MICROLEAKAGE AND WATER STABILITY OF RESIN CEMENTS

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Statement of Problem : Recently, resin cements have become more widely used and have been accepted as prominent luting cements. Current resin cements exhibit less microleakage than conventional luting cements. However, the constant contact with water and exposure to occlusal forces increase microleakage even in resin cements inevitably.
Most bonding resins have been modified to contain a hydrophilic resin such as 2-hydroxyethylmethacrylate (HEMA) to overcome some of the problems associated with the hydrophobic nature of bonding resins. By virtue of these modifications, bonding resins absorb a significant amount of water, and there may also be significant stresses at bonding interfaces, which may adversely affect the longevity of restorations. Therefore the reinforcement of water stability of resin cement is indispensable in future study.
Purpose : This study was conducted to examine the influence of water retention on microleakage of two resin cements over the period of 6 months.
Materials and Methods : 32 extracted human teeth were used to test the microleakage of a single full veneer crown. Two resin cements with different components and adhesive properties - Panavia F (Kuraray Co., Osaka, Japan) and Super-Bond C&B (Sun Medical Co., Kyoto, Japan)- were investigated. The storage medium was the physiological saline solution changed every week for 1 month, 3 months, and 6 months. One group was tested after storage for 1 day.
At the end of the each storage period, all specimens were exposed to thermocycling from 5° C to 55° C of 500 cycles and chewing simulation of 50,000 cycles, and then stained with 50% silver nitrate solution. The linear penetration of microleakage was measured using a stereoscopic microscope at $\times 40$ magnification and a digital traveling micrometer with an accuracy of ± 3 ㎛.
Values were analyzed using two-way ANOVA test, Duncan's multiple range tests (DMRT).
Results :
Statistically significant difference of microleakage was shown in the 3-month group compared with the 1-day or 1-month group in both systems ($p<0.05$) and there were statistically significant differences in microleakage between the 3-month group and the 6-month group in both systems ($p<0.05$).
The two systems showed different tendency in the course of increased microleakage during 3 months. In Panavia F, microleakage increased slowly throughout the periods. In Super-Bond C&B, there was no significant increase of microleakage for 1 month, but there was statistically significant increase of microleakage for the next 2 months. For the mean microleakage for each period, in the 3-month group, microleakage of Super-Bond C&B was significantly greater than that of Panavia F. On the other hand, in the 6-month group, microleakage of Panavia F was significantly greater than that of Super-Bond C&B ($p<0.05$).
Conclusion : Within the limitation of this study, water retention of two different bonding systems influence microleakage of resin cements. Further studies with the longer observation periods in viro are required in order to investigate water stability and the bonding durability of the resin cement.

CLINICAL IMPLICATIONS

Microleakage at the Cement-tooth interfaces did not necessarily result in the failure of the crowns. But it is considered to be a major factor influening the longerity of restorations. Further clinical approaches for decreasing the amount of microleakage are required.

Key Words
Resin cement, Microleakage, Water stability, Bonding interfaces, Thermocycling

The clinical longevity of a full veneer crown, which was restored in a suitable occlusal relationship and had an acceptable fit and marginal adaptation, is inseparably related to the constancy of luting cement. Cement loss is the most important cause of recurrent caries or tooth fracture.

Microleakage is defined as the clinically undetectable passage (gap) of bacteria, fluid, molecules, or ions between enamel or dentin and the restorative material applied to it.[1] Microleakage at the tooth/restoration interfaces is considered to be a major factor influencing the longevity of dental restorations. It may lead to staining at the margins of restorations, hastening of the breakdown at the marginal areas of the restorations, recurrent caries at the tooth/restoration interface, hypersensitivity of restored teeth, and the development of pulpal pathology.[2]

