# Influence of adhesive systems composition on dual-cured resin cements

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The purpose of this study was to evaluate the influence of different adhesive system's composition on the sealing ability of a dual cured resin cement when used for luting resin composite veneers. Thirty teeth were prepared for veneers with the preservation of the enamel margins on previously extracted sound human anterior teeth. Polyglass veneers were prepared following manufacturer's instructions. Teeth were randomly assigned to three groups (n=10) and veneers were luted with a dual-cured resin cement in conjunction with one of the following adhesive systems: 3 steps etch&rinse, 2 steps etch&rinse and self-etch all-in-one. Teeth were thermocycled and immersed in 0.5% basic fuchsine solution for 24 hours. The specimens were cut in a bucco-lingual plane in order to obtain a section at the middle of the mesio-distal dimension of the veneer. Dye penetration along the tooth-cement (MTC) interface was evaluated at 40x and microleakage values recorded. For each interface microleakage values were refered to the total length of that interface and data were subjected to statistical analysis by Student's t and Kolmogorov - Smirnov test at a $p<0.05$ level of significance. Significantly less microleakage was associated with the use of etch & rinse adhesive systems comparing with the self-etch adhesive.

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## 1. Introduction

The weak link with indirect restorations is eventual debonding of the luting cement. Several factors may affect the wall-to-wall integrity. The use of resin cements (RC) for luting indirect restorations has the benefit of adhesives, wich have the potential of eliminating surface flaws [1,2]. However, incomplete adhesive diffusion throughout the demineralized dentin has been reported for conventional bonding agents [3] leading to post-operative sensitivity as a result of the exposed collagen [4]. Besides luting material and technique, also substrate conditions represent a critical factor in the quality of a durable wall-to-wall integrity. As a result of its morphologic variability, the properties of the organic component, and the changing conditions of humidity, dentin is the least predictable and undependable substrate for bonding [5,6].

Current adhesive research focuses on the simplification of application procedure [7]. Reduction of the number of application steps should reduce manipulation time, and abate technique sensitivity, thus improving bonding effectiveness. This trend in adhesive dentistry has led to the introduction of self-etch adhesives, of which the one-step self-etch adhesives (1-SEAs) or the so-called all-in-one adhesives are the most user-friendly adhesive systems nowadays on the market [7]. Research, however, so far has demonstrated that simplified systems do not bring the expected improvement in bonding effectiveness, whereas used with resin cements for luting indirect restorations, or with resin based composites (RBC) for direct restorations [8–14].

The purpose of this study was to evaluate the influence of different adhesive systems on the sealing ability of Nexus NX3 (Kerr) dual cured resin cement when used for luting resin composite veneers. The quality of the marginal seal achieved with the materials on trial was assessed *in vitro* through a microleakage test and light-microscopic observations of the tooth-cement interface after thermo-cycling.

## 2. Experimental

Thirty freshly extracted sound human anterior teeth were used. The teeth were cleaned, by removing calculus and soft tissue deposits with a hand scaler, and then stored in 0.9% NaCl containing 0.02% sodium azide at 4 $^0$C until used. All the teeth were prepared for veneers using a medium-grained diamond bur (SG881KS.014, Edenta AG, Switzeland) in a water-cooled high-speed turbine. The preparation was made with the preservation of enamel margins, in order to obtain the following dimensions: 0.2-0.3 mm cervical, 0.5 mm in the middle third of the buccal surface and 0.8-1 mm at the incisal margin. The preparations followed to plans, a cervical plan parallel with the longitudinal axe of the tooth and an oblique incisal plan (Figure 1). The cervical margins were prepared at $90^0$, and all internal angles were rounded.

An impression of the prepared anterior teeth was made using a standard metalic tray and type 0 and 3 polivinylsiloxane (Kohler Sil 1 Soft, Kohler Medizintechnik), using sandwich technique. Thirty dies were made using type IV high-strength stone.

*Fig.1. Teeth prepared for composite veneers.*

The veneers were made with poly-glass composite (BelleGlass NG, Kerr) in the laboratory following manufacturer's instructions.

The teeth were randomly divided into three groups of 10 teeth (n=10) and luting of veneers was accomplished using a dual-cured resin cement (Nexus NX3, Kerr) in conjunction with one of the following adhesive systems: Group I, OptiBond FL (Kerr), three-step etch&rinse adhesive; Group II, OptiBond Solo Plus (Kerr), two-step etch&rinse adhesive and Group III, Optibond All-In-One (Kerr), one step self-etch adhesive (Table 1).

