline bond strength than Secure adhesive. SEM showed different surface appearance of Eclipse resin with various mechanical and chemical preparations.

1. Introduction

The most commonly used material for denture construction is polymethyl methacrylate (PMMA) denture base resin. However there has been also some interest in alternative materials such as urethane dimethacrylate, which was advocated partly because of its biocompatibility [1]. Denture processing method and mode of polymerization for this material also differs from the conventional heat-curing PMMA. The most recent urethane-based denture resin known as Eclipse requires both light and heat for polymerization. It uses high intensity visible light of 400–500 nm wavelength that reacts with hydroquinone present in the resin to trigger the polymerization process. To ensure complete polymerization, a high temperature is required and according to the manufacturer the maximum temperature of 129 °C is achieved during polymerization.

Laboratory investigation by the manufacturer and other independent studies reported that Eclipse resin possess better hardness, flexural and impact strength than the conventional PMMA [2–5]. It has also been shown that Eclipse material fulfilled the ISO 1567:1999 [6] requirement for type IV (light-curing) denture base material for flexural strength and modulus with adequate flexural strength after repair [5].

However, a poor bond strength of urethane dimethacrylate to reline resin compared to PMMA denture resin is still a concern [7] as this could lead to staining and bacterial harbouring. Complete bond failure could result in dilamination of reline layer from the denture surface [8–11] and therefore failure of the prosthesis to maintain its function.

The problem with highly cross-linked polymer such as Eclipse is that the penetration of reline monomer to the denture base to form strong bond is somewhat restricted[7,12]. An interwoven polymer network could only be established if the reline material is able to effectively penetrate the denture base resin [13]. Chemical agents such as dichloromethane and ethyl acetate have been used for surface preparation to improve the bond between PMMA denture base and auto-polymerizing resins [7,12,14,15]. However with urethane dimethacrylate, the effectiveness of these chemicals is not well documented except for our previous study [7].

Mechanical means of surface treatment to improve the bond of denture reline had also been suggested. Modification such as grinding with burs or abrading using air-borne particles were

☆ This study was supported by Vote F funding, University of Malaya, Kuala Lumpur, Malaysia.
* Corresponding author. Tel.: +603 79674881; fax: +603 79674535.
E-mail address: norsiah@um.edu.my (N. Yunus).

0143-7496/$ - see front matter © 2010 Elsevier Ltd. All rights reserved.
doi:10.1016/j.ijadhadh.2010.11.011

aimed at increasing the surface area and improve mechanical retention [13,16,17]. Most manufacturers recommend adjustment of denture fitting surface before relining and the same recommendation has been made for Eclipse by the manufacturer, however there is almost no information available with regards to the effect surface preparation on the bond strength to reline resins, since this material is comparatively new in the market. Previous study [7] compared reline bond strength of Eclipse with PMMA denture base resin and in that study the roughening of denture base specimens was performed on silicon carbide paper before relining. This type of surface preparation may not reflect actual clinical practice as denture is normally trimmed using rotary tungsten carbide bur.

The objective of the study is therefore to investigate the effect of mechanical roughening using different tungsten carbide burs and chemical agents on the shear bond strength of UDMA Eclipse denture base resin. Only one type of reline material (Kooliner) was selected for this study so as to control this variable since the composition and amount of monomer available at the bonding surface during polymerization could affect the bond strength [18]. It is also of concern due to the fact that Kooliner reline material has been shown to have relatively weak bond to Eclipse denture base resin [7]. The hypothesis that mechanical roughening and chemical surface treatment would have an effect on shear bond strength of relined Eclipse denture base resin was tested.

2. Materials and methods

The materials used in this study are shown in Table 1. Specimen of Eclipse was prepared by investing brass column measuring 15 mm in diameter and 4 mm height in plaster mould that was lined at the bottom with glass slab. The stone mould and Eclipse base plate resin were preheated in the conditioning oven (Dentsply Inc., York, USA) to 55 °C for 2 min before the resin was adapted using finger pressure into the mould. Polymerization was carried out for 10 min in a processing unit (Dentsply Inc., York, USA); the equipment and the process have already been described in a previous study [2]. Specimens were immersed in distilled water at 37 °C and after 30 days were embedded in epoxy resin (Mirapox, Miracon, Kuala Lumpur, Malaysia) with its surface that was processed against the glass slab slightly protruding above the resin. They were then divided into three groups of twenty four specimens. Each group was mechanically roughened using one of the following tungsten carbide rotary instruments (Edenta, Hauptstrasse, Switzerland): standard carbide (ISO 500 104 274190 060), fine carbide (ISO 500 104 274140 060) burs and one group without roughening that served as control. The standard and fine cross-cut tungsten carbide burs used have similar taper, diameter and shape except for the number of cutting flutes.

