研　　究

Bonding effectiveness of self-adhesive resin cements to enamel and dentin

Rintaro TERATA, Nobuo OKADA, Minoru KUBOTA
Department of Operative Dentistry and Endodontics, School of Dentistry, Iwate Medical University.
(Chief : Prof. Minoru KUBOTA)
[Received : January 9, 2009 ; Accepted : March 5, 2009]

Abstract : The purpose of this study was to evaluate the tensile bond strength of resin cements to enamel or dentin, one-hundred-and-twenty freshly extracted bovine incisor teeth were ground to expose the enamel and dentin surfaces. Two kinds of self-adhesive resin cements (Maxcem™: Kerr and Smartcem™;Dentply-Sankin) and two kinds of self-etching primer resin cements (Linkmax™: GC and Resicem™: Shofu) were used in accordance with each manufacturer's instructions. In the Maxcem and Smartcem groups, a subset of samples were treated by etching with 40% phosphoric acid for 15 seconds and applied to both the enamel and dentin prior to luting, and were designated as the etched Maxcem and etched Smartcem groups. All resin cements were bonded in a stainless steel mold onto enamel or dentin. After bonding, the specimens were stored in water at 37℃ for 24 hours, and the tensile bond strength was measured at a crosshead speed of 0.5 mm/minute. The data were analyzed statistically by one-way ANOVA and post-hoc Tukey-Kramer test. Maxcem and Smartcem had lower bond strength to both enamel and dentin than Linkmax and Resicem ($p<0.05$). Phosphoric acid etching significantly increased the tensile bond strength of Maxcem and Smartcem to enamel ($p<0.05$) but not to dentin. These results suggested that self-adhesive resin cement had a lower bonding effectiveness to enamel and dentin than self-etching primer resin cement.

Key words : self-adhesive resin cement, bond strength, phosphoric acid, enamel, dentin

Bonding effectiveness of self-adhesive resin cements to enamel and dentin
Rintaro TERATA, Nobuo OKADA, Minoru KUBOTA
(Chief : Prof. Minoru KUBOTA)
Department of Operative Dentistry and Endodontics, School of Dentistry, Iwate Medical University.
1-3-27, Chuo-dori, Morioka, Iwate, 020-8505, Japan

Introduction

Progress in adhesive dentistry has been accompanied by the choice of resin cement for ceramic, composite resin, and metal restorations[1]. Currently, all resin cements are based upon the use of an etch-and-rinse adhesive or a self etching-primer along with a low viscosity resin composite[2]. A multi-step application technique is complex and rather technique sensitive, and consequently may compromise bonding effectiveness[3]. Recently, a new generation of resin cement with a novel concept called "self-adhesive" resin cement has been brought to the dental market. This material has a two-part paste/paste dual-cure resin cement that is able to perform both self-etching and self-adhering. The unique formulation combines the etchant, primer, adhesive, and resin cement into one material. Therefore, the application of adhesive is a simple and easy operation. Rely X Unicem™ is undoubtedly the most thoroughly investigated self-adhesive resin cement in the current literature. Rely X Unicem has satisfactory bond strength to dentin but appears to be a weak link in its bond strength to enamel[2]. The strength of adhesion in other currently marketed self-adhesive resin cements to enamel and dentin has not been clarified.

This study evaluated the tensile bond strength of self-adhesive resin cement to enamel and dentin.

Materials and Method

One-hundred-and-twenty bovine mandibular incisor teeth were used within 12 hrs after slaughtering. Flat labial enamel or dentin surfaces in the central crown were ground with Surfmet™ (Buehler, Lake Bluff, IL, USA.). The surfaces were finished with 200, 400, and 600 grit silicon carbide paper to enamel thickness of 2.0 mm or dentin thickness of 2.0mm.

Two kinds of self-adhesive resin cements (Maxcem™: Kerr Corp., Orange, CA, USA. and Smartcem™: Dentsply-Sankin KK, Tochigi, Japan.) and two kinds of self-etching primer resin cements (Linkmax™: GC Corp., Tokyo, Japan. and Resicem™: Shofu Inc., Kyoto, Japan.) were used in accordance with each manufacturer's instructions (Table 1 and Table 2).