Recently, resin cements have become more widely used and have been accepted as predominant luting cements. Current resin cements exhibit less microleakage than conventional luting cements.[3-5] The constant contact with water and exposure to occlusal forces increase microleakage in resin cements inevitably. To overcome some of the problems associated with the hydrophobic nature of bonding resins when bonding to the intrinsically wet dentin surface, recent bonding resins have been modified to contain both hydrophilic and hydrophobic moieties, or a hydrophilic resin such as 2-hydroxyethylmethacrylate (HEMA) has been added. HEMA enhanced the penetration capability of bonding resin and facilitated the formation of a hybrid layer.[6] By virtue of these modifications, bonding resins absorb a significant amount of water, which may help reduce small gaps between the resin and dentin. In addition, swelling of the resin may reduce some of the stresses induced during polymerization. Conversely, there may also be significant stresses placed at the tooth-resin interfaces, which may adversely affect the longevity of restorations.[7] Therefore, the reinforcement of water stability of resin cement is indispensable in future study.

In this study, single crowns made on natural teeth were cemented with two resin cements and stored in the physiological saline solution for the scheduled period. Then the specimens were exposed to thermocycling and chewing simulation, and were stained for measuring of microleakage. The purpose of this study was to examine the effect of water retention on microleakage and to investigate water stability of resin cements.

MATERIALS AND METHODS

32 extracted human third molars, of comparable crown length, were selected. The teeth were prepared to receive a complete cast gold crown with a medium round-end tapered diamond bur (No. 767.9 Espe-Premier, Norristown, Pa, USA) at high speed, cooled with an air/water spray. The occlusogingival length was approximately 5mm and all margins were placed above the cementoenamel junction. A paralleling device (F2, Degussa Korea Inc, Seoul, Korea) was used to prepare the axial walls of these teeth to ensure a consistent degree of taper, a 6-degree convergence angle and the chamfer finish line.

Putty-wash impressions of the finished preparations were made with a polyvinyl siloxane material (Express, 3M Dental Products, St, Paul, Mn, USA). High-strength stone (Die Keen, Columbus Dental, St. Louis, Mo, USA) was then poured into the impressions to construct the dies. The dies were trimmed by the use of a x10 power microscope and coated with three layers of die spacer (YETI, Engen, Germany) to approximately 1mm from the finish line, as suggested by the manufacturer.

Copings were formed with a wax dipping technique and the margins were refined with a cervical wax (Plastodent, Degussa, Germany) under x10 magnification. The patterns were sprued, invested with a gypsum-bonded investment material (Cristobalite, Whip Mix Corp., Louisville, Ky, USA) and cast with type II gold alloy (INLAY MEDIUM, Heesung Engelhard, Seoul, Korea). The castings were divested and the internal positive defects were removed with a half-round bur under x10 magnification. The castings were finished, polished and then adapted to the dies. The seating interferences were identified when the castings were seated on dies coated with a thin layer of die lubricant. The castings were fitted to their natural teeth with a silicone disclosing medium (Fit-checker, GC International, Tokyo, Japan). The internal surfaces of the castings were air abraded with 50μm aluminum oxide at 40 lb/in^2.

To assess the fit of the castings before cementation, the castings were placed on the natural teeth and marginal opening was measured at four predetermined reference marks on each casting, which had been placed in the wax patterns at the midfacial, lingual, mesial, and distal surface of wax coping. Marginal opening was determined as the vertical space between the finish line and the most apical end of the casting margin. Three independent measurements were made at each point. The measurements were made at x50 magnification by the use of a digital traveling micrometer (Mitutoyo Mfg Co. Ltd., Tokyo, Japan). The teeth and the castings were separated and cleaned again.

Two bonding systems were evaluated in this study: Panavia F, Super-Bond C&B (Table I)

32 teeth were divided randomly into the two groups and bonded with one of the bonding systems following manufacturer's instructions (Table II).