Surface roughening was performed by one operator using a straight hand-piece (Shick Dental Gmgh, Schemmerhosen, Germany) by running the bur along the surface of the specimen in one direction to create a plane that was level with the edge of the mounting resin (Fig. 1). The speed of hand-piece was set at 20,000 rpm and each specimen was trimmed for approximately 1 min with a new bur replaced after every eight specimens. For each bur group, specimens were further subdivided into three groups of eight specimens and submitted to one of the following chemical agents: Secure adhesive, Eclipse BA or without chemical treatment, which served as chemical control. Two layers of Secure adhesive were applied using brush on specimen surface and was left to dry before relining. For Eclipse BA, the manufacturer's instruction for bonding of Eclipse base plate to acrylic tooth was followed. Specimens were left immersed for 4–6 min in the bonding agent, which was warmed to 40 °C. All the specimens were then relined where split brass ring of 6 mm internal diameter and 2.5 mm height was placed at the centre of the exposed Eclipse specimen surface to secure the reline material. A cylindrical tube was then placed over the ring to prevent any movements during the relining procedure.

The reline resin (Kooliner) was mixed according to the recommended powder to liquid ratio of 15 mg powder to 6 ml liquid monomer. The mixture was then poured into the brass ring, which was placed over Eclipse specimen. Polymerization was carried out in an incubator for 10 min at a temperature of 37 °C to simulate the temperature of oral cavity. Specimens were stored in distilled

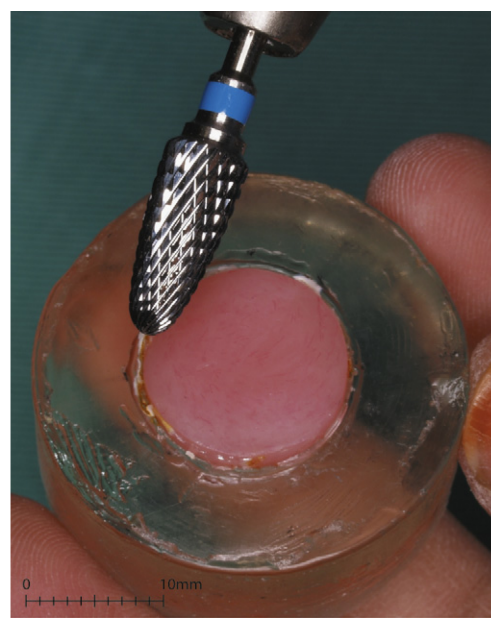

Fig. 1. Tungsten carbide bur used for mechanical preparation of Eclipse specimen, which has been mounted in epoxy resin.

Table 1
Denture base material, reline resin and chemical agents used in the study.

Brand name	Material type	Main composition	Manufacturer (batch number)
Eclipse base resin	Light-curing UDMA	Matrix: urethane dimethylcrylate Filler: silica and polymethacrylate beads	Dentsply Int., York, USA (Lot 051012)
Kooliner	Intraoral auto-polymerizing reline material	Powder: polyethyl methacrylate Liquid: isobutyl methacrylate, dimetyl p-toluidine	GC America, Alsip, USA (Lot no.-Powder: 0711021) (Lot no.-Liquid: 0710161)
Secure adhesive	Adhesive	Dicloromethane	Imtec Corp., Ardmore, USA (Lot 0807729)
Eclipse BA	Bonding agent	Methyl acetate	Dentsply Int., York, USA (Lot 060804A)

water at a temperature of 37 °C for 24 h before testing as described in ISO specification 11405:2003 [19] for short-term water storage.