Table 1. Materials used in this study

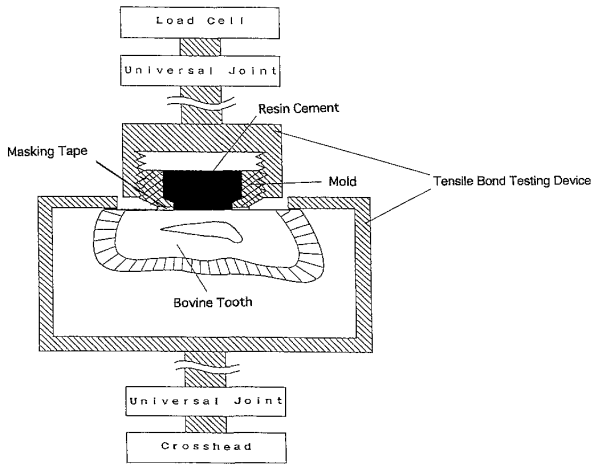
Self-adhesive resin cement
Maxcem: Kerr Corp., Orange , CA, USA; Lot No. 2712175
Base: UDMA, Camphorquinone, Fluoroaluminosilicate glass
Catalyst: Bis-GMA, TEGDMA, Glycerophosphoric acid dimethacrylate, Bariumaluminoborosilicate glass
Smartcem: Dentply-Sankin KK, Tochigi, Japan; Lot No. 419-001
Base: HEMA, 4-MET, PEM-F, Polymerization initiator
Catalyst: Butylene dimethacrylate, Sufinic acid, Tertiary amine
Self-etching primer resin cement
Linkmax: GC Corp., Tokyo, Japan; Lot No. 0704191
Base: Methacrylic acid ester, Aluminosilicate glass
Catalyst: Methacrylic acid ester, Aluminosilicate glass
Self-etching primer
EP-A: Water, Ethanol, 4-META, Methacrylic acid ester
EP-B: Ethanol, Catalyst
Resicem: Shofu Inc., Kyoto, Japan; Lot No. 0107
Paste A: UDMA, TEGDMA, Fluoroaluminosilicate glass, Polymerization initiator
Paste B: UDMA, TEGDMA, Fluoroaluminosilicate glass, 4-AET, 2-HEMA Polymerization initiator
Self-etching primer
Primer-A: Water, Acetone, Polymerization initiator
Primer-B: 2-HEMA, 4-AET, Acetone,

Table 2. Handling procedure

Self-adhesive resin cement
Maxcem
Dispensing via automix syringe
Cement placed in a mold onto enamel or dentin
Dual cured (exposed to light for 40sec and held for 3 min)
Smartcem
Dispensing via automix syringe
Hand Mixing of cement for 20sec
Cement placed in a mold onto enamel or dentin
Dual cured (exposed to light for 40sec and held for 3 min)
Self-etching primer resin cement
Linkmax
Mixing EP-A and EP-B
Self-etching primer was applied to enamel or dentin for 30sec
Enamel or dentin was dried with oil-free compressed air
Hand Mixing of cement for 10sec
Cement placed in a mold onto enamel or dentin
Dual cured (exposed to light for 40sec and held for 3 min)
Resicem
Mixing Primer-A and Primer-B
Self-etching primer was applied to enamel or dentin for 20sec
Enamel or dentin was dried with oil-free compressed air
Dispensing via automix syringe
Cement placed in a mold onto enamel or dentin
Dual cured (exposed to light for 40sec and held for 3 min)

In the Linkmax group, etching primer A and etching primer B were mixed, applied to both the enamel and dentin for 30 seconds and then gently air-dried. In the Resicem group, primer A and primer B were mixed, applied to both the enamel and dentin for 20 seconds and then gently air-dried (Table 2). All resin cements were mixed and placed in a stainless steel mold (diameter 4.0 mm, height 3.5 mm) onto enamel or dentin (Figure). In the Maxcem and Smartcem groups, a subset of samples were treated by etching with 40% phosphoric acid (K-etchant™: Kuraray Medical Inc., Tokyo, Japan.) for 15 seconds and applied to both the enamel and dentin prior to luting, and were designated as the etched Maxcem and etched Smartcem groups. The resin cement was covered with a plastic matrix and pressed gently from above. The specimens were held firmly for 3 minutes by a gripping device to prevent slippage and then exposed to visible-light for 40 seconds using a hand-held unit (New Light VL-II™: GC Corp., Tokyo, Japan.). All specimens were left standing at room temperature for 15 minutes and stored in water at 37℃ for 24 hours. The specimens were directly positioned in tensile bond testing devices attached to a universal testing machine (Instron 4204™: Instron Corp., Canton, MA, USA.) The tensile bond strength was measured at a crosshead speed of 0.5 mm/minute (Figure). Ten specimens were measured for each testing group.

Figure. Diagram of tensile bond strength test



The data were analyzed statistically by a one-way ANOVA and post-hoc Tukey-Kramer test with the value of statistical significance set at the 0.05 level.