After seating, the specimens were placed under a constant axial force of 5kg in a paralleling jig for 10 minutes.[8] The specimens in each bonding group were randomly assigned to four sub-groups and kept in physiological saline solution at 37℃ for 24 hours, 1 month, 3 months and 6 months (Group A, B, C, D). The storage solution was stored at room temperature before use and heated to 37℃ prior to changing it

Table I. Materials, manufacturers, and compositions of two bonding systems

	Panavia F	Super-Bond C&B
Etchant	Self-etching primer MDP, HEMA, 5-NMSA, sodium benzene sulfinate, N,	(for dentin) Citric acid(10%) $FeCl_3$ (3%)
Primer	N-Diethanol p-toluidine	Monomer: 4-META, MMA
Resin	Bis-phenol A polyethoxy dimethacrylate, MDP, hydrophobic dimethacrylate, hydrophilic dimethacrylate, BPO, silanated barium glass powder, silanated silica powder, surface treated sodium fluoride, sodiuim aromatic sulfinate, N,N-Diethanol p-toluidine, photo sensitizer	Polymer: PMMA Catalyst : TBB, acetone
Manufacturer	Kuraray Co., Ltd, Osaka, Japan	Sun Medical, Co., Ltd, Nagoka, Japan

MDP=10-Methacryloyloxydecyl dihydrogen phosphate
HEMA=2-Hydroxyethyl methacrylate
5-NMSA=N-Methacryloyl 5-aminosalicylic acid
BPO=Benzoyl peroxide
TBB=Tri-n-butyl borane

Table II. Bonding Procedures

	Bonding procedure
Panavia F	Self-etching primer : Apply ED primer to tooth surface. Leave undisturbed for 60 seconds. Air dry mildly.
	Bonding : (If the restoration is precious metal, apply ALLOY PRIMER to the inside of restoration) Mix the Paste A and Paste B for 20 seconds. Apply the mixed paste to the restoration and cement to the tooth. Apply visible light for 20 seconds or apply OXYGUARD II for 3minutes.
Super-Bond C&B	Etching : Etch the tooth surface 5-10 seconds, wash and dry (red activator for dentin)
	Bonding : (If the restoration is precious metal, apply V-PRIMER to the inside of restoration) Prepare activated liquid (mix the monomers and catalyst at the ratio of 4/1). Mix polymer powder with the activated liquid. Apply the mixed paste to the restoration and cement to the tooth for 8-10minutes.

every week.

At the end of each storage period, the specimens were thermocycled from 5℃ to 55℃ of 500 cycles, with 15 seconds of dwelling at each temperature and no intermediate pause (Fig. 1). After thermocycling, the specimens were exposed to chewing simulation of 50,000 cycles by chewing simulator (MTS 858 Mini Bionix II system, MTS systems corp., Minn. USA. Fig. 2). The axial force was in the range of 6-100 N and lateral excursion was 0.4mm at a frequency of 2Hz.

The specimens were then treated with a silver nitrate stain. The specimens were placed in a 50% silver nitrate solution for 24 hours in darkness. Thereafter, they were rinsed in running water for 5 minutes, immersed in photo-developing solution and exposed to a fluorescent light for 8 hours to reduce silver ions to metallic silver. After rinsed with water for 1minute, the specimens were sectioned parallel to the long axis of the tooth buccolingually and mesiodistally using a microtome saw (Isomet, Buehler Ltd., Evanston, Il, USA). This created two interfaces for each point (eight separate interfaces per specimen) for measuring microleakage. All the cut surfaces were polished with increasingly wet silicone carbide abrasive paper up to 2400 grit, using running water as a lubricant.

The linear penetration of silver nitrate stain, from the external margin of cement where the cement interfaced with the tooth, was measured by the use of a stereoscopic microscope (MZ 6, Leica AG, Frankfurt, Germany) and a digital traveling micrometer (Mitutoyo Mfg Co. Ltd., Tokyo, Japan) with an accuracy of $\pm 3\mu m$. Three independent measurements were made by two different researchers on each interface. Eight points were on each specimen, with four specimens per group and total 32 points per group were yielded.