Shear bond testing was carried out on a Universal Testing Machine (Shimadzu, Tokyo, Japan) at 1.0 mm/min crosshead speed. Compressive load was applied using a knife-edged blade, which was placed parallel to the materials interface. The test was performed dry under uniform atmospheric conditions at a temperature of 23 °C. The following formula was used to calculate the SBS:

$$F = N/A,$$

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

After testing, the interfaces of the specimens where failure occurred were examined under a stereomicroscope (Kyowa SD-2PL, Tokyo, Japan) at a magnification of $\times 10$ to determine mode of failure. Failure was categorized either as cohesive (more than 50% trace of reline material on Eclipse surface or vice versa), adhesive (no traces of reline material on Eclipse surface or vice versa) or mixed (less than 50% traces of reline material on Eclipse surface or vice versa). One examiner was involved with the recording.

Before relining, the two groups of specimens with mechanical preparation and control were also examined for surface roughness (*Ra*) using contact stylus profilometre (Ambios XP-1, Santa Cruz, USA). Measurement was made with the diamond stylus moving across the specimen surface under constant pressure of 10 mg with the speed of 0.1 mm/s. *Ra* is the mathematical average of the absolute values of the measured profile height of surface irregularities. The cut off length of each tracing was 2 mm and the measuring length was 10 mm. Three measurements were made for each specimen and the mean average *Ra* values were used for the statistical analysis.

To determine the morphological changes to Eclipse surfaces after mechanical roughening, one sample specimen for each bur type and control was additionally prepared and viewed under Scanning Electron Microscope (SEM) (Quanta 200, FEI, USA). Low vacuum image mode was used. In addition, two specimens mechanically prepared using standard bur were also examined each after submission to the chemical agent.

Two-way ANOVA and post-hoc Tukey HSD test were used for the analysis using SPSS for Windows (Release 12.01, SPSS Inc., Chicago, IL, USA).

3. Results and discussion

The mean reline shear bond strength of Eclipse resin mechanically roughened using different tungsten carbide burs and chemically treated with two different chemical agents are shown in Fig. 2. Two-way ANOVA indicated significant differences in SBS for the effect of mechanical roughening ($p < 0.001$), chemical treatment ($p < 0.001$) and their interactions ($p < 0.001$). The results showed that with mechanical roughening only, the bond strength was lower compared to the group without roughening where specimens were processed against glass. This was significant when roughened using fine bur but not significantly different with standard bur preparation. Based on the SEM views, Eclipse specimens showed different surface configurations for the 3 groups (Fig. 3). Fine bur preparation produced large area of smooth surface with some irregular surfaces (Fig. 3C) while standard bur produced more even distribution of waviness (Fig. 3B). These views are distinguishable from Eclipse surface that was not mechanically prepared (Fig. 3A).

The results for surface roughness (Fig. 4) showed significant difference in the *Ra* values between bur preparations; higher surface roughness value was shown by standard bur compared to fine bur preparation and the lowest was with control group. Radford et al. [20] in their study on denture base resin also showed that courser bur, which had lesser number of flutes, produced rougher surface than fine bur that had more cutting flutes. The manufacturer of the bur recommended using standard bur for gross trimming of denture base while fine bur is for final smoothening before polishing.

A rougher surface has always been thought to improve the reline bonding to denture base. In this study, although preparation of Eclipse surface with tungsten carbide bur increased the surface roughness value, this did not offer any additional advantage in terms of improved reline bond strength. One explanation for this was that since Eclipse resin was composite in nature, more filler particles were likely to be exposed to the surface after removal of resin matrix-rich outermost layer during trimming as

