Original Article

Microleakage of Self-Adhesive Resin Cements Compared With Resin Cements Containing Etch & Rinse Adhesives

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Abstract

Background and Aim: Current self-adhesive resin cements have become popular for esthetic restorations. The purpose of this study was to compare the microleakage of 2 self-adhesive resin cements and 2 etch and rinse versions of the same brands.

Materials and Methods: Forty human third molars were randomly assigned to 4 experimental groups: 1-Rely X Unicem, 2- Rely X ARC+ Acid etch 37% + Single Bond ,3- Nexus 3 Acid etch 37% + Optibond Solo, and 4- Maxcem Elite. Microleakage of the specimens were then measured with a four point scoring system at both the cervical &occlusal areas, with the aid of a stereomicroscope. Microleakage scores were compared using Kruskul-Wallis analysis, followed by relative Dunn test.

Results: The microleakage of Rely X Unicem and Nexus 3 were significantly less in the occlusal region ($p<0.05$), whereas in the cervical area there was no significant difference in microleakage between Rely X Unicem and Rely X ARC ($P= 0.0087$). The microleakage of Nexus 3 was significantly less than that of Rely X Unicem.Maxcem Elite showed the highest level of microleakage. ($p<0.05$)

Conclusion: Microleakage of both cements using etch & rinse adhesive systems were significantly lower in comparison withtheir self- etch counterparts both at the cervical &occlusal areas. ($p< 0.05$)

Key Words: Microleakage, Self-Adhesive resin cement, Dentin adhesion, Enamel adhesion

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Introduction

Methyl methacrylate-based resin cements were introduced in 1952 for the purpose of indirect tooth-colored restorations [1]. Because brittle ceramic restorations require a strong substructure to render them resistant against occlusal forces and on the other hand bondable to the tooth and restoration, considerable advances have been made [2]. Etch and rinse bonding systems have been used for cementation of tooth-colored indirect restorations. Post-operative hypersensitivity is considered as one of the disadvantages of these materials which has been attributed to the patency of dentinal tubules followed by acid etching [2-4]. In addition numerous clinical stages can pose difficulties in handling of the material and increase the likelihood of procedural failure [5-8]. Bonding ability to the tooth structure, microleakage and recurrence of caries specifically in tooth-cement interface have been considered as indices of longevity for indirect restorations [2]. Microleakage causes post-restoration or post-cementation sensitivity [9]. Resin cements

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based on self-etch bonding technology have been developed in order to prevent post-cementation sensitivity and to decrease technique sensitivity and operation complexity [10]. Self-adhesive resin cements were introduced in 2002 [5], in which etching, priming and bonding components are incorporated in the cement preparation. It appears that chemical bond to tooth structure can be achieved to some extent [5]. There is not enough information including long-term clinical data about the composition of these materials. On the other hand controversial data exist concerning leakage comparison of self-adhesive and etch-and-rinse cement systems [1,3,5,8,11]. Al-Saleh and colleagues in 2010 demonstrated that ScotchBond Multipurpose had the lowest leakage in enamel margin and Rely-X and Breeze showed the lowest leakage in dentinal margins [12]. According to the laboratory results, bondability of self-adhesive cements to dentin and different restorative materials was favorable and comparable to other multi-stage resin cements. On the other hand [5]. The purpose of this study was to compare microleakage of RelyX ARC and Nexus3 resin cements using etch-and-rinse bonding system and Rely X Unicem and Maxcem Elite self-adhesive resin cements 24 hours after application.

Materials and Methods

This experimental study was carried out on human extracted teeth using standard ISO TR11405 method [13]. Forty extracted third molars obtained from 20- to 30-year-old patients which were stored in normal saline for 3 months were selected. The teeth were stored in 0.1% thymol solution for 48 hours. A one-millimeter round cavity 3 mm in diameter was prepared in midbuccal surface of each tooth. The occlusal and cervical margins were placed in enamel and cervical dentin/cementum, respectively. The teeth were randomly divided into four groups of ten for bonding (See table 1).

Group1. Enamel and dentin surfaces were rinsed and air-dried before application of RelyX Unicem (3M ESPE, Seefeld,Germany). Curing was performed using a 400 mW/cm^2 light intensity for 40 seconds, using Coltolux curing device (Coltene, Switzerland).