The adhesive systems were applied as follows:

***Group I:*** The surface was etched with 37% phosphoric acid gel for 30 s at enamel surface and 15 s for dentine surface, rinsed for 15 s and gently dried for 2 s. The primer was applied with a microbrush for 15 s with a light brushing motion and air-thinned for 5 s with canned compressed air to achieve a visibly uniform layer. The adhesive was applied using a microbrush for 15 s, air-thinned for 5 s and light-cured for 20 s.

***Group II:*** The surface was etched with 37% phosphoric acid gel for 30 s at enamel surface and 15 s for dentine surface, rinsed for 15 s and gently dried for 2 s. The primer and adhesive solution was applied with a microbrush for 15 s with a light brushing motion and air-thinned for 3 s with canned compressed air to achieve a visibly uniform layer, than light-cured for 20 s.

***Group III:*** The Group III solution was well mixed for 3 s and than applied for 20 s with a light brushing motion. A second layer of solution was placed using the same protocol and the excess solvent was evaporated by air drying for 5 s. The adhesive was light-cured for 20 s.

Luting of the veneers was accomplished by applying the dual-cured resin cement, Nexus NX3 (Kerr) following manufacturer's instructions. Each veneer was light-cured with a Demetron II curing unit (Kerr Corp.) operated in standard mode at a light intensity of 740 ± 36 mW/cm$^2$ for 20 s from the cervical and incisal, than excess was removed followed by another 20 s curing.

The restorations were finished with Kerr BluWhite diamond burs and polished using a series of abrasive disks (OptiDisc, Kerr Corp.) and rubber points (HiLuster Dia Polishers, Kerr Corp.).

The restored teeth were stored in 37$^0$C water for 24 h, then subjected to thermocycling between 5$^0$C/55$^0$C for 1000 cycles, with a transfer time of 10 s and a dwell time of 25 s.

*Table 1. Adhesives used in this study.*

| Group (n) | Adhesive system | Composition |
|---|---|---|
| G I (10) | OptiBond FL | Etchant: 37% phosphoric acid<br>Primer: HEMA, GPDM,BHT ethanol, water, PAMA, CQ<br>Adhesive: BisGMA, HEMA, GDMA, CQ;<br>filler (15wt%):<br>fumed $SiO_2$, barium aluminoborosilicate, $Na_2SiF_6$;<br>coupling factor A174 |
| G II (10) | Optibond Solo Plus | Etchant: 37% phosphoric acid<br>Adhesive: Bis-GMA, HEMA, GDMA,GPDM, ethanol, CQ, ODMAB, BHT;<br>filler (15wt%):<br>fumed $SiO_2$, barium aluminoborosilicate, $Na_2SiF_6$;<br>coupling factor A174 |
| G III (10) | Optibond All-in-one<br><br>pH – 1.7 | HFGA-GMA, GPDM, ethanol, water, acetone, MEHQ, Bis-GMA, HEMA, ODMAB, BHT, CQ;<br>filler (15wt%):<br>fumed $SiO_2$, barium aluminoborosilicate, $Na_2SiF_6$;<br>coupling factor A174 |

HEMA = 2hydroxythylmethacrylate
GPDM = Glycerophosphate-dimethacrylate
BHT = 2,6-di-(tert-butyl)-4-methylphenol
PAMA = phtailic acid monomethacrylate
CQ = camphorquinone
Bis-GMA = bis-phenol-A-bis-(2-hydroxy-3-methacryloxypropyl)ether
GDMA = glycerol dimethacrylate
ODMAB = 2-(ethylhexyl)-4-(dimetylamino)benzoate
HFGA-GMA = hexafluoroglutaric anhydride-Glyceroldimethacrylate adduc
MEHQ = 4-methoxyphenol
A174 = gamma-methacryloxypropyltrimethoxysilane

The apices of the teeth were sealed with resin composite and the tooth surfaces were covered with two layers of nail varnish with the exception of 1 mm around the tooth-veneer interface. The teeth were then immersed in 0.5% basic fuchsine dye for 24 h. They were removed, washed, dried and their roots mounted in self-curing acrylic resin.