The measuring method of microleakge was as the following. In Fig. 3, two lengths, L_1 and L_2, are given. Theses two lengths (L_1 and L_2) were chosen by the eye to represent two straight lines for the best fit for the curved microleakage path. The sum of these lengths represents the extent of the microleakage.[9]

The mean marginal opening of the uncemented specimens was calculated to ensure that the specimens were within the range of clinical acceptability.[10]

The mean of microleakage was calculated for

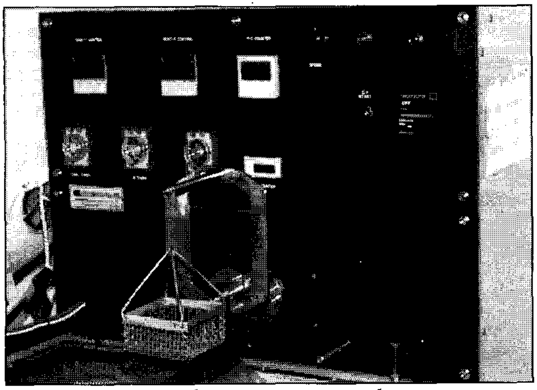

Fig. 1. Thermocycling machine.

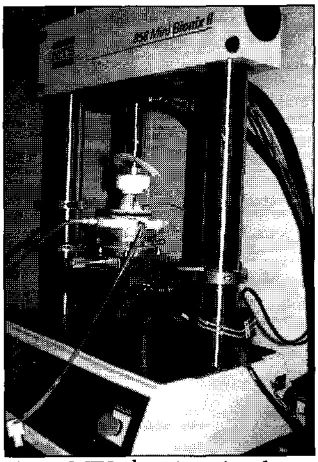

Fig. 2. MTS chewing simulator.

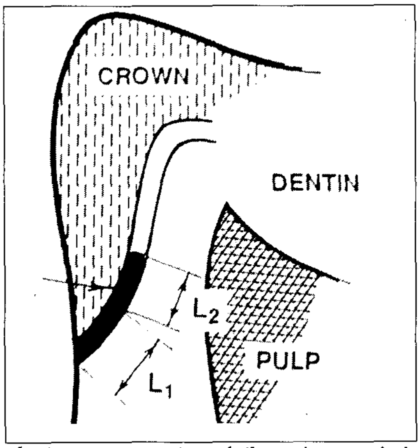

Fig. 3. The linear penetration of silver nitrate stain in measuring of microleakage.

each cement and storage group. Following this, a two-way ANOVA was used to search for statistically significant differences. The groups were subjected to multiple comparisons using Duncan's multiple range tests at $P<0.05$ as a level of significance.

RESULTS

The mean marginal opening of the uncemented crowns was 58.4㎛(±14.3㎛) and the specimens were clinically acceptable.[10]

The measurement points from different sites within crowns could be treated as independent and all points for each group could be pooled. Some studies provide the evidence to support the independence of measurement sites.[11-13]

Microleakage was only observed at the tooth-cement interfaces (Fig. 4). The results coinsided with those of other studies.[12,13] It seemed that the microblasting with 50㎛ alumina powder and the use of the metal primer were adequate to prevent microleakage at the metal-cement interfaces.

In this study, the mean microleakage of Panavia F was 240, 270, 351, 1008㎛ and that of Super-Bond C&B was 277, 343, 696, 825㎛ for each group.

The results of Two-way ANOVA test are presented in Table Ⅲ.

As the results of Two-way ANOVA analysis, there were statistically significant differences in all factors. All treatments, including the storage period and bonding system, had significant influences on the results. Especially there was a significant interaction effect between the storage period and bonding system at $p<0.01$ level.

The results of multiple comparisons using Duncan's multiple range tests are presented in Table Ⅳ and Fig. 5.

