

Nanoleakage Of Giomer Resin Bonded With Either Total- Or Self-Etch Adhesives

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Abstract: Objectives: To evaluate the effect of lateral-load and thermal cycling on the nanoleakage along tooth/restoration interface of giomer resin restorations bonded with two different adhesives. **Methods:** A giomer self-etch adhesive system (FLBond II, Shofu) and a total-etch adhesive system (Prime & Bond NT, DENTSPLY) were used with Beautifil II (nano-hybrid resin-based giomer material, Shofu). Wedge-shaped cavities were prepared on the buccal surfaces of eighty extracted human premolars. Cavities were then divided randomly into two groups according to the used adhesive. Each group was further divided into four subgroups (n=10): A; control, B; subjected to lateral-load cycling (90N at 45 degrees for 5000 cycles), C; subjected to thermo-cycling (5-55°C for 500 cycles) and D; subjected to both lateral-load and thermo-cycling (90N at 45 degrees for 5000 cycles/5-55°C for 500 cycles). Nanoleakage was then tested using Quanta Environmental SEM and EDAX. Data were statistically-analyzed using Two-way Analysis of Variance (ANOVA) and Tukey's post-hoc tests. **Results:** The adhesive type and the combination of lateral-load and thermal cycling had a statistically significant effect on the nanoleakage ($P \leq 0.05$). Meanwhile, no significant differences observed between A, B and C subgroups for both tested adhesive systems. **Conclusions:** The type of the adhesive system affected the seal ability of the giomer restorations. Combining both lateral load and thermal cycling, in an attempt to simulate oral conditions, had a dramatic effect on nanoleakage. [Abo El Naga A. **Nanoleakage Of Giomer Resin Bonded With Either Total- Or Self-Etch Adhesives.** Journal of American Science 2012;8(2):27-34]. (ISSN: 1545-1003). <http://www.americanscience.org>. 5

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Introduction:

Esthetic dentistry has been widely advocated, since there was an increasing demand for materials that resemble the natural tooth. One of the key functions of these materials is to seal the exposed tooth structure from the oral environment, De Munck et al., (2005). The quality and durability of the marginal seal are of major importance for the longevity of restorations, Li et al., (2000). This is considered as a significant factor in preventing consequent hypersensitivity, secondary caries and pulpal damage. For this, there was a continuous and fairly rapid turnover in restorative and adhesive materials to improve their formulations and mechanical properties, Kubo et al., (2002).

On the two extreme ends of the continuum of direct tooth colored restorative materials are glass ionomer and resins composite. Various hybrid materials have been developed to combine the advantage of the glass ionomer cement with the aesthetic property and good handling properties of composite, Yap et al., (2003). Recently, a new category coined as "Giomer" (Glass ionomer + polymer) was introduced, whereby Giomer products are resin-based dental materials that comprise pre-reacted glass-ionomer (PRG) fillers, Matis et al., (2004). This material is claimed to be more effective in fluoride recharging characteristics than other resin matrix materials, thus preventing caries recurrence.

Similarly, self-etching fluoride-releasing adhesives were introduced lately aiming for prevention of caries recurrence together with simplicity, Ikemura et al.,

(2003). Nevertheless, simplification does not guarantee equal or improved bonding effectiveness, since, etch & rinse adhesives still perform best in laboratory and clinical researches, Peumans et al., (2005). Meanwhile, increased incidence of nanoleakage in the self-etch adhesives was reported by many researchers, Tay et al., (2002). In the clinical situation, teeth are subjected to mechanical stresses during chewing and swallowing, Frankenberger et al., (2005). Such stresses had been reported to dislodge restorations at the cavosurface margin, Van Meerbeek et al., (1993). Consequently, load cycling either occlusal or lateral had a significant effect on leakage and could adversely affect the marginal integrity of resin composite restorations. Accelerated in vitro aging tests for resin-dentin bonds by using lateral load cycling may be of some help to evaluate the long-term stability of these interfaces under more clinically relevant conditions.