Group 2: After etching the surface with 37% phosphoric acid (3M ESPE, Seefeld, Germany) for 15 seconds, the teeth were rinsed with water for 10 seconds and dried. Then a layer of Single Bond (3M ESPE,Seefeld,Germany) was applied and air-dried after 20 seconds for 2-5 seconds and cured under previously-mentioned conditions. According to the recommendations of the manufacturer two layers of bonding was applied and the mentioned stages were repeated twice.) Afterwards, Rely X ARC (3M ESPE, Seefeld, Germany) was applied within the cavity and light-cured under the mentioned circumstances.

Group 3 Enamel and dentin surfaces were rinsed. After removing the moisture with a mild flow of air, Maxcem Elite (Kerr Hawe, Switzerland) was applied within the cavity and light-cured according to the previous conditions.

Group.4: After etching enamel and dentin surfaces with 37% phosphoric acid (3M ESPE, Seefeld, Germany) for 15 minutes, rinsing and drying, one layer of Optibond Solo (KerrHawe, Switzerland) was applied with gentle bush motions for 15 minutes and mildly air-blasted for 3 seconds and cured for 20 seconds. Then, Nexus 3 (KerrHawe, Switzerland) was applied and cured.

In all groups, extra cements were removed with a surgical blade and polished with extra-fine to coarse Soflex(3M ESPE) polishing discs. In order to prevent penetration of silver nitrate solution specifically in apical foramen, all tooth surfaces were sealed with a nail varnish except for a one-millimeter margin around the cemented area . The samples were stored in 37 degrees centigrade for 24 hours and placed in 50 weigh percent silver nitrate solution for 24 hours. Then, the samples were immersed into radiographic developing solution under fluorescent light for 24 hours. All teeth were subsequently sectioned using a 0.7mm blade (Mecatome, Pressi Inc., France). Two longitudinal cuts were made so that four surfaces, including coronal and cervical parts of the prepared cavities were

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Table 1. Description of the materials used in the present study

Group	Resin cement	Surface treatment	Adhesive
Group 1 **N=10**	Rely X Unicem (3M ESPE, Seefeld,Germany)	----	----
Group 2 **N=10**	Rely X ARC (3M ESPE,Seefeld,Germany)	37% phosphoric acid	Single Bond (3M ESPE,Seefeld,Germany)
Group 3 **N=10**	Maxcem Elite (KerrHawe, Switzerland)	----	----
Group 4 **N=10**	Nexus 3 (KerrHawe, Switzerland)	37% phosphoric acid	(Optibond Solo KerrHawe, Switzerland)

created to evaluate leakage. Therefore, two cross-sections, each containing two observable margins were available. Leakage evaluation in both coronal and cervical margins was performed under 10x magnification in distal, mid-distal, mesial and mid-mesial areas according to ISO/TR11405 standards using a stereomicroscope (Nikon, C-DS, Japan) [13]. Penetration of silver nitrate was evaluated and recorded according to the ISO scorings as follows:

Score 0. No penetration

Score 1. Penetration into enamel part of the cavity

Score 2. Penetration into dentinal part of the cavity without involvement of the pulpal floor

Score 3. Penetration into pulpal floor of the cavity

Data pertaining to the leakage of four cements were compared using Kruskal-Walis statistical test. Pairwise comparisons were made using complementary Dunn test.

Results

Leakage scores in different experimental groups are demonstrated in tables 2 through 4. Because two sections were made in each cavity and four points in each section was evaluated, microleakage in some areas was not detectable due to thinness and brittleness of the sections. Therefore such samples were excluded from the study. According to the Kruskal-Walis and complementary Dunn test there was a significant difference among all experimental groups ($p<0.0087$). In occlusal margin, Nexus 3 and RelyX ARC showed a significantly less microleakage in comparison with Rely X Unicem and Maxem Elite. In the cervical margin, Nexus 3 and RelyX ARC there was also a less microleakage compared with RelyX Unicem and Maxem Elite. RelyX Unicem indicated a significantly lower microleakage in comparison with Maxem Elite in both occlusal and cervical margins.