Each tooth was sectioned in a bucco-lingual plane using a water-cooled microtome (Isomet Low Speed Saw, Buehler Ltd) in order to obtain a 1.5 mm section thickness at the middle of the mesio-distal dimension of the veneer. Dye penetration along the tooth-cement (MTC) interface was evaluated with an inverted microscope (Olympus KC301, Olympus America Inc.) at 40x. The dye penetration evaluation was carried out using a quantitative method, microleakage being recorded (μm) using a QuickPhoto Micro 2.2 software (Olympus Inc) (Fig. 2). For each interface microleakage values were referred to the total length of that interface (MTCr) and data were subjected to statistical analysis by Student's t and Kolmogorov Smirnov test at a $p<0.05$ level of significance.

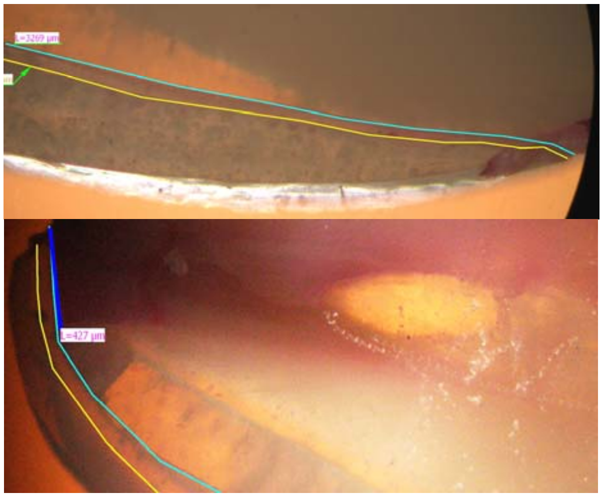

*Fig. 2. No dye penetration along the tooth-cement interface for Group I (above). Dye penetration measurement (427 μm, dark blue line) for Group III (below). Light-blue line represents the measurement for tooth-cement interface, and yellow line for cement-veneer interface.*

## 3. Results

Student t test did not reveal any significant differences between the tooth – cement interface length (MTC) of the three groups (Table 2 and Fig. 3).

*Table 2. Student t test – tooth – cement interface length by stereomicroscopic analysis between groups (p<0.05).*

| Group | Mean value | Standard deviation | p |
|---|---|---|---|
| I | 5501.70 | 579.60 | 0.86 |
| II | 5539.00 | 327.41 | |
| I | 5501.70 | 579.60 | 0.90 |
| III | 5467.30 | 605.45 | |
| II | 5539.00 | 327.41 | 0.90 |
| III | 5467.30 | 605.45 | |

Significantly less microleakage was observed for Group I (mean value 0,01±0,02) than for Group III (mean value 0,32±0,40)(p=0,04). The same statistically significant difference was observed between Group II (mean value 0,03±0,03) and Group III (mean value 0,32±0,40) (p=0,049) (Table 2). However, no statistically significant difference was determined between Group I and II (p=0,08).

Higher standard deviation was determined for Group III (SD 0.40) than for Group I or II (SD 0.02, respectively 0.03).

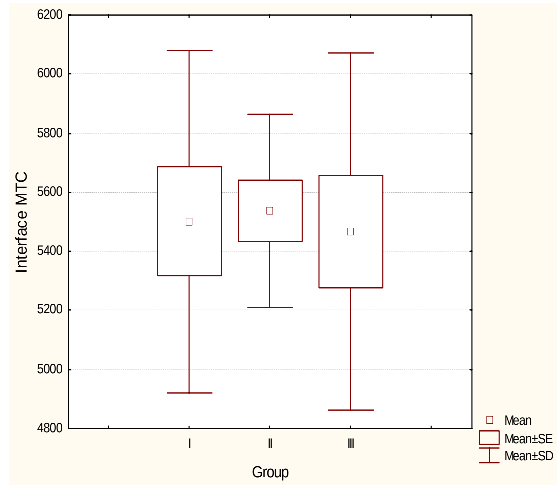


*Fig. 3. Tooth – cement interface length within groups (MTC).*

*Table 3. Student t test - overall microleakage levels by stereomicroscopic analysis between groups; significant differences are in red color (p<0.05).*

| Group | Mean value | Standard deviation | p |
|---|---|---|---|
| I | 0.01 | 0.02 | 0.08 |
| II | 0.03 | 0.03 | |
| I | 0.01 | 0.02 | 0.04 |
| III | 0.32 | 0.40 | |
| II | 0.03 | 0.03 | 0.049 |
| III | 0.32 | 0.40 | |