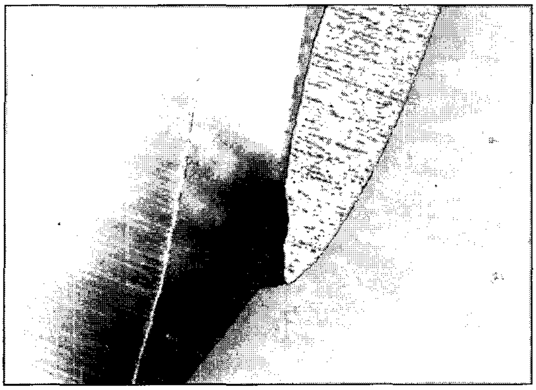

Fig. 4. Microleakage at the tooth-cement interface with silver nitrate stain(×40).

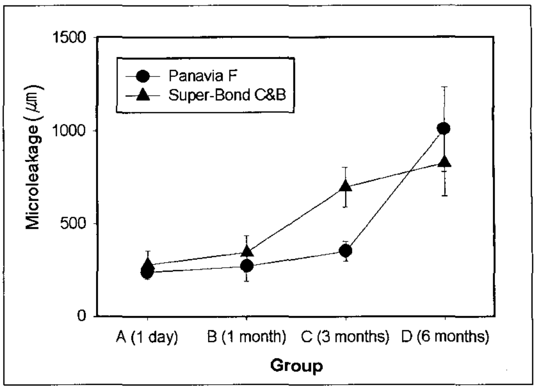

Fig. 5. Mean microleakage (㎛).

Table III. The results of Two-way ANOVA test

Source of variations	Degrees of freedom	Sum of squares	Mean square	Fs
Treatments	7	9.906	1.415	
Storage period	3	8.635	2.878	190.313*
Bonding system	1	0.147	0.147	9.719*
Interaction	3	1.123	0.374	24.749*
Error	120	1.815	0.015	
Total	127	32.165		

* : There were statistically significant differences between the groups (P<0.01).

Table IV. Mean microleakage(㎛)

Bonding agent	Group			
	A (1 day)	B (1 month)	C (3 months)**	D (6 months)**
Panavia F	240±39a*	270±80ab	351±52b	1008±229c
Super-Bond C&B	277±77a	343±92a	696±107b	825±178c

* : a,b,c values with the same letter are not significantly different at p>0.05

** : There were statistically significant differences in microleakage between the bonding systems in same group(p<0.05).

As the results of the Duncan's multiple range tests (p<0.05), in Panavia F, there were statistically significant differences in microleakage between group A and C, and between group B and D. There were statistically significant differences in microleakage between group D and all other groups. On the other hand, there was no statistically significant difference in microleakage between group A and B, and between group B and C. In Super-Bond C&B, there were statistically significant differences in microleakage between group B and C, and between group C and D.

In group C and D, there were statistically significant differences in the mean microleakage for two systems within the same group (P<0.05). In the group C, microleakage of Super-Bond C&B was significantly greater than that of Panavina F. On the other hand, in the group D, microleakage of Panavia F was significantly greater than that of Super-Bond C&B.

DISCUSSION

Current resin cements exhibit less microleakage than conventional luting cements.[3-5] Panavia EX showed 202㎛ (±43㎛) and Flecks Zinc cement did 1475㎛ (±110㎛) in *in vitro* microleakage test. Panavia EX was the former type of Panavia F and Flecks Zinc cement was in the family of zinc phosphate cement and the specimens were stored for 14 days in 37℃ water and exposed to thermocycling of 1,500 times.[5] In this study, microleakage of Panavia F was 240, 270, 351, 1008㎛ and that of Super-Bond C&B was 277, 343, 696, 825 ㎛ for each storage period.