Table 2. Amount and frequency of microleakage in study groups

Leakage Score		Rely X ARC	Nexus 3	Maxcem Elite	Rely X Unicem	P.V
0	Frequency	13	21	11	2	P ≤0/05
	Amount	%19	%31	%16	%3	
1	Frequency	30	40	10	31	
	Amount	%44	%59	%15	%47	
2	Frequency	17	1	19	20	
	Amount	%25	%1	%28	%30	
3	Frequency	8	6	28	13	
	Amount	%12	%9	%41	%20	

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Table 3. Amount and frequency of microleakage in enamel (occlusal) margins of the study

Leakage Score		Rely X ARC	Nexus 3	Maxcem Elite	Rely X Unicem	P.V
0	Frequency	12	15	11	0	P ≤0/05
	Amount	%32/3	%44	%30	%0	
1	Frequency	20	19	10	21	
	Amount	%58/8	%55/9	%29/4	%53/5	
2	Frequency	2	0	10	9	
	Amount	%8	%0	%29/4	%27/6	
3	Frequency	0	0	3	3	
	Amount	%0	%0	%9	%9	

Table 4. Amount and frequency of microleakage in dentinal (cervical) margins of the study

Leakage Score		Rely X ARC	Nexus 3	Maxcem Elite	Rely X Unicem	P.V
0	Frequency	1	3	0	2	P ≤0/0087
	Amount	%2/9	%17/6	%0	%6	
1	Frequency	10	21	0	10	
	Amount	%29/7	%61/6	%0	%30/3	
2	Frequency	15	1	9	11	
	Amount	%44/4	%2/9	%26/4	%33/3	
3	Frequency	8	6	25	10	
	Amount	%23/5	%17/6	%73/6	%30/3	

Discussion

In the current study, two resin cements, i.e. RelyX ARC (3M ESPE, Seefeld, Germany) and Nexus 3 (KerrHawe, Switzerland) with etch-and-rinse bonding systems were compared with two self-adhesive resin cements i.e., RelyX Unicem (3M ESPE, Seefeld, Germany) and Maxcem Elite (KerrHawe, Switzerland) for their microleakage in cemento-enamel and cemento-dentinal junction areas. According to the statistical tests, etch-and-rinse bonding systems showed less microleakage in comparison with self-adhesive counterparts in both coronal and cervical regions. Since the cavity margin in occlusal area ends in enamel and in cervical area in dentin and cementum, microleakage evaluation in any of these areas can show the bonding quality of the cement to enamel as well as dentin and cementum. In this study, the methodological details such as sample storage, cavity dimensions, and leakage scorings were performed according to ISO/TR 11405 specifications. This rendered our results comparable to other similar works [11].

In order to more accurately compare the effect of bonding systems of the cements, self-adhesive resin cements of two manufacturers i.e., Kerr and 3M ESPE were compared with resin cements using etch-and-rinse bonding systems of the same manufacturing companies. Although reducing the treatment steps and technique sensitivity in self-adhesive resin cements can have a favorable effect in the procedural outcome, their microleakage evaluation and comparison with other resin cement systems are of paramount importance. In vitro leakage studies are the primary method for evaluation of seal at the restorative material-tooth substance interface [11]. In case resin cements are contemplated for cementation of indirect restorations, the cement type, regarding the bonding system used, and other properties such as setting time, and cement thickness can be influential in microleakage.

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Superiority of resin cements in terms of reduced microleakage, and increased strength over conventional cements such as zinc phosphate cement is well established [10]. Albert, also compared microleakage of resin cements with a glass ionomer , a resin-modified glass ionomer , and zinc phosphate cement with resin cement showing the least amount of microleakage [7]. Microleakage evaluation can be performed in tooth-cement and cement-restoration interfaces. The aim of this study was to evaluate microleakage at the tooth-cement interface. Type of indirect restoration such as different ceramics and composites can affect the thickness of the cement layer and eventually shrinkage and other properties of the bonding surface. On the other hand, bond between the cement and different indirect restorations such as composites and ceramics is another important factor. In case leakage happens through cement-restoration interface, the exact origin of the leakage is difficult to diagnose and the results cement-tooth microleakage is distorted. Therefore, it was preferred that the cement be evaluated without application of an indirect restoration. Similarly, in a study by Al Saleh et al in 2010 resin cements were used as liners under composite resin restorations and the microleakage was evaluated in enamel and dentin areas [12]. Creation of four cross-sections for evaluation of microleakage through two longitudinal cuts and leakage evaluation in four points are among the strong points of this study.