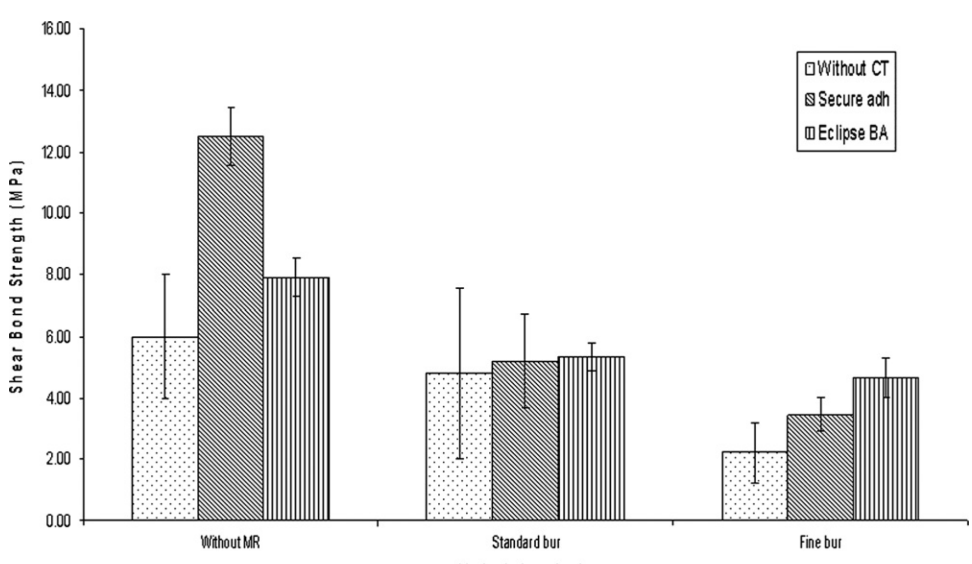

Fig. 2. Mean reline SBS (MPa) of Eclipse specimens mechanically roughened using different carbide burs and chemically treated using various chemical agents.

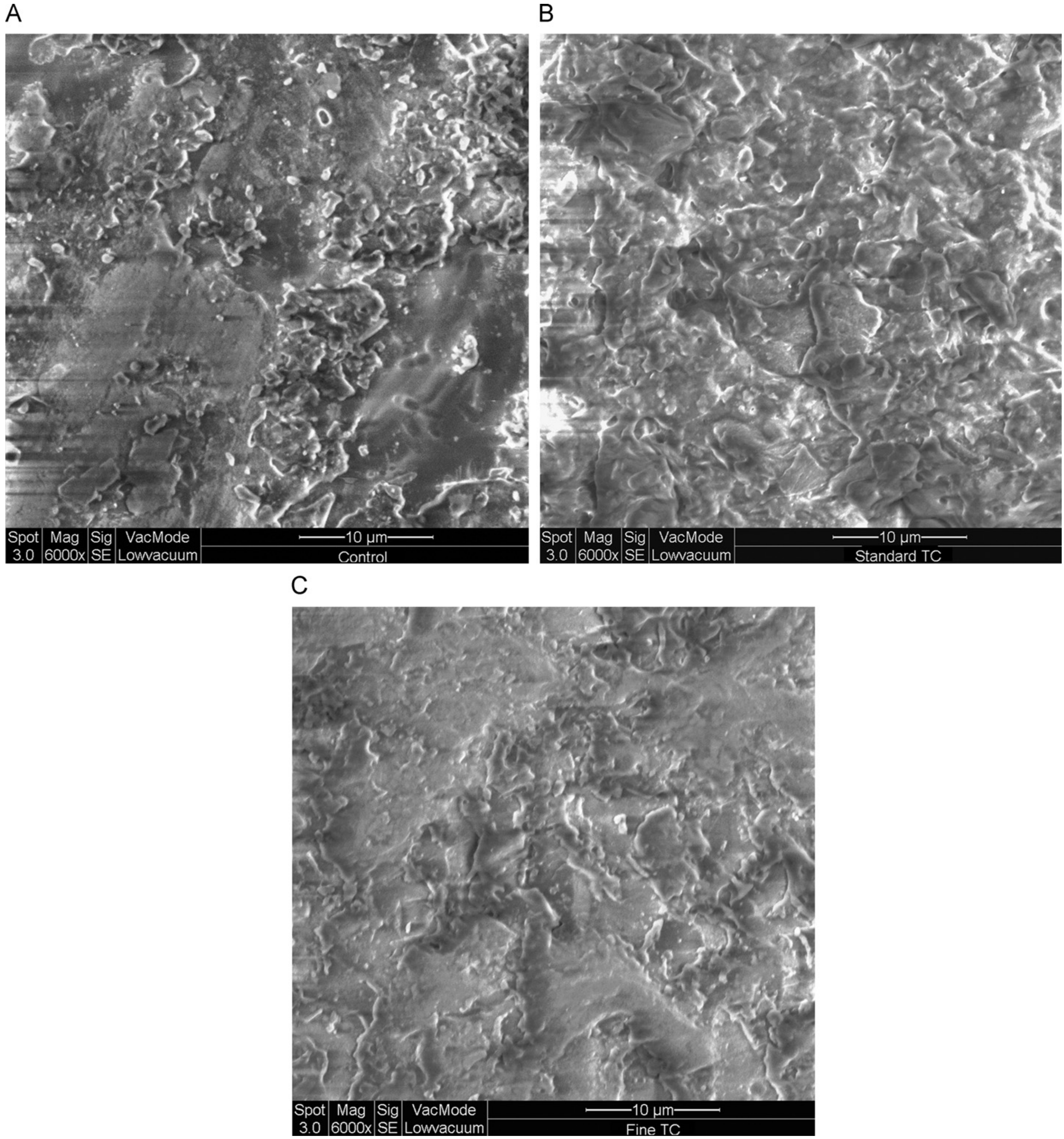

Fig. 3. (A) SEM view of Eclipse specimen surface without mechanical roughening ($\times$ 6000 magnification), (B) SEM view of Eclipse specimen surface with mechanical roughening using standard bur ($\times$ 6000 magnification) and (C) SEM view of Eclipse specimen surface with mechanical roughening using fine bur ($\times$ 6000 magnification).

illustrated in Fig. 5. Less area of bonding between urethane dimethacrylate matrix and reline resin was therefore available and this might have also explained for the lower bond strength for specimen roughened using fine bur (Fig. 5B). However with standard bur, where higher bond strength value was obtained, the deeper part of resin matrix might have also got roughened due to its cutting flutes (Fig. 5C). The additional roughening of resin matrix by courser bur in this study could have therefore increased the surface area for bonding with reline resin, which corroborated with higher bond strength when compared to fine bur.

The results of this study also showed that without mechanical preparation, higher shear bond strength was achieved where specimens were chemically treated with Secure adhesive compared to Eclipse BA. The SEM view of Secure adhesive-treated surface demonstrated aggressive dissolution of resin matrix to

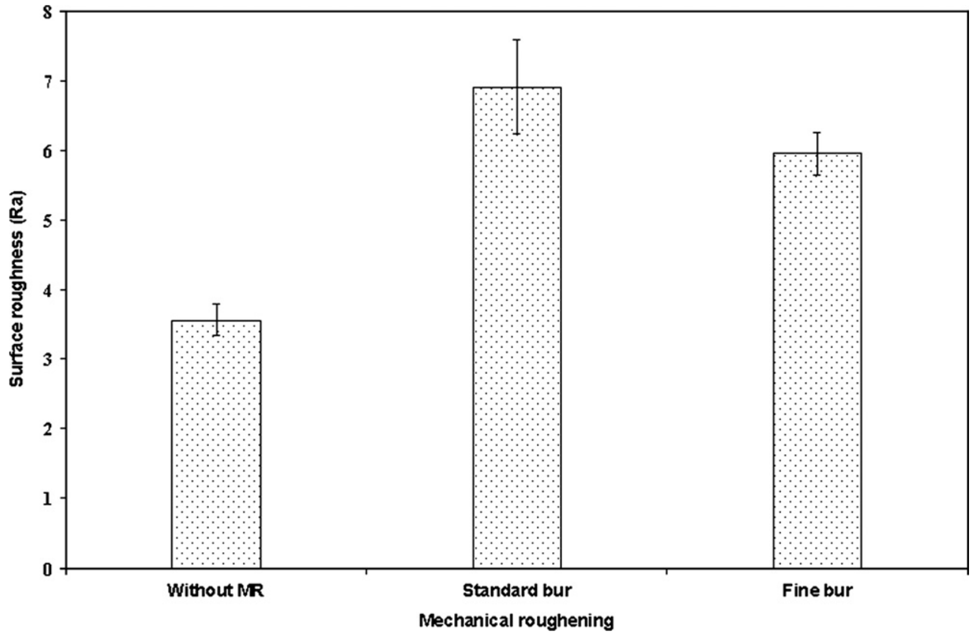

Fig. 4. Mean surface roughness (*Ra*) of Eclipse specimens with mechanical roughening only using two different carbide burs in comparison to specimens without roughening.