Many researchers have investigated the influences of thermal and mechanical cycles on the microleakage. Thermal and mechanical stresses are commonly used to mimic oral aging processes. Thermocycling is carried out to evaluate the effect of disparities in the coefficients of thermal expansion between the restoration and the tooth structure. In *in vitro* microleakage test, the restored teeth should be subjected to both thermal and occlusal stresses for more close simulation of oral conditions.[14] But neither the number of thermal and mechanical cyclings nor the load employed has been standardized yet.

Most microleakage tests had a tendency to measure the leakage score. In this study, direct linear measurement was used, and the linear penetration of silver nitrate stain from the external margin of cement was measured.[9]

Kitasako *et al.* presented that the storage condition influenced the long-term durability of dentin bonding with resin cements.[15] The shear bond strengths in the daily changed storage solution groups were significantly lower than those where the storage solution remained unchanged (p<0.05). The storage solution was changed every week in this study and the change of solution may have accelerated hydrolysis at the resin-dentin interfaces.

There was statistically significant difference in microleakage of group C and D compared with group A or B in both systems of this study. It means that water retention for 3 months influenced the increase of microleakage. And in the course of increased microleakage during 3 months, the two systems showed different tendency. In Panavia F, for the first 3 months, microleakage increased slowly throughout the periods. In Super-Bond C&B, there was no significant increase of microleakage for 1 month, but for the next 2 months, there was statistically significant increase of microleakage. These results of microleakage might be related to the water absorption at the resin-dentin interfaces.

Slow water absorption by the bonded constituents may lead to degradation of the bonding strengths due to the plasticizing effects of water on bonding resin and collagen fibrils, and may cause the hydrolysis of those at the base of the hybrid layer.[16] Nakabayashi *et al.* have determined that composite restoratives bonded to human dentin in vivo might be subject to failure in a zone of exposed collagen, which occurred between the hybrid layer and the underlying normal dentin.[17]

For the target region of hydrolysis, Gendusa reported that the hybrid layer did not degrade in water.[18] On the other hand, the loss of resin was observed in the spaces between the collagen fibrils in the resin-dentin interfaces after 1 year in another study. They reported that the hybrid layer would be a weak link and be susceptible to hydrolytic attack.[19]

Dentin bonding durability has been investigated in relation to the change of mechanical properties of cement due to water retention. Diaz-Arnold *et al.* reported that the different bonding systems and the storage period (2 days and 30 days at 37℃) influenced the change of the bonding strengths.[20] Gwinnett and Yu found the significant loss of initial bonding strengths after water retention for 6 months.[21] For the more long-term storage and bonding strengths, Burrow *et al.* reported that after 3 years of water retention, the initial bonding strength obtained for primed specimens had decreased markedly, being almost same as that of the nonprimed group.[16] On the other hand, Miears *et al.*, from the two groups stored for 1 day and for 3 months in 37℃ water, concluded that time had no significant effect for bonding strengths.[22]

Sano *et al.* also reported Super-Bond C&B had the significantly higher microleakage scores than other bonding systems after storage in water at 37℃ for 24 hours.[23] In this study, Microleakage of Super-Bond C&B after storage for 3 months was greater than that of Panavia F in group A, B and C, though the statistically significant difference was shown only in group C. The structural characteristics and its plastic deformation under the influence of water solubility and bonding durability might lead to the increase of microleakage of Super-Bond C&B for the first 3 months in this study.

The polymerized Super-Bond C&B consists of linear polymers of MMA without inorganic fillers and it has a microhardness and flexural modulus substantially lower than those of composite resin cements. Because of its low modulus of elasticity, it displayed high plastic deformation. White and Yu reported that Super-Bond C&B underwent considerable plastic deformation, which did not allow this material to demonstrate the compressive strength recommended by established specifications for cement.[24]

Yoshida *et al.* reported than there was no significant difference in the solubility of Panavia 21 and Super-Bond C&B.[25] Panavia 21 was the former type of Panavia F. Also, the bonding strength of Super-Bond C&B to extracted bovine teeth was not significantly different from that of other bonding systems.[17] However, water solubility and the bonding strength might have influenced the results of this study.