Results

Table 3 shows the tensile bond strength of resin cements to enamel and dentin. Maxcem and Smartcem had lower bond strengths to both enamel and dentin than Linkmax and

Table 3. Tensile bond strength of resin cement (MPa ± S.D., n = 10)

	Maxcem	Smartcem	Linkmax	Resicem
Enamel	3.5 ± 1.9a	3.8 ± 1.0a	8.2 ± 2.8b	15.1 ± 4.3c
Dentin	3.7 ± 2.0a	3.8 ± 1.7a	7.3 ± 2.6b	8.7 ± 2.6b
Etched enamel	13.8 ± 3.2c	10.2 ± 1.4b		
Etched dentin	3.6 ± 3.2a	3.8 ± 1.4a		

Different letters express statistically significant differences ($p<0.05$).

Resicem ($p<0.05$). Etching treatment significantly increased the tensile bond strength of Maxcem and Smartcem to enamel ($p<0.05$) but not to dentin.

Discussion

It is well known that the length of storage time and the storage conditions of extracted teeth affect their characteristics[4-7]. To avoid these factors, freshly-extracted teeth were used in this study.

Self-adhesive resin cement can be used on enamel and dentin without the need for any surface treatment because the material combines the etchant, primer, and adhesive resin that are mixed into a single paste before being used. This concept could reduce the length of time of treatment and minimize the possibility of contamination during the bonding procedure. The self-adhesive material can be useful for a clinician and a patient if it can have sufficient bond strength to enamel and dentin.

The present study showed that Maxcem and Smartcem, which are self-adhesive resin cements, had less bond strength to both enamel and dentin than two kinds of self-etching primer adhesive resin cements, Linkmax and Resicem. Bishara *et al.*[8] reported that the shear bond strength of Maxcem was 5.9 ± 2.7 MPa to enamel. Goracci *et al.*[9] also reported that the microtensile bond strength of Maxcem was 7.9 ± 3.2 MPa to enamel and 5.2 ± 1.6 MPa to dentin. These results were slightly higher than our findings. Bishara *et al.*[8] reported Maxcem had lower bond strength to enamel than the two-step self-etch primer/adhesive system. The limited micro-mechanical retention by the self-adhesive system might be responsible for the lower bond strength to enamel. Indeed, the phosphoric acid etching to enamel increased the bond strength of self-adhesive resin cement and made the same bond strength as that of self-etching primer resin cement. Hikita *et al.*[10] and De Munck *et al.*[11] reported that phosphoric acid enhanced the bond strength of self-adhesive " RelyX Unicem" resin cement to enamel. Ibarra *et al.*[12] reported that the Single Bond™ system improved the microleakage of porcelain veneer restoration when bonding to enamel. These results indicated that phosphoric-acid etching enhanced the bond strength of self-adhesive resin cement and agreed with our findings. The etching effect of the self-adhesive system alone was insufficient. Acidic monomers, such as glycerophosphoric acid dimethacrylate, 4-MET, 4-META, 4-AET, react with water in the enamel and dissolve the smear layer. The limited working time and high viscosity of cements might bring about insufficient and limited etching effects.

On the other hand, phosphoric-acid etching could not improve the bond strength of self-adhesive resin cement to dentin. Hikita *et al.*[10] and De Munck *et al.*[11] reported that

phosphoric acid decreased the bond strength of self-adhesive "RelyX Unicem" resin to dentin. These results indicated that phosphoric-acid etching did not improve the bond strength of self-adhesive resin cement, or that the etching effect of the self-adhesive system did not play an important role in bonding to dentin. Phosphoric-acid etching removed the weak smear layer and exposed the thick and compact collagen mesh. The penetration and diffusion of an adhesion promoting monomer might play an important role in bonding to dentin. In the self-adhesive system the penetration and diffusion of monomer might be insufficient on dentin because of the viscosity of the cement and the limited working time.

The lower adhesion of the self-adhesive resin cement must be attributed to the limited etching effect, penetration, and diffusion of the monomer because of the viscosity of the cement and the limited working time. Consequently adhesion might be made only on the superficial surface of the enamel and dentin. The improvement of adhesion to enamel or dentin and the evaluation of long-term clinical performance are necessary for self-adhesive resin cements.

Conclusion

It can be concluded that self-adhesive resin cement had a lower bonding effectiveness to enamel and dentin than self-etching primer resin cement.

Acknowledgements

This study was supported by Grants-in-Aid for High Performance Bio-Medical Materials Research Project and Open Research from Ministry of Education, Science, Sports and Culture of Japan.