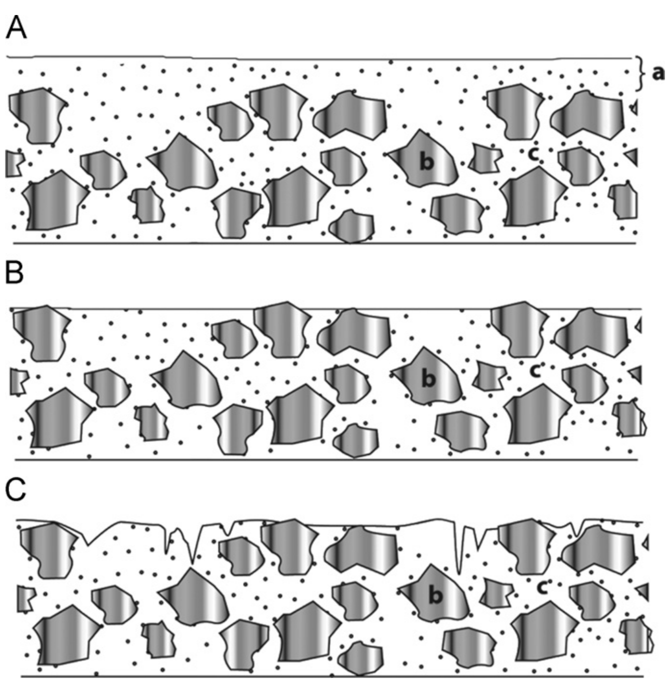

Fig. 5. Schematic diagram illustrating surface configuration of Eclipse specimens with mechanical roughening, (A) without roughening, showing the resin matrix-rich surface (*a) and the presence of filler particles (*b) underneath, (B) fine bur removes resin matrix-rich outermost layer exposing some fillers on the surface and (C) standard bur removes resin matrix-rich outermost layer and additionally roughens the deeper part of resin matrix (*c). (Diagram not drawn to exact scale.)

produce defined shape of filler particles with exposure of some fillers on the surface (Fig. 6A). The change in surface structure by the action of dichloromethane, which was the main composition of Secure adhesive, might have facilitated penetration of reline material into denture base resin [12–15]. The interlocking structure established at the interface could have therefore improved the bonding, which was also reflected by the highest SBS values among all groups. It was also observed that about 40% of the specimens in this group exhibited mixed mode of adhesive and cohesive failures compared to all other groups that showed 100% adhesive failure. Similarly with Eclipse BA, surface dissolution of resin matrix was evident however with a different appearance where rounded irregularities around the filler particles were observed (Fig. 6B).

In both mechanically prepared groups, treatment using Secure adhesive and Eclipse BA showed significantly higher bond strength than without chemical treatment, with Eclipse BA resulting in higher bond strength than Secure adhesive. SEM observation did not reveal remarkable differences where both chemicals produced layering effect on the Eclipse surfaces (Fig. 7A and B) with more filler particles exposed on the surface chemically treated with Secure adhesive (Fig. 7A). The reduction in the effective bonding area to reline resin as a result of more filler exposure may partially explain for the observed lower bond strength with Secure adhesive. The type of failure was 100% adhesive failure, which was in agreement with the results of previous study on relined Eclipse resin [7].

The study showed that chemical agents such as Secure adhesive and Eclipse bonding agent may be clinically useful in improving the shear bond strength of Eclipse denture base to a reline resin. However, the effect of chemical agents may be less on mechanically roughened Eclipse surface as compared to the surface without roughening.

However it needs to be mentioned that Eclipse denture is conventionally processed on stone cast, whereas in the present study control specimens were prepared by adapting against glass. Another limitation of the study was that the present study method and variables did not simulate all clinical conditions. Relined specimens were also tested 24 h after the procedure therefore the results could not be extrapolated for long-term effect. At the same time only one brand of reline resin was tested for the bond strength to Eclipse resin therefore the results cannot be generalized to other materials. Further studies are therefore recommended to investigate the long term performance of Eclipse with relining and to test using different types of reline resins available in the market.