Panavia F contains 78 wt% filler contents. In this study, microleakage of Panavia F after storage for 3 months was more smaller than that of Super-Bond C&B in group A, B, and C. Water absorption in bonding resin is closely related to the filler content. Microleakage of Panavia F for the first 3 months might be influenced by this characteristic in this study. Kitasako *et al.* reported that Protect Liner F system absorbed the least amount of water and stabilized rapidly. They concluded that this was most likely due to the filler particles present (30% w/w), reducing the amount of resin matrix and absorbed water.[15]

Another reason for smaller microleakage for the first 3 months in Panavia F may be related to the improvement in the bonding strength. Panavia F is composed of a self-etching primer and a bonding resin. Panavia F produces mild demineralization of the dentin surface.[26] The thinness of the demineralization was reported to provide excellent resin penetration of the dentin surface and higher bonding strengths.[25,27]

On the other hand, microleakage of Panavia F of group D was significantly greater than that of Super-Bond C&B. This means that microleakage of Panavia F significantly increased for the late 3 months, and several factors may have related to it.

Burrow *et al.* reported that bonding resins containing hydrophilic constituent like as HEMA, MDP, PENTA (dipentaerythritol pentacrylate phosphoric acid ester) showed a rapid water absorption of a significant amount.[7] Besides, Sano *et al.* reported that the silane coupling seemed to be the weakest link between the filler particles and resin matrix in the presence of water. This could provide more spaces where water could easily infiltrate throughout the resin cement.[19] Also, bonding resins contain less filler particles than resin materials in order to penetrate to dentin. The degree of water absorption has been shown to be much less for resin materials compared with bonding resins, that is, the quantity of water absorbed by resin materials tended to be less than 1% (w/w) compared with approximately 4%(w/w) for the bonding resin.[7] Therefore, a possibility may exist for stresses to occur at the interfaces and weakening of the bonding due to a difference in dimensional change may possibly affect the longevity of a restoration.

Several methods for decreasing the amount of microleakage were suggested. Microleakage at the cement-tooth interfaces did not necessarily result in the failure of the crowns. Zinc phosphate cement, in a considerable range of microleakage (432±353 ㎛), could successfully serve as a luting cement for 20 years in clinical situations.[9] Gwinnett and Kanca reported that the gap formation in the restorative interfaces could not be occurred when the current dentin bonding systems were used.[28] Van Meerbeek *et al.*[29] have suggested the formation of an elastic bonding layer. The application of low viscosity resin in a sufficient thickness was used to relieve the polymerization contraction stresses and such a flexible intermediate resin layer may better transmit and distribute stresses induced by thermal changes and water absorption.[30] In clinical situations, a slightly viscous resin may be more easily applied in a constant thickness and it will exhibit a more even contraction and expansion during the polymerization and water uptake phases. The techniques that the pooling of the resin can be avoided, such as gentle air thinning, may assist in providing a more even thickness of the resin. Further studies with the longer observation periods in vivo are required in order to investigate water stability and the bonding durability of the resin cement.

CONCLUSION

Within the limitation of this study, water retention of different bonding systems influence microleakage of resin cements.

1. Statistically significant differences of microleakage was shown in the 3-month group compared with the 1-day or 1-month group in both systems ($p<0.05$).
2. There were statistically significant differences of microleakage between the 3-month group and the 6-month group in both systems ($p<0.05$).
3. The two systems showed different tendency in the course of increased microleakage during 3 months. In Panavia F, microleakage increased slowly throughout the periods. In Super-Bond C&B, there was no significant increase of microleakage for 1 month, but for the next 2 months, there was statistically significant increase of microleakage.
4. For the mean microleakage for each period, Super-Bond C&B showed significantly greater microleakage than Panavia F in the 3-month group. On the other hand, in the 6-month group, Panavia F showed significantly greater microleakage than Super-Bond C&B ($p<0.05$).

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