The most pronounced clinical manifestation of microleakage is post-cementation hypersensitivity. Primary gap between cement and tooth can occur following polymerization shrinkage of the cement, even in case of thinness of the cement layer. Therefore, lack of an adequate seal is as a result of the fact that the bond between the tooth structure and cement failed to endure shrinkage forces of initial polymerization stages. This can also occur due to inability of establishing an acceptable bond between tooth structure and the cement. In most of the microleakage studies, leakage evaluation is performed after thermo-cycling of the samples. The increasing effect of the thermo-cycling in microleakage is well documented in most of the investigations [1]. Since such increasing effect in microleakage was clear, and the main purpose of this study was to evaluate the leakage behavior of the cement materials, thermo-cycling was not carried out. In a study by Ibarra.G et al., microleakage of Variolink II, Panavia F, Resinomer, and Fuji plus was shown to be increased following thermo-cycling [4]. All cements indicated more leakage in dentinal in comparison with enamel margins. This was in accordance with our results [4]. In ISO specifications, thermo-cycling is not mandatory to be performed [13]. In the present investigation penetration depth of the silver nitrate was considered as the indicator of microleakage. Silver nitrate contains very small particles (0.059 nanometers in diameter) in an acidic solution with a pH of 4. Therefore, penetration of this material is more than that of other dyes. On the other hand, its acidic pH can dissolve phosphate and calcium salts at the bonding interface resulting in overestimation of leakage [4]. In this study, the buffered solution with a pH of 9 was utilized in order to prevent the effect of the acidic pH of the silver nitrate solution. In a study by Piwowarczyk [6] it was concluded that the self-adhesive resin cement, RelyX Unicem, showed the least amount of microleakage in comparison with other studied cements both in enamel and dentin. It was posited that less leakage ensues in self-adhesive cements due to lack of different interfacial bonding layers that compromises adaptability. It is worth noting that in this study, more marginal gaps are observed in self-adhesive resin cements, an ambiguity which requires more investigations to be elucidated. Contrarily, in this study, self-adhesive resin cements showed more microleakage in both dentinal and enamel margins. Such incongruence can be attributable to the point that in Piwowarczyk's study, these cements were used for luting of the crowns with gold alloys. More favorable adaptation of the gold alloy with tooth structure in comparison with other restorations, results in a decreased thickness of cement material thereby decreasing polymerization shrinkage. Sadr et al compared self-adhesive resin cements, showing no

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statistically significant difference in cervical microleakage of Unicem and Maxcem. There was a better coronal marginal seal in Maxcem in comparison with Unicem. Totally, there was no significant difference between coronal and cervical margins of tested materials [3]. Such differences between these results and those of ours can be related to the use of bovine teeth by Sadr et al with different enamel and dentin structures. Also, in the aforementioned study, methylene blue dye was used to indicate leakage which might have posed differences in the results, due to less penetration of methylene blue compared with silver nitrate. In a study by Uludag et al, RelyX ARC, Variolink II and Panavia 21 were compared with each other. The highest amount of leakage was reported to be related to Panavia 21 [14].

Panavia 21 bonding system is a self-etch primer that combines etching and priming steps. It has less treatment steps that does an etch-and-rinse bonding system. Panavia 21 indicated an increased microleakage in enamel showing congruence with our results. In Panavia self-etch primers are used to prime dentin and enamel surfaces. Since acidity of self-etch primers are lower than that of 37% phosphoric acid and enamel is more mineralized than dentin, a favorable etching pattern for penetration of resin into enamel cannot be achieved. It is suggested that etching be performed separately on enamel surfaces and self-etch primers be subsequently used [15]. More treatment steps in etch-an-rinse cement systems can increase accuracy of the procedure and therefore enhance favorability of bonding. In etch-and-rinse cement systems, etching with 37% phosphoric acid is separately performed in enamel and dentin. This can ensure formation of more pronounced microtags in enamel and prepared collagen fibers of dentin. Self-adhesive resin cements cannot form a hybrid layer similar to that formed by etch-and-rinse systems without separate etching and priming [15]. Morphological findings following application of self-adhesive cements at the cementodentinal junction have a significant difference with those of resin cements that require surface preparation prior to cement application [5] Although acceptable clinical results have been obtained, more clinical studies are required to confirm long-term results. Totally, self-adhesive cement systems have lower bond strengths to enamel in vitro [5]. Unicem was the first self-adhesive resin cement introduced in 2002 and its detailed composition is not clear [5]. In some investigations, this cement showed superiorities with respect to other self-adhesive cements in terms of leakage and bond strength.

Self-adhesive resins are preferred to be used in cementation of restorations with mechanical retention. In cementation of restorations such as laminate veneers which rely on bonding technology, cements with separate adhesive systems are preferred.

Conclusion

It was finally concluded that:

1- Cements with etch-and-rinse bonding systems showed less microleakage at the dentinoenamel junction.

2-Microleakage was more observed in self-adhesive cement systems.

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