Effect of Timing of Light Curing on the Shear Bond Strength of Three Self-adhesive Resin Cements

Yeon-Kwon Yoo, DDS, MSD[1], Sung-hun Kim, DDS, PhD[2]
Jae-Jun Ryu, DDS, MSD, PhD[3], Jae-Jun Ryu, DDS, MSD, PhD[4]

1 Graduate student, Graduate School of Clinical Dentistry, Korea University, Korea
2 Associate professor, Department of Prosthodontics and Dental Research Institute, Seoul National University, Korea
3 Full Professor, Aesthetic Restoration Dentistry, Graduate School of Clinical Dentistry, Korea University, Korea
4 Aesthetic Restoration Dentistry, Graduate School of Clinical Dentistry, Korea University, Korea

Corresponding Author

Jae-Jun Ryu, DDS, MSD, PhD
Aesthetic Restoration Dentistry, Graduate School of Clinical Dentistry, Korea University, Korea
Tel: 82-31-412-5370, E-mail: koprosth@unitel.co.kr

• Abstract

Objectives. The objectives of this study were: 1) to compare the effect of varying timing of light curing on shear bond strength, and; 2) to compare the shear bond strength of three self-adhesive cements.

Materials and methods. A total of 72 extracted non-carious teeth were divided into 24 for Unicem tests, 24 for Maxcem tests, and 24 for Biscem tests; they were assigned 3 * 2 subgroups of 12 teeth each. The specimens were prepared as follows: 1) The calculus and periodontal ligament were removed from the teeth; 2) The teeth were stored in normal saline; 3) The occlusal enamel of each tooth was removed using high-speed coarse diamond burs under water cooling, and; 4) Finally, the teeth were flattened by 600-grit silicone carbide paper disks. Resin blocks were adhered using either Unicem, Maxcem, or Biscem. Light curing timing was divided into two groups: U10, M10, and B10 were exposed to light after 10 seconds, and; U150, M150, and B150 on the other side were exposed to light after 150 seconds. Shear bond strength was measured by a Universal testing machine with cross head speed of 1mm/min. T-test and One way ANOVA were used for the statistical analysis of data.

Results. The shear bond strength of U150 was not significantly higher than that of U10 (U150: 20.55.7Mpa, U10: 18.73.80Mpa). On the other hand, the shear bond strength of M150 was significantly higher than that of M10. The shear bond strength of B150 was also significantly higher than that of B10 (M150:14.45.7Mpa, M10: 9.94.2Mpa, B150: 24.38.3Mpa, B10: 17.27.3Mpa). When the light curing timing was 10sec after bonding, the shear bond strength of Unicem was highest; the shear bond strength of Biscem was highest when the light curing timing was 150sec after bonding (U10: 18.73.80Mpa, B150: 24.38.3Mpa).

Significance. Since Unicem is less sensitive based on light curing timing, dentists seem to use it without considering the light curing timing. Maxcem showed the lowest bonding strength (especially M10). Thus, when using Maxcem, dentists need to delay the light curing after adhesion.

• Keywords : Self-adhesive resin cement, Unicem, Maxcem, Biscem, Shear bond strength

Ⅰ. Introduction

Conventional resin cement entailed a cumbersome process, i.e., primer and bonding were applied after acidic corrosion to teeth and simultaneous bonding onto a prosthesis such as crown or in-lay was executed. From the dental aspect of adhesion, the use of the one-bottle system[1] developed through 5 generations and self-etching primer[2] has made resin cement simple to use and commercially available. Nonetheless, it still carried the inconvenience of going through multiple processes. To address such inconvenience, self-bonding resin cement called self-adhesive resin cement ("SARC") was developed. Recently, its use has been on the rise3). As brand examples, Unicem®(3M ESPE, USA), Maxcem® (Kerr, USA), and Biscem®(Bisco, USA) have been commercially available. This cement offers a one-step adhesive process by simplifying the conventional multiple procedure. In other words, without the prerequisite dentin process, adhesion with dental fiber was enabled. This cement contains multi-functional phosphoric acidic methacrylates, and this plays a significant role in adhesion[4]. For Unicem®, many studies revealed that it has similar adhesive power compared to other conventional resin cement[5-7]. Moreover, in terms of margin fit, it was found to be superior to other cements[8,9]. Unicem®, Maxcem®, and Biscem® are all dual-curing types of cement, and their manufacturers recommend waiting for 30~60 seconds before executing light curing. During this period, multi-functional phosphoric acidic methacrylates are known to react to mineral filler and tooth structure[4]. Nonetheless, there are no known studies on the impact of the range of varying timing of light curing on adhesive power.

Therefore, this study proposes examining the effect of varying timing of light exposure on SARC adhesion and comparing the adhesive intensity among commercially available cements: Unicem®, Maxcem®, and Biscem®.

Ⅱ. Materials and Test Method

1. Materials

(1) 72 extracted healthy caries-free teeth
(2) 3M's Unicem®
(3) Kerr's Maxcem®
(4) Bisco's Biscem®

2. Preparation of test samples

72 caries-free healthy teeth stored in saline solution following extraction were embedded with self-curing resin SNAP®(Parkell, USA) in a cylinder-shaped mold. Afterward, the crowns were immediately subjected to diamond cutting. The exposed dentin was ground with a 600-grit silicone carbide paper, cleansed in running water, and stored in distilled water. At the time of adhesion onto dentin, keel resin blocks were created in a cylinder with diameter of 3 mm and height of 5 mm. For consistent surficial roughness, a low-speed diamond saw was used for cutting. After cement application, the prepared resin blocks were adhered onto each tooth (Fig. 1). Based on the classified test groups, light curing was differently applied. One group was subjected to light curing 10 seconds after cement application, and another group, 150 seconds after cement application. Bluephase (Ivoclar Vivadent, Germany), a light curing unit, was used.

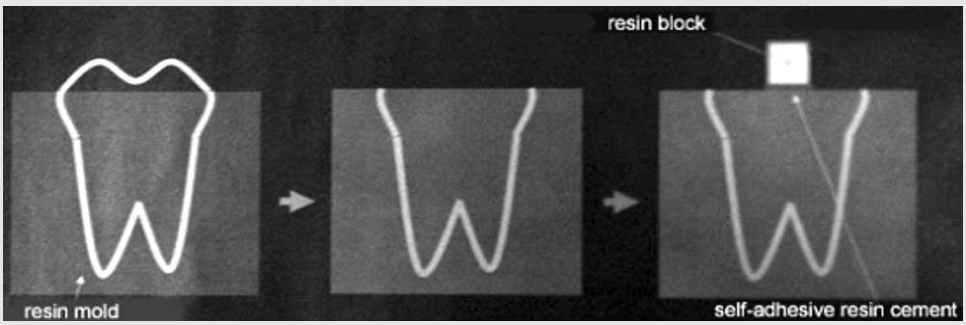

Fig. 1. Preparation step for specimen

3. Classification of Test Groups

(1) Unicem® group

A total of 24 resin blocks were coated with Unicem® and adhered onto 24 teeth using light pressure; light curing was then performed. The group subjected to light curing 10 seconds later was classified as U10 (n=12), and that subjected to light curing 150 seconds later, as U150 (n=12).

(2) Maxem® group

After Maxem® application, 24 resin blocks were adhered to 24 teeth using light pressure; light curing was then performed. The group subjected to light curing 10 seconds later was classified as M10 (n=12), and that subjected to light curing 150 seconds later, as M150 (n=12).

(3) Biscem® group

After Biscem® application, 24 resin blocks were adhered onto 24 teeth using light pressure; light curing was then performed. The group subjected to light curing 10 seconds later was classified as B10 (n=12), and that subjected to light curing 150 seconds later, as B150 (n=12).

4. Measurement of shear strength

All test samples were stored in 37°C distilled water for 24 hours. Afterward, shear strength between dentin and resin blocks was measured using a universal tester (Universal Testing Machine, Instron 4485, USA). The samples were secured, and shear force was applied onto the connective interface at 1 mm/min cross head speed. Shear strength was measured at the division moment of resin blocks (Fig. 2).

Table 1. Sameness and difference between two materials

	Sameness	Difference
Maxcem®	No adhesive needed Excellent mechanical properties Good aesthetics Minimal fluoride release Good bond strength	Paste/Paste Auto syringe
Unicem®	No adhesive needed Excellent mechanical properties Good esthetics Minimal fluoride release Good bond strengths	Powder/Liquid Mixing machine
Biscem®	No adhesive needed Excellent mechanical properties Good esthetics Minimal fluoride release Good bond strengths	Paste/Paste Auto syringe Curved tip

Table 2. Composition of Rely X Unicem®

Powder	Liquid
Glass powder Initiator Silica Substituted pyrimidine Calcium hydroxide Peroxy compound Pigment	Methacrylated phosphoric ester Dimethacrylate Acetate Stabilizer Initiator

Table 3. Composition of Maxcem®

Base	Catalyst
UDMA	GPDM
HEMA	H2O
GDM	HEMA
EDMAB	GDM
MEHQ	BISGMA

Table 4. Composition of Biscem®

Paste A	Paste B
Glass filler + Nano silica Acidic monomers TEGMA Self-cure initiator Inhibitors	Glass filler + Nano silica Bis-GMA TEGMA Self-cure initiator Photoinitiator Inhibitors

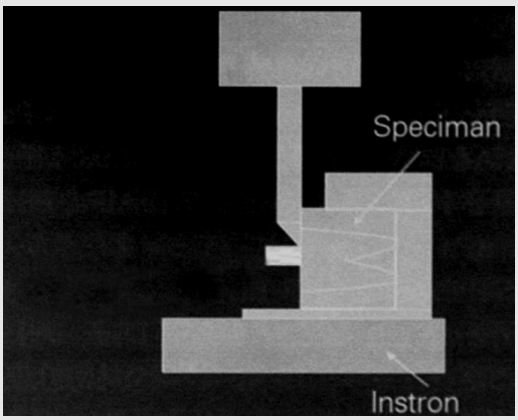

Fig. 2. Cross-sectional view of loading.

5. Statistical Analysis

To examine the shear strength difference depending on the character of groups, T-test, a verification method based on the mean difference, and one-way analysis of variance (One way Anova) were conducted. The empirical analysis of this study was verified at a level of significance of p<.05; for the statistical analysis, the WIN12.0 program was used.

Ⅲ. Test Results

1. Test 1:

U150/U10, M150/M10, and B150/B10 were compared to determine if the groups subjected to light curing 150 seconds later have higher level of adhesion strength than those subjected to light curing 10 seconds later. The following are the test results:

(1) U10 and U150: The mean of U10 was 18.77, and that of U150, 20.48. The difference was not statistically significant (P<.05) (Table 5, Fig. 3).

(2) M10 and M150: The mean of M10 was 9.8, and that of M150, 14.38. A statistically significant difference was noted (p<.05) (Table 6, Fig. 4).

(3) B10 and B150: The mean of B10 was 17.2, and that of B150, 24.34; thus showing a statistically significant difference (P<.05) (Table 7, Fig. 5).

Table 5. T-test between U10 and U150

	N	Mean (Mpa)	SD	T	P-Value
U10	12	18.7727	3.80896	-.848	.406
U150	12	20.4846	5.69354	-.848	.406

*P<.05

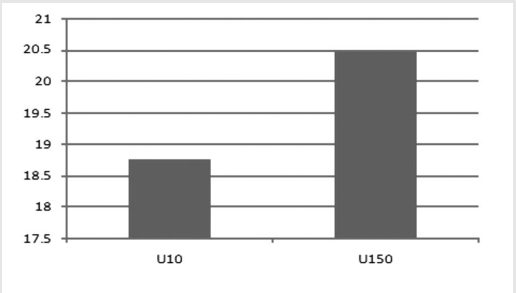

Fig. 3. Comparison between U10 and U150 in terms of shear bonding strength

Table 6. T-test between M10 and M150

	N	Mean (Mpa)	SD	T	P-Value
M10	12	9.8800	4.16701	-2.082	.050
M150	12	14.3833	5.67304	-2.082	.050

*P<.05

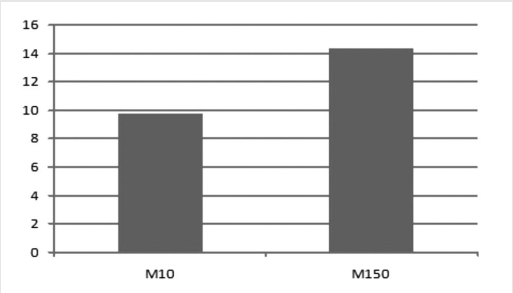

Fig. 4. Comparison between M10 and M150 in terms of shear bonding strength

Table 7. T-test between B10 and B150

	N	Mean (Mpa)	SD	T	P-Value
B10	12	17.2000	7.31528	-2.289	.032
B150	12	24.3417	8.28750	-2.289	.032

*P<.05

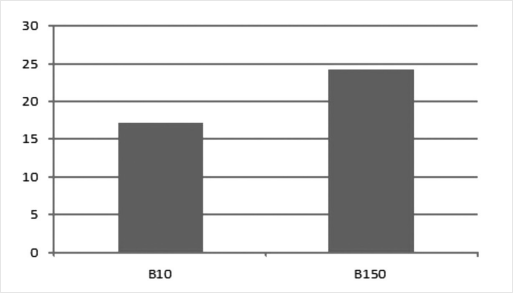

Fig. 5. Comparison between B10 and B150 in terms of shear bonding strength

2. Test 2:

The U10, B10, and M10 groups were compared for bonding strength; ditto for the U150, B150, and M150 groups. Afterward, the respective bonding strength of each brand was compared under the same condition. The following are the test results:

Table 8. Comparison of shear strength among U10, M10, and B10

	N	Mean (Mpa)	SD	F	P-Value	Scheffe
U10	12	18.7727	3.80896	7.740	.002	A B
M10	12	9.8800	4.16701	7.740	.002	A
B10	12	17.2000	7.31528	7.740	.002	A B
Total	36	15.5559	6.54737	7.740	.002	A B

*p←.01

Table 9. Comparison of shear strength among U150, M150, and B150

	N	Mean (Mpa)	SD	F	P-Value	Scheffe
U150	12	20.4846	5.69354	6.871	.003	A B
M150	12	14.3833	5.67304	6.871	.003	A
B150	12	24.3417	8.28750	6.871	.003	A B
Total	36	19.7568	7.64517	6.871	.003	A B

*P←.01

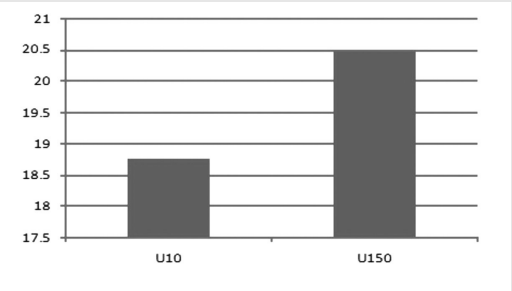

Fig. 6. Comparison of shear bonding strength among U10, M10, and B10

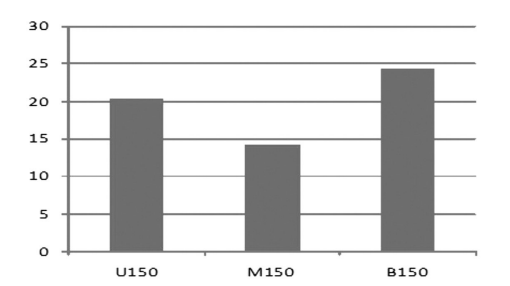

Fig. 7. Comparison of shear bonding strength among U150, M150, and B150

(1) U10, M10, and B10: U10 recorded the highest mean with 18.77 followed by B10 with 17.2 and M10 with 9.88. The difference was statistically significant. In the subsequent verification, U10 and B10 were at a higher level than M10 ($p<.01$) (Table 8, Fig. 6).

(2) U150, M150, and B150: B150 recorded the highest mean with 24.34 followed by U150 with 20.48 and M150 with 14.38. The difference was statistically significant. The subsequent verification revealed that U150 and B150 were at a higher level than M150 ($P<.01$) (Table 9, Fig. 7).

Ⅳ. Analysis

Adhesion dentistry has steadily evolved over the last 40 years. Countless studies on dentin adhesion have been conducted, and consistent progress has been realized up to the present 7th generation. Smear layer has been discovered, and efforts toward its removal ensued[10]. The removal of the smear layer had continued until the 4th generation, and the concept of a hybrid layer was introduced[11]. Since their introduction, the 5th-generation one-bottle system and self-etching primer[12] have been widely used until now. The most recent 6th generation has been introduced, enabling one-solution dentin adhesion[13]. Despite reports of reduced bonding capacity for enamel adhesion[14], the continued development of adhesives has improved user convenience. Likewise, resin cement has made steady progress, thereby enabling a simplified adhesion procedure for users.

minimizing fine leaks and offering aesthetically high color coordination[17-19]. With fragile and brittle ceramic restorations, the selection of a dental adhesion system is

crucial. Especially in this case, resin cement can be very useful. Many studies on the use of resin cement revealed that all ceramic restorations increase fracture resistance[20]. Note, however, that the use of resin cement requires greater skills compared to other adhesion systems. Applying resin cement through wet bonding on water-soluble dentin necessitates advanced technique as well as additional time for treatment[21]. To address these disadvantages, the latest SARC products - which, similar to resin cement itself, allow the simultaneous application of etching, priming, and bonding - have been widely used.

This study conducted 2 tests using 3 types of SARC. The first test was done based on the hypothesis that adhesion strength between the resin block and dentin would differ depending on the timing of light curing. The group subjected to light curing 150 seconds later was expected to exhibit greater bonding strength than the 10-second group, since the bonding strength will develop differently over time because the acid contained in SARC reacts with dentin. Consequently, Maxcem® and Biscem® yielded significant results, whereas Unicem® showed no significant difference. Despite the insignificant difference, however, a somewhat higher strength was noted in Unicem®. In other words, the group subjected to light curing 150 seconds later exhibited greater bonding strength than the 10-second group. Braga, et al found that only when a dual-curing type of resin cement was self-cured did lower bonding strength occur compared to simultaneous light curing[22-24]. In other words, both cases of self-curing and light curing within 10 seconds do not yield a positive result in terms of bonding strength. Good timing is required for light curing for optimal bonding strength.

The other test involved a comparison of bonding strength among 3 types of cement under the condition of same timing of light curing. There has yet to be any clear study on the mechanism through which the acid contained in SARC reacts with dentin. Adhesion is known to occur with dentin; ditto for the current 5th generation self-etching system[25]. For the full processing of dentin through conventional self-etching, the decalcification and hybridization of dentin have been observed. With SARC, however, no such phenomena have been noted[26]. The bonding strength of resin cement has been determined by the quality of the hybrid layer generated through the pretreatment of dentin. If this layer is generated in porous form, water molecules permeate; the subsequent hydrolysis then causes reduced bonding strength[27]. With SARC, however, the priming step for the pre-treatment of dentin is skipped; hence the relatively little change in bonding strength compared to other resin systems. This test result showed standard deviation of 5-7 Mpa, which is lower than the test result of De Munck[28], et al (9-13 Mpa) with the use of Panavia-F® and Rely- X®. For this test result, Unicem¢Á exhibited the highest bonding strength among the groups subjected to light curing 10 seconds later. Among groups subjected to light curing 150 seconds later, however, Biscem® showed the highest strength. With Unicem®, no significant difference was observed between the 10-second group and the 150-second group. Both groups showed adequate level of bonding strength. Thus, it can be said to be a convenient material for clinical use. With Biscem®, a somewhat significant difference was observed in the 150-second group than the 10-second group. Both groups showed decent or higher level of bonding strength, however. With Maxcem®, the 10-second group yielded a significantly low result compared to other groups. Therefore, the clinical use of Maxcem® requires sufficient time for light curing.

This study used resin blocks with diameter of 3 mm. Note, however, that the use of resin blocks with minimum diameter could have yielded more precise results since the distribution of dentinal tubules in dentin differs by tooth. Furthermore, since the teeth used in this test were extracted from different people at different times, the tooth condition varied by tooth. As such, precisely predicting the result was difficult. Such weakness can be addressed through a study that compares bonding strength using resin blocks with smaller diameter and adhering multiple resin blocks onto one tooth. Finally, using a bigger number of test samples at 30-second intervals will most likely yield the most clinically efficient timing for light curing.

Ⅴ. Conclusion

With Unicem®, the different timing of light curing did not have a significant impact; it generally showed overall good bonding strength. With Maxcem® and Biscem®, however, a 150-second lapse following cement application yielded a clinically better result in terms of adhesion strength. In other words, Unicem® requires no clinical consideration in terms of the timing of light curing. With Biscem® and Maxcem®, allowing 150 seconds before light curing is recommended

for better results. Note, however, that Maxcem® showed a generally low level of bonding strength. In particular, light curing within 10 seconds resulted in inadequate bonding strength. Therefore, special attention is required in clinical use.

References

1. Ferrari M, Goracci G, Garcia-Godoy F. Bonding mechanism of three "one-bottle" systems to conditioned and unconditioned enamel and dentin. Am J Dent 1997;10(5):224-230
2. Fabianelli A, Vichi A, Kugel G,Ferrari M. Influence of self etching priming bonding systems on sealing ability of Class II restorations: leakage and SEM evaluation. Paper presented at annual meeting of the International Association for Dental Research; April 6, 2000; Washington, D.C.
3. Han L, Okamoto A, Fukushima M, Okiji T. Evaluation of physical properties and surface degradation of self-adhesive resin cements. Dent Mater J 2007;26(6):906-914
4. Gerth HU, Dammaschke T, Züchner H, Schäfer E. Chemical analysis and bonding reaction of RelyX Unicem and Bifix composites—a comparative study. Dent Mater 2006;22(10):934-941.
5. Komine F, Tomic M, Gerds T and Strub JR. Influence of different adhesive resin cements on the fracture strength of aluminium oxide ceramic posterior crowns. J Prosthet Dent 2004;92(4):359-364
6. Burke FJ, Fleming GT, Abbas G, Richter B. Effectiveness of a self-adhesive resin luting system on fracture resistance of teeth restored with dentin-bonded crowns. Eur J Prosthodont Restor Dent 2006;14(4):185-188
7. Piwowarczyk A, Lauer HC, Sorensen JA. In vitro shear bond strength of agents to fixed prosthodontic restorative materials. J Prosthet Dent 2004;92(3):265-273
8. Behr M, Rosentritt M, Regnet T, Lang R, Handel G. Marginal adaptation in dentin of a self-adhesive universal resin cement compared with well-tried systems. Dent Mater 2004;20(2):191?197.
9. Fabianelli A, Goracci C, Bertelli E, Monticelli F, Grandini S and Ferrari M, In vitro evaluation of wall-to-wall adaptation of a self-adhesive resin cement used for luting gold and ceramic inlays. J. Adhes Dent 2005;7(1):33-40
10. Khdemat S, Shockouhinejad N. Comparison of the efficacy of three chelating agents in smear layer removal. J Endod;200834(5):599-602
11. Nakabayashi N, Kojima K, Masuhara E. The promotion of adhesion by the infiltration of monomers into tooth states. J. Biomed Mat Res 1982;16(3):265-273
12. Kaneshiro AV, Imazato S, Ebisu S, Tanaka S, Tanaka Y, Sano H. Effects of a self-etching resin coating system to prevent demineralization of root surfaces. Dent Mater 2008;11[Epub ahead of print]
13. Blunck U, Zaslansky P. Effectiveness of all-in-one adhesive systems tested by thermocycling following short and long-term water storage. J Adshes Dent 2007;9(2):231-240
14. De Munck J, Vargas M, Van Landuy K , Hikita K, Lambrechts P, Van Meerbeek B. Bonding of an auto-adhesive luting material to enamel and dentin. Dental Mater 2004; 20(10):963-971
15. Shortall AC, Fayyad MA, Williams JD, Marginal seal of injection molded ceramic crowns cemented with three adhesive systems, J Prosthet Dent 1989;61(1):24-27
16. White SN, Sorensen JA, Kang SK, Caputo AA. Microleakage of new crown and fixed partial denture luting agents, J Prosthet Dent 1992;67(2):156?161
17. DeLong R, Pintado M, Douglas WH. Measurement of change in surface contour by computer graphics. Dent Mater 1985;1(1):27?30
18. Pintado MR, Conry JP, Douglas WH. Measurement of sealant volume in vivo using image-processing technology. Quintessence Int.1988;19(9):613-617
19. Li JC and White SN. Mechanical properties of dental luting cements. J. Prosthet Dent 1999;597-609
20. Rosenstiel SF, Land MR, Crispin JB. Dental luting agents: a review of the current literature . J Prosthet Dent 1998; 280 -301
21. Yoshida Y, Van Meerbeek B, Nakayama Y, Snauwaert J, Hellemans L, Lambrechts P, Manherle G, Wakasa K. Evidence of chemical bonding at biomaterial-hard tissue interfaces. J Dent Res 2000;79(2):709-714
22. Braga RR, Cesar PF, Gonzaga CC. Mechanical properties of resin cements with different activation modes. J Oral Rehabil 2002;29(3): 257-262
23. Darr AH, Jacobsen PH. Conversion of dual cure luting cements. J Oral Rehabil 1995;22(1):43-47
24. Rueggeberg FA, Caughman WF. The influence of light exposure of polymerisation of dual-cure resin cements. Oper Dent 1993;18(2):48-55
25. Donmez N, Belli S, Pashley DH, Tay FR. Ultrastructural correlates of in vivo/in vitro bond degradation in self-etch adhesives. J Dent Res 2005;84(4):355-359
26. Pashley DH, Ciucchi B, Sano H, Carvalho RM . Bond strength versus dentin structure: a modeling approach. Arch Oral Biol 1995;40(12):1109-1118
27. Tay FR, Pashley DH, Suh BI, Carvalho RM, Itthagarun A. Single-step adhesives are permeable membranes. J. Dent 2002;30(7-8):371-382
28. De Munck J, Vargas M, Van Landuy K , Hikita K, Lambrechts P, Van Meerbeek B. Bonding of an auto-adhesive luting material to enamel and dentin. Dental Mater 2004; 20(10):963-971