In vitro thermocycling is a common mean of testing dental materials to aid in establishing durability for their in vivo use. Since, clinically, there was fluctuation in temperatures due to hot and cold drinks and food that ranges between 5 to 55°C. These fluctuations may induce stresses between the tooth substrate and the restorative material, Asaka et al., (2007), which consequently may lead to cracks that propagate along bonded interfaces, thus accelerating chemical degradation of the bonds. Therefore, thermal cycling is applied to simulate oral conditions and its effect on the bond strength of different adhesives was reported in several studies, Harari et al., (2002).

Although a number of fluoride-releasing dental adhesives are already commercially utilized, little review on their sealing ability has been reported. Thus, it was found of value to study the influence of each of the following variables on the nanoleakage of class V cavities: lateral load and thermal cycling using two different bonding systems used to bond a giomer restorative with an electron microscopy.

The objectives of this study were to evaluate the effect of lateral load and thermal cycling on the bonded interface and to compare the seal ability of two adhesive systems (a self-etch adhesive system to that of a total etch adhesive system) by examining the nanoleakage pattern along the resin dentin interface.

2. Materials and methods:

2.1. Materials:

2.1.1. samples:

Eighty sound extracted human premolars were used in this study. Teeth were cleaned, examined, sterilized using chloramine T solution and stored refrigerated.

They were used within a maximum of one month from their extraction.

Standardized wedge-shaped Class V cavity was prepared at the cemento-enamel junction on the buccal surface of each tooth. Half of the prepared cavity was above the cemento-enamel junction and the other half below it. The prepared cavities were measured 5 mm occlusogingivally, 3 mm mesiodistally and 2.5 mm in depth, Jang et al, (2001). The outline of the cavities was standardized using a stainless steel matrix band into which a window representing the selected length and width was cut into its middle.

The teeth were randomly divided into two groups according to the adhesive used. Adhesive systems' composition and manufacturers are shown in table (1). A giomer self-etch adhesive system (FL Bond II) and a total-etch bonding system (Prime & Bond NT) were used to treat the cavity walls of groups I and II respectively according to manufacturer's instructions. The treated cavities were then filled with Beautifil II (table 1) using oblique incremental technique.

Table (1): Manufacturers, manufacturers' instructions and compositions of the used restorative materials.

Material	Principal components	Manufacturer's Instructions	Manufacture
FL Bond II 2-step, self etching, fluoride releasing adhesive system	The primer: is acetone free adhesion promoting monomer with no incorporation of HEMA. Contains ethanol, methacrylic adhesive monomer, pure water and 4-AET (4-Acryloxyethyltrimellitic acid). The bonding agent: contains the S-PRG filler (Surface Pre Reacted Glass-ionomer). 2-Hydroxy Ethyl Methacrylate, UDMA and TEGDMA	1-Dispense the Primer and apply thoroughly on the enamel and dentin surfaces for 5 seconds. 2. Leave undisturbed for 10 seconds then dry with oil-free air for 5 seconds. 3-Dispense the Bonding Agent and apply an even layer on the entire surface for 5 seconds then light-cure for 10 seconds.	SHOFU INC., Kyoto, Japan
Prime & Bond NT light-cure self-priming dental adhesive.	Caulk 34% Tooth Conditioner Gel: contains phosphoric acid, highly dispersed silicon dioxide, colorant and water. Prime & Bond NT: contains Di- and trimethacrylate resins, PENTA (dipentaerythritol penta acrylate monophosphate), photoinitiators, stabilizers, nanofillers-amorphous silicone dioxide, cetaylamine hydrofluoride and acetone.	1. Apply etchant on enamel and dentin for 15 seconds. 2. Rinse for 10 seconds and blot dry conditioned areas with a moist Cotton pellet. 3. Apply generous amounts of Prime & Bond NT adhesive, leave undisturbed for 20 seