

In Vitro Effects of Different Moisture Level and Curing Method on Microleakage of Resin Cements to Pulp Chamber Dentine

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Abstract - This study evaluated the microleakage of resin cements in the pulp chamber dentin. Fifty specimens of sound human molars were divided into five groups. Composite cores cemented using Clearfil SA Luting in the first group to a dried dentin and in the second group to a moistened dentin and then light-cured. In third and fourth groups, cement was placed on dried and moistened dentin and self-cured respectively. In fifth group, composite cores were cemented by Panavia F2.0. After thermocycling, microleakage was evaluated using fluid filtration technique. The highest microleakage mean value was observed in the group with light-cured to a dry dentin. The mode of curing in contrast to moisture value had significant effect on microleakage. The microleakage of self-adhesive resin cement used in this study was lower in case of self-cured than in case of light-cured and was not related to the dentin surface moisture.

KEY WORDS: Composite core, Microleakage, Resin cement, Curing mode, Moisture

INTRODUCTION

Coronal sealing is just as important as apical sealing for successful endodontic treatment. To overcome the shrinkage of composite resin during polymerization and side effects such as microleakage, one approach is to make indirect composite restorations and cement them using a variety of resin cements¹⁻⁴. For indirect tooth colored restorations, the cement used for bonding acts as adurable barrier preventing coronal microleakage. This is a fundamental step in inhibition of bacterial invasion, secondary caries and decementation⁵. In contrast to conventional resin cements, self-adhesive resin cements (SARCs) can be used in a single step without any pretreatment before use⁴. To establish adhesion, SARCs rely not only on the intrinsic dentinal water, but also on water remaining on the surface of the dentin after rinsing. Chemical formulations and initiator mechanisms of each SARCs also affects the polymerization efficacy of resin bonding⁶⁻⁸. A previous report indicated that adhesion of an SARC was compromised on air-dried dentine surfaces and extra moisture resulted in better adhesion⁹; however, few other studies have investigated the effect of dentine moisture on the microleakage of other SARCs particularly bonding of resin cements to the pulp chamber walls. The SARCs are considered to be dual-cured resin cements in which both light-activating and chemical-activating mechanisms are provided. The degree of conversion of resin cements may increase in the light activation mode compared to with chemical-activation

alone, enhancing physical properties¹⁰. Polymerization contraction of resin luting materials is known to produce high stresses in the interface, particularly under the high C-factor of a prepared cavity. With chemical activation alone, resin cements produced a uniform polymerization, lower shrinkage, and lower stress rates than the light-cured resins, particularly at the bottom of deep cavities where access to curing light is limited¹¹. The efficiency of chemical activation without photo-irradiation of the resin cements has been inconstant among different resin cements, with some products revealing poor polymerization and reduced adhesion when no light was applied¹²⁻¹⁵. The aim of this study was to evaluate the microleakage of resin cements in the pulp chamber dentine under different moisture conditions and curing methods. The null hypothesis of the study was that there was no difference in the leakage of resin composite core restorations that were cemented with resin cements under various curing methods and moisture level in pulp chamber dentine.

MATERIALS AND METHODS

Fifty human healthy, caries-free molar teeth were selected after the individuals' informed consent was obtained under the recommendations of the Ethics Committee of Mashhad University of Medical Sciences (IRB approval code; 900165/2011). Teeth were disinfected by storage in 0.02% thymol solution for 24 hours prior to the experiment. The coronal portion of teeth above the cervical third and roots up to 1-2 mm below the furcation were removed with a low-speed diamond saw under water cooling and then the pulp chamber roof was removed and standard diverged access cavities (3H × 3W × 4L mm) were prepared by cutting the pulp chamber roof and walls, without touching the pulpal floor. The coronal dentine was further reduced by 400-grit silicon carbide (SiC) paper to reach a depth of

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around 4 mm for exposed occlusal surface (from top to pulp chamber floor). The root canals were widened by the Gates-Gliden drill (# 2 & 3), washed carefully with normal saline and sealed by Gutta-percha (Dia Dent, Choongchong Buk DO, Korea) without using any type of sealer (Figure 1). Teeth were randomly divided into five groups of ten and the composite core build-up procedures were performed for each sample by the indirect method using composite resin; A2 shade, Clearfil AP-X. Hydroxyl Ethyl Cellulose (1%, Wako Pure Chemical Industries, Osaka, Japan) applied on the cavity surfaces as a spacer, before composite placement. The composite cores were light-cured from the occlusal aspect by Optilux 501 (700 mW/cm²; SDS Kerr, Danbury, CT, USA) for 40 seconds. Cores were then removed from the prepared teeth and further polymerized for 7 minutes at 125° C using a Coltene D.I.-500 oven (Coltene AG, Alstatten, Switzerland) specified for resin composite. For core bonding, the internal surface of composite cores was sandblasted with 50 μ alumina powder at 2 bar pressure for 7 seconds, followed by etching by application of 37% phosphoric acid (3M, St, Paul, MN, USA) for 60 seconds, rinsing for 30 seconds, and drying. Clearfil SE Bond primer was mixed with Clearfil porcelain bond activator (Kuraray Co. Ltd.) in a 1:1 ratio and applied to the internal surface of the composite cores with a brush and allowed to dry for 30 seconds. In the first group, the cavity was completely air dried from 2 cm above using a three-way air syringe at 3 kg/cm² for 20 seconds, leaving the dentine surface dry, with no visible moisture. Clearfil SA luting cement (A2, Kuraray Noritake Dental, Tokyo, Japan) was then mixed on glass slabs for 10 seconds, placed into the cavity and on the adhesive surface of the core and inserted gently inside the cavity. The cement was then light-cured for 5 seconds, the excess cement was removed using an instrument, and the cement was light-cured again for 40 seconds using Optilux

501. In the second group, the surface of the washed cavity was dried using moisture-absorbing paper for 30 seconds, leaving the surface visibly moist and shiny (wet surface). Cores were then cemented in the cavity using the same method used for the first group. The surface moisture level of the third and fourth groups was set correspondingly as the first and second groups, respectively. Chemical curing, in both the groups consisted of leaving, restorations in darkness for an initial 3 minutes, followed by removal of excess cement, after which the restoration was left in dark for an additional 5 minutes, according to the manufacturer's instructions. In the fifth group, the restorations were cemented using Panavia F2.0 with light curing according to the manufacturer's instructions. Chemical compositions, application procedures of the resin cements, and composite resin used in the study are shown in Table 1. The samples in all groups were then stored in water at 37°C for 24 hours. Specimens were subjected to 1000 thermal cycles and then external surfaces of the teeth, except the apex, were sealed with two layers of nail varnish. For leakage evaluation using the fluid filtration method, a pool was prepared 1mm above the resin core restorations. This space created a reservoir for the insertion of protruded metal tubing. The rationale for this statement is that this technique eliminated clogging of the metal tubing and prevented the generation of false negative results. The cut surfaces of the pulp chambers were then cemented on to 2 x 2 x 0.7 cm pieces of Plexiglass with a cyanoacrylate adhesive (Zapit, DVA, Corona, CA, USA). The pieces of Plexiglass had 18-gauge stainless-steel tubes placed through their centers, ending flush with the upper surfaces. The access opening of the tooth segments were then positioned over the tubes to allow a direct communication between the pulp chamber and the microsyringe system. Each Plexiglass-pulp chamber assembly was attached via polyethylene tubing (Fisher Scientific, Pittsburg, Penn, PA,

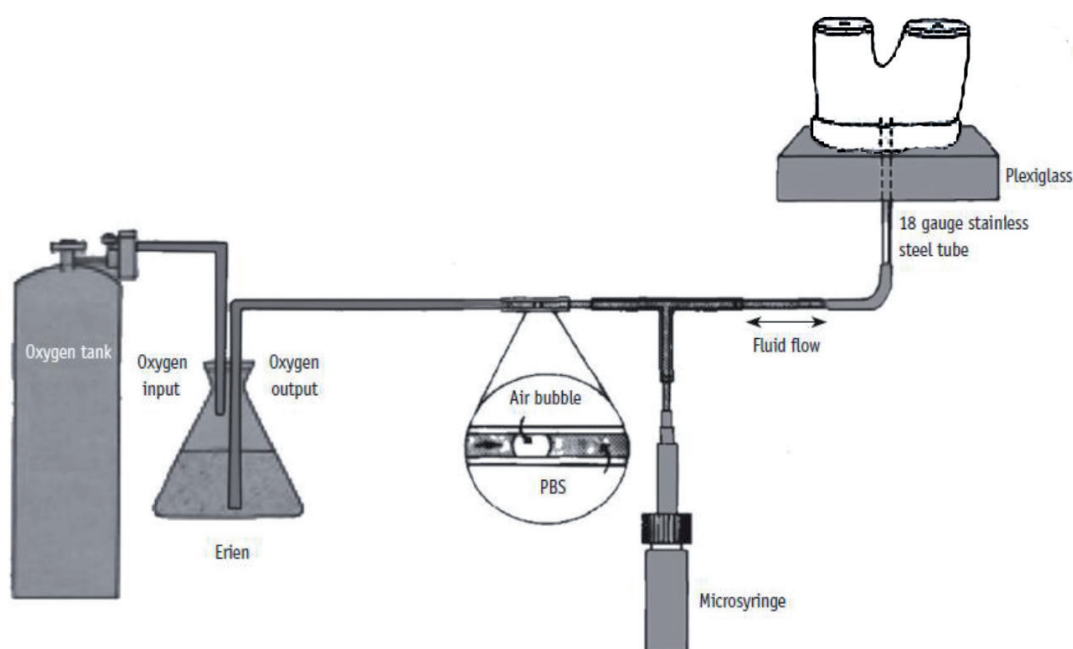


Figure 1. Fluid filtration device used to measure the microleakage of composite core bonded under different moisture level and curing method with resin cements

Table 1. Chemical composition and application procedure of used resin cement and composite

Material (Manufacturer)	Material composition	Procedure
Resin cement: Clearfil SA Luting, (Kuraray Noritake Dental, Tokyo, Japan)	Paste A: Bis-GMA,TEGDMA, MDP, hydrophobic aromatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, dl-camphorquinone, benzoyl peroxide, Initiator Paste B: Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, surface treated sodium fluoride, accelerators, pigments (0291BA)	After mixing the equal amounts of paste A and B, apply the cement paste mix to the restoration and cavity prepared.
Panavia F2.0, (Kuraray Co. Ltd.)	ED Primer A: HEMA, 10-MDP, 5-NMSA, water, N,N-diethanol-p-toluidine (00237A) ED Primer B: 5-NMSA, N,N-diethanol-p-toluidine, water, sodium benzene sulfinate (00115A) Pasta A: 10-MDP, silanated colloidal silica, bisphenol A polyethoxydimethacrylate, hydrophobic and hydrophilic DMA, silanized silica filler, benzoyl peroxide, DL-camphorquinone (00244A) Paste B: hydrophobic and hydrophilic DMA, sodium 2,4,6-triisopropyl benzene sulfinate, N,N-diethanol-p-toluidine, bisphenol A polyethoxydimethacrylate, colloidal silica, sodium fluoride, silanized barium glass filler, silanized titanium oxide	Mix one drop of each ED primer liquid A and B for 5s, air dry gently after 60s (Enamel and Dentin Pre-treatment). Apply K-etchant gel for 5s, rinse, air dry, mix one drop of each Clearfil SE primer and porcelain Bond Activator for 5s (For Composite Pre-Treatment). Mix universal and catalyst paste for 20s, after removal excess cement, light cure for 40s (For Luting agent Mixing).
Resin composite: Clearfil AP-X (Kuraray Co. Ltd.)	Bis-GMA, TEGDMA, dimethacrylate, filler, photo/chemical, initiator	Incrementally placement and curing with direct and indirect methods.

HEMA, 2-hydroxyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 5-NMSA, N-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol-A-diglycidylether dimethacrylate; DMA, dimethacrylates; TEGDMA, triethylene glycol dimethacrylate.

Table 2. The microleakage mean values ($\mu\text{L}/\text{min}/\text{cmH}_2\text{O}$) and standard deviation (SD) for the experimental groups and pairwise comparisons by Tukey test

Type of curing	Resin cement		Clearfil SA Luting (Experimental groups)		Panavia F2.0 (Control group)
			Moisture level		(As manufacturer instruction; dry/Light)
	(Wet) Mean (SD)	(Dry) Mean (SD)	(Wet) Mean (SD)	(Dry) Mean (SD)	Mean (SD)
Light-curing	(G1) 20.5×10^{-6} (0.1 $\times 10^{-6}$) ^{aA}	(G2) 34.4×10^{-6} (0.4 $\times 10^{-6}$) ^{aB}	(G3) 14.3×10^{-6} (0.1 $\times 10^{-6}$) ^{ba}	(G4) 10.10×10^{-6} (1.0 $\times 10^{-6}$) ^{ba}	(G5) 11.47×10^{-6} (1.0 $\times 10^{-6}$) ^b
Self-curing	(G1) 20.5×10^{-6} (0.1 $\times 10^{-6}$) ^{aA}	(G2) 34.4×10^{-6} (0.4 $\times 10^{-6}$) ^{aB}	(G3) 14.3×10^{-6} (0.1 $\times 10^{-6}$) ^{ba}	(G4) 10.10×10^{-6} (1.0 $\times 10^{-6}$) ^{ba}	(G5) 11.47×10^{-6} (1.0 $\times 10^{-6}$) ^b

Groups (G 1-5) with the same small letter are not significant in rows.
Groups (G 1-4) with the same capital letter are not significant in columns.

USA.) to a fluid filtration device. The pulp chambers were filled with water through the 18-gauge needle, taking care to remove all air bubbles that could be visible through the transparent Plexiglass. Empty root canals beneath the sealing materials were also filled with water to maintain hydration of the dentine. The fluid filtration method was used for the quantitative evaluation of leakage. A small 1- to 2- μL air bubble was introduced into the system with a microsyringe and travelled within a 25- μL micropipette, serving as an indicator of fluid movement. All tubing, pipette and syringe were filled with distilled water under a pressure of 239 cmH_2O or 23.4 Kpa ³. The linear motion of air bubbles in millimeter was recorded in 2 minutes intervals for 8 minutes and converted

to numerical data with the help of Photoshop software CS6. The multiple reading averages were used to quantify the amount of microleakage for each sample ($\mu\text{L}/\text{min}/\text{cmH}_2\text{O}$). The ANOVA and Tukey's tests were used and the significance level for all the tests was defined as 5% ($p < 0.05$).

RESULTS

The highest and lowest mean values of microleakage were observed in the second and fourth group, respectively (Table 2). The one-way analysis of variance test indicated significant differences among the five groups ($P = 0.002$). To conduct pairwise comparisons, the Tukey test was used and

showed significant differences between the microleakage in some group pairs (Table 2). Two-way ANOVA showed no significant interaction between curing modes and moisture levels ($P > 0.05$). It also indicated that the curing mode had a significant effect on microleakage ($P = 0.001$), whereas the moisture level did not have any significant effect on the microleakage ($P = 0.157$). The results showed that the microleakage mean values were significantly higher using the light-cured method compared to the self-cured method ($P = 0.002$).

DISCUSSION

This study determined the microleakage of cemented composite cores to the pulp chamber dentine with self-adhesive and self-etching resin cements using the fluid filtration method that has been described in previous studies^{16,17}. Panavia F 2.0 resin cement is a conventional self-etch resin cement, the use of which has been validated in several studies^{6,13,14}. However, Clearfil SA Luting is a recently introduced self-adhesive resin cement that is chemically similar to Panavia F 2.0; therefore, Panavia F 2.0 resin cement was used as a control in this study. Cementation to pulp chamber dentine may affect bonding and adhesion due to the natural variability of dentine topography¹⁸. During the endodontic procedure, since some area of pulp chamber remains unprepared, this area does not have a dominant smear layer. So the adequacy of adhesive systems mainly depends on pre-dentine enriched with collagen, dilated tubules, and to a lesser degree, on inter-tubular dentine. The cement bond strength values to pulp chamber dentine surfaces was usually less than the bond strength of occlusal and flat coronal dentine^{4,19}. In addition, self-adhesive cements may not infiltrate as well into the dentine substrate due to their high viscosity compared to direct bonding adhesives that form a thin hybrid layer²⁰. The performance of resin cements depends on their functional monomer for demineralization, penetration, and the stability of the chemical bond with calcium ions that forms the micro-mechanical and chemical binary bonding mechanism²¹. Self-adhesive resin cement such as Clearfil SA Luting and self-etching resin cement (Panavia F2.0) contain the functional monomer 10-MDP, which has been shown to be the most effective monomer for chemical bonding because it reacts strongly with the calcium of dentine to form a stable and insoluble salt²². To develop effective procedures for SARC, it should be recognized that water plays an important role providing the required hydrogen ions to demineralized surface. However, the SARC (Clearfil SA Luting) does not have any water in its mixture²³. Therefore; these types of materials rely on the water inside of dentine or even the water remaining on the surface of the dentine after rinsing. The results of the present study show that the amount of water remaining on the dentin surface did not significantly affect microleakage in the experimental groups. Even after drying the surface, the internal water existing inside the dentine was adequate for the cement reaction, and adding extra water to dentine may destroy the consistency and coherency of the polymer and cause some defects within the matrix. The results of this experimental study rejected the null hypothesis, because the curing type, as opposed to the surface moisture, was

shown to be the significant determinant of microleakage. In the past, it was thought that the self-curing method of some dual-cured resin cement systems may not have an effect on improving the bond strength²⁴. The results of the current study have shown that the chemical curing of resin cement causes less microleakage than light curing and this indicates that the chemical initiator benzoyl peroxide existing in self-adhesive resin cement is adequate for a sufficient curing reaction. It has been clarified that although light-curing of resin cements can result in more complete curing in a shorter period of time, this high speed process may increase the polymerization shrinkage in a cavity with a high C-factor, causing more leakage²⁵. The current study used box-shaped cavities for cementation of composite cores with high C-factor. Previous research has shown that a cementation process with reduced C-factor gives the same results as using the layered method in placing the composite restorations and produces high bond strength²⁶. On the other hand, polymerization shrinkage during one-step cement placement can cause stresses that lead to the formation of a gap and bond breakage. The magnitude of these stresses depends on the cavity geometry, the material properties of cement and the light radiation energy in light-cured systems²⁷. Therefore, slow polymerization as occurs in chemical curing, may reduce the stresses on the junction and bonding location because it may reduce shrinkage stress in the resin. Based on this, it is suggested that chemical curing be used for SARC because, the delays in exposure lead to improvement of results under a high C-factor condition²⁸, if chemical hardening reaches an adequate level and sufficient isolation is provided during the hardening process in clinic. Numerous studies suggest that light curing results in improvement of the properties of the resin cement significantly²⁹⁻³³. Nevertheless, the translucency of resin cement and the environmental temperature and restoration height, indirectly affect the level of success of cement curing³⁴. The current study has shown that the microleakage of cemented samples using two types of cements did not differ significantly, although more microleakage occurred in samples using self-adhesive resin cement than in those using self-etched cement. Considering the fact that the self-etch cements were placed in two phases and the self-adhesive cement was placed in one phase²⁷, and that the chemical structure of both cements were almost identical, this result is expected. The limitations of this study include: missing the likely groups for both resin cements, the limited number of resin cements used for comparison (particularly total-etch mechanism), and the fact that crown specimens were not fully restored. We suggest that future studies be done to examine other types of resin cements for use in bonding indirect cores or restorative material and to test various substrates with respect to microleakage and bond strength.

CONCLUSION

Considering the limitations of the current study, we can conclude that the microleakage of self-adhesive resin cement used in this study was lower in the case of self-cure than in the case of light-cure and was not related to the dentine surface moisture.

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