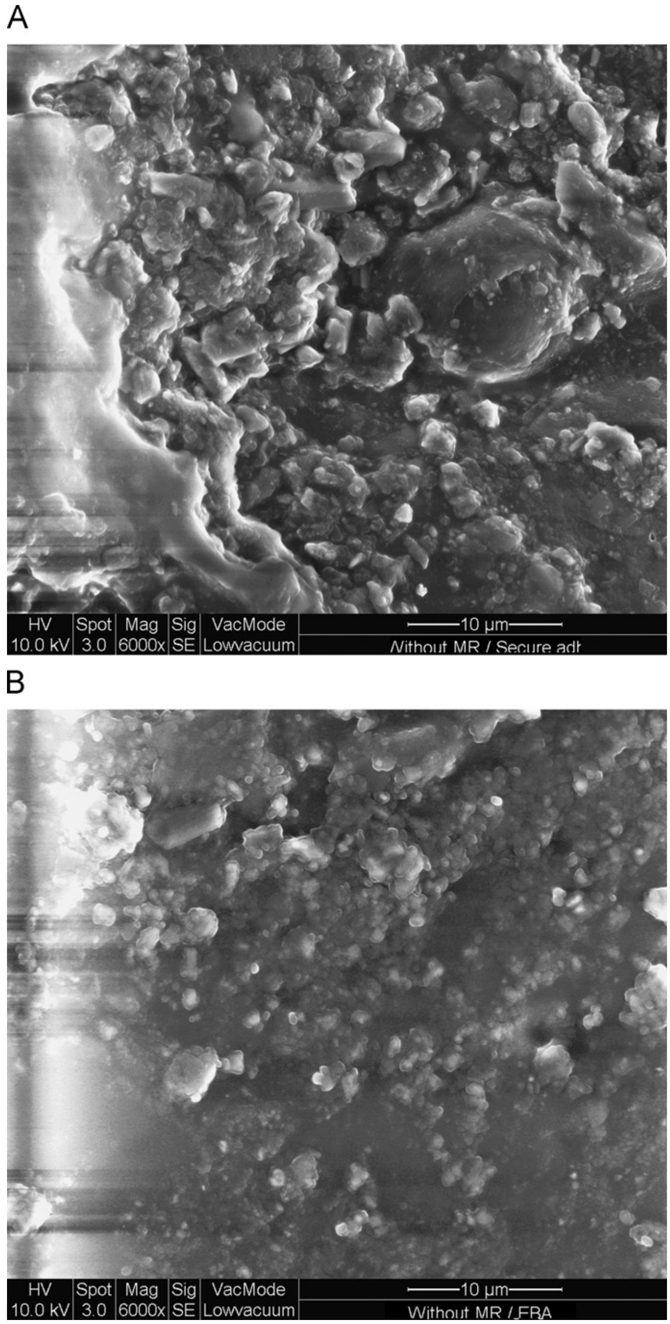

Fig. 6. (A) SEM view of Eclipse specimen without mechanical roughening and with chemical treatment using Secure adhesive ($\times$ 6000 magnification) and (B) SEM view of Eclipse specimen without mechanical roughening and with chemical treatment using Eclipse BA ($\times$ 6000 magnification).