References

1. Rosenstiel, S. F., Land, M. F., and Crispin, B. J. : Dental luting agents: a review of the current literature. *J. Prosthet. Dent.* 80 : 280-301, 1998.
2. Radovic, I., Monticellii, F., Goracci, C., Vulicevic, R. Z., and Ferrai, M : Self-adhesive Resin Cements: A Literature Review. *J. Adhes. Dent.* 10 : 251-258, 2008.
3. Mak, Y. F., Lai, S. C., Cheung, G. S., Chan, A. W., Tay, F. R., and Pashley, D. H. : Micro-tensile bond testing of resin cements to dentin and an indirect resin composite. *Dent. Mater.* 18 : 609-621, 2002.
4. Kimura, S., Shimizu, T., and Fujii, B. : Influence of dentin on bonding of composite resin: Part 1.-Effect of fresh dentin and storage conditions. *Dent. Mater. J.* 4 : 68-80. 1985.
5. Retief, D. H., Wendt, S. L., Bradley, E. L., and Denys, F. R. : The effect of storage media and duration of storage of extracted teeth on the shear bond strength of Scotchbond 2 / Silux to dentin. *Am. J. Dent.* 2 : 269-273, 1989.
6. Goodis, H. E., Marshall, G. W. Jr., and White, J. M. : The effects of storage after extraction of the teeth on human dentine permeability in vitro. *Arch. Oral Biol.* 36: 561-566, 1991.
7. Goodis, H. E., Marshall, G. W. Jr., White, J. M., Gee, L., Hornberger, B., and Marshall, S. J. : Storage effects on dentin permeability and shear bond strengths. *Dent. Mater.* 9 : 79-84, 1993.
8. Bishara, S., Ajiouni, R., Laffoon, J. F., and Warren, J. J. : Comparison of shear bond strength of two self-etch primer/adhesive systems. *Angle Orthod.* 76 : 123-126, 2006.
9. Goracci, C., Cury, A. H., Cantoro, A., Papacchini, F., Tay, F. R., and Ferrai, M. : Microtensile bond strength and interfacial properties of self-etching and self-adhesive resin cements used to lute composite onlays under different seating forces. *J. Adhes. Dent.* 8 : 327-335, 2006.
10. Hikita, K., Meerbeek, B. V., Munck, J. D., Ikeda, T., Landuyt, K. V., Maida, T., Lambrechts, P., and Peumans, M. : Bonding effectiveness of adhesive luting agents enamel and dentin. *Dent. Mater.* 23 : 71-80, 2007.
11. De Munck, J., Vargas, M., Van Landuyt, K., Hikita, K., Lambrechts, P., and Van Meerbeek, B. : Bonding of an auto-adhesive luting material to enamel and dentin. *Dent. Mater.* 20 : 963-971, 2004.
12. Ibarra, G., Johnson, G. H., Geurtsen, W., and Vargas, M. A. : Microleakage of porcelain veneer restorations bonded to enamel and dentin with a new self-adhesive resin-based dental cement. *Dent. Mater.* 23 : 218-225, 2007.

セルフアドヒーシブレジンセメントのエナメル質および象牙質に対する接着性能

寺田林太郎，岡田　伸男，久保田　稔
岩手医科大学歯学部歯科保存学第一講座
（主任：久保田　稔　教授）

（受付：2009年1月9日）
（受理：2009年3月5日）

抄録：この研究の目的は，120本のウシ永久下顎前歯唇面を削り露出させたエナメル質および象牙質に対するセルフアドヒーシブレジンの接着強さを評価することにある。2種類のセルフアドヒーシブレジンセメント（Maxcem およびSmartcem）並びに2種類セルフエッチングプライマーレジンセメント（Linkmax およびResicem）を製造業者指示に従って使用した。さらにMaxcem およびSmartcem では15秒間40%リン酸前処理を行ったものを酸処理群とした。レジンセメントはモールドを用いエナメル質および象牙質に接着させた。試料は24時間37℃水中に保管後，クロスヘッドスピード0.5mm/min にて引張り接着強さを測定した。得られた値はOne-way ANOVA と Tukey-Kramer test を用いて行った。Maxcem およびSmartcem はエナメル質および象牙質のいずれでもLinkmax およびResicem よりも接着強さが低かった（$p < 0.05$）。リン酸処理はMaxcem およびSmartcem のエナメル質への接着強さを改善（$p < 0.05$）したが，象牙質への接着強さを改善できなかった。本研究では，セルフアドヒーシブレジンはエナメル質および象牙質への接着性能がセルフエッチングプライマーレジンセメントよりも低いことが明らかとなった。