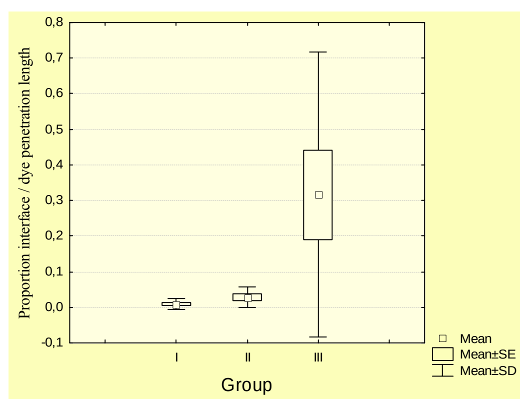


*Fig 4. Proportion interface MTC/dye penetration length.*

## 4. Discussion

As it has been pointed out by several authors, marginal leakage is one of the major drawbacks of a tooth-colored indirect composite restoration [6,7,15].

When luting resin based composite veneers, the weak interface is between the luting cement and the tooth. In respect of this, some aspects of the luting procedure with resin cements need to be considered. On one hand, a thin layer of cement would be desirable in order to reduce the stress generated by the material on curing [16], whilst on the other hand, luting with adhesive techniques is an operator-sensitive procedure because extreme care has to be given to the condition of the substrates. To withstand the latter, new resin cements have been introduced to satisfy the demand for an easy-handling, "user-friendly" material, but different opinion on their ability to seal the interface are available now [11,14,17].

Another way of simplifying the adhesive technique is to reduce the steps of the adhesive system used with conventional dual-cured resin cements.

Contemporary self-etch adhesive systems have been developed by increasing the concentration of acidic resin monomers and combining them with HEMA [18]. Then an increased interest on the study of the interaction between these systems and smear layer thicknesses occurred.

Self-etch adhesives have been classified based on their ability to penetrate smear layers and their depth of demineralization as mild, intermediary strong, and strong [13,19]. It has been speculated that self-etch systems with higher pH are less effective in solubilizing thick smear layers and demineralizing solid enamel or dentine surfaces for hybridization than adhesives with lower pH [7,13,14,20]. This observation seems to be more important especially on enamel [15,20], where hybridization is based mainly on micromechanical interlocking. The self-etch adhesive we used in this study (Optibond All-In-One) can be considered as strong because of its pH, 1.7. Even though the results showed that it was not capable of demineralizing the enamel, thus creating a hybrid layer more prone to degradation than etch&rinse adhesives. This could be explained by the fact that concentration of the acidic monomers in all-in-one adhesive systems is reduced due to dilution with solvent and hydrophilic/ hydrophobic resin monomers in the same solution [21].

In this study we used several types of simplified adhesive systems, either two-steps etch&rinse (Group II), or one-step self-etch (Group III). While no significant differences where found between the sealing capacities at the enamel margin for the first adhesive system, important differences where found for the all-in-one adhesive comparing with the classic three-step system (Group I). Results suggest that using simplified one-step self-etch adhesives on enamel margins significantly reduce the sealing ability of the resin cement.

Another important aspect is the amount of the solvent included in the self-priming solution. Water is present in all the self-etch systems employed because it is an essential component to enable ionization of the acidic monomers and demineralization of dental hard tissues. Consequently, the thickness of the adhesive layer may be thinner when greater quantities of solvent are used, and incomplete polymerization due to oxygen inhibition may occur [22-25]. Thus partially explains why applying several coats of all-in-one adhesive usually determine less microleakage. In our study we applied two coats of all-in-one adhesive system, which were than air-streamed for 5 seconds, in order to increase the time for the acidic monomers and primer to prepare the substrate, and than to stimulate the evaporation of the solvent, but despite this more leakage was found after thermocycling with this type of adhesive.

Despite the fact that simplified adhesive systems were developed in order to reduce technique sensitiveness of etch&rinse systems, our study revealed more inconstant results (SD +/- 0.40) with Optibond All-In-One than with etch&rinse adhesives.

Although several studies have reported the discrepancy between etching depth and adhesive penetration for etch&rinse adhesives [26-28], our study is in agreement with other studies that reveal one-step self-adhesive systems have no better performances especially when applied to enamel surfaces [17,20,29,30].

## 5. Conclusions

Within the limits defined in the experimental design, the following conclusions may be drawn:

- Significantly better sealing performances were recorded for the dual-cured resin cement used in conjunction with one of etch&rinse adhesive system.
- Different luting material combinations and procedures can affect the sealing ability of resin cements used for luting composite indirect restorations.

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