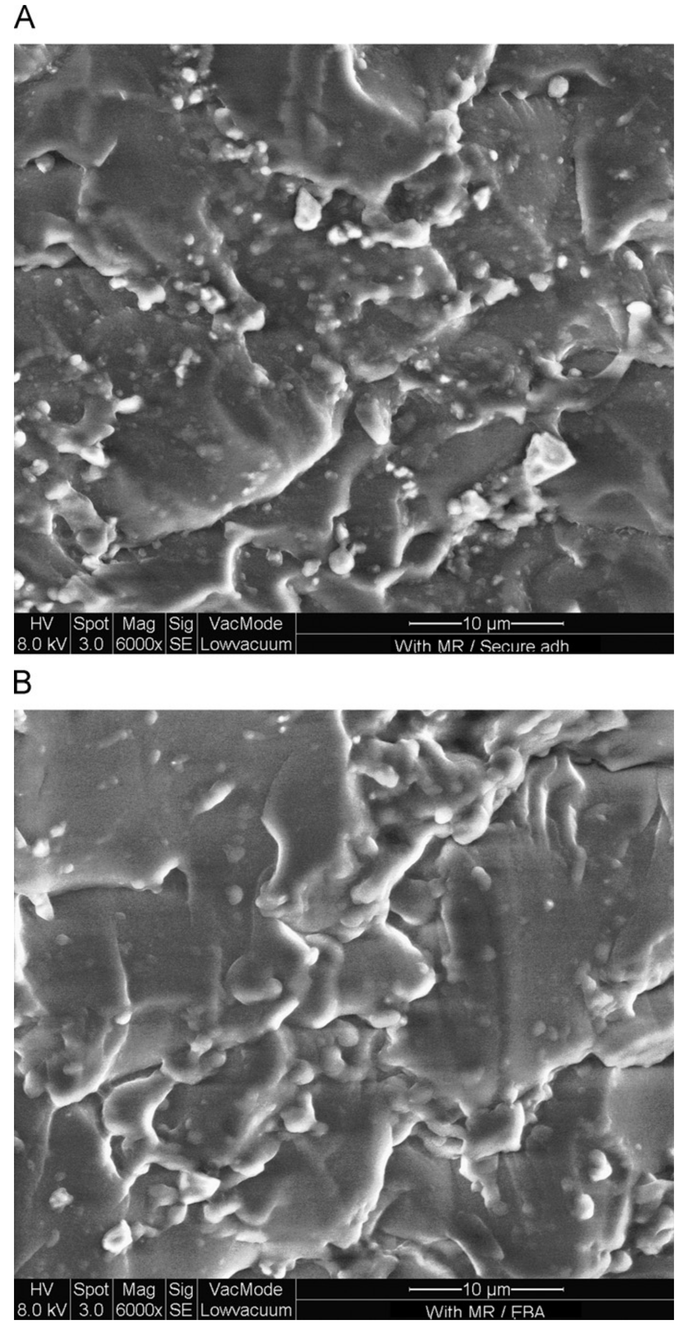

Fig. 7. (A) SEM view of Eclipse specimen with mechanical roughening and chemical treatment using Secure adhesive ($\times$ 6000 magnification) and (B) SEM view of Eclipse specimen with mechanical roughening and chemical treatment using Eclipse BA ($\times$ 6000 magnification).

4. Conclusion

Within the limitations of the study, the following conclusions were made:

- Relined SBS was significantly affected by mechanical roughening, chemical treatment and their interactions ($p < 0.05$).
- Higher reline SBS values were observed for Eclipse specimens without mechanical roughening when compared to roughening.
- Chemical treatment using Secure adhesive on specimens without roughening resulted in the highest SBS value among all groups.
- For mechanically roughened specimens, Eclipse BA showed significantly higher SBS than Secure adhesive.

References

[1] Ogle RE, Sorensen SE, Lewis EA. J Prosthet Dent 1986;56:497.
[2] Ali IL, Yunus N, Abu Hassan MI. J Prosthodont 2008;17:545.
[3] Machado C, Sanchez E, Azer SS, Uribe JM. J Dent 2007;35:930.
[4] Diaz AM, Vargas MA, Shaull KL, Laffoon JE, Qian F. J Prosthet Dent 2008;100:47.
[5] Pfeiffer P, An N, Schmage P. J Prosthet Dent 2008;100:293.
[6] International Organization for Standardization, Dentistry-denture base polymers, ISO 1567: 1999.
[7] Ahmad F, Yunus N. J Prosthodont 2009;18:596.

[8] Haywood J, Basker RM, Watson CJ, Wood DJ. Eur J Prosthodont Restorative Dent 2003;11:157.
[9] Matsumura H, Tanoue N, Kawasaki K, Atsuta M. J Oral Rehabil 2001;28:640.
[10] Arima T, Nikawa H, Hamada T, Harshini. J Prosthet Dent 1996;75:457.
[11] Arena CA, Evans DB, Hilton TJ. J Prosthet Dent 1993;70:126.
[12] Takahashi Y, Chai J, Takahashi T, Habu T. Int J Prosthodont 2000;13:59.
[13] Takahashi Y, Chai J. Int J Prosthodont 2001;14:531.
[14] Shimuzu H, Ikuyama T, Hayakawa E, Tsue F, Takahashi Y. Acta Odontol Scand 2006;64:159.
[15] Shimizu H, Kakigi M, Fujii J, Tsue F, Takahashi Y. Int J Prosthodont 2008;17:451.
[16] Jagger RG, Al-Athel MS, Jagger DC, Vowles RW. Int J Prosthodont 2002;15:55.
[17] Curtis DA, Eggleston TL, Marshall SJ, Watanabe LG. Dent Mater 1989;5:314.
[18] Minami H, Suzuki S, Minesaki Y, Kurashige H, Tanaka T. J Prosthet Dent 2004;91:164.
[19] International Organization for Standardization, Testing of adhesion to tooth structure, ISO/TS 11405:2003.
[20] Radford DR, Watson TF, Walter JD, Challacombe SJ. J Prosthet Dent 1997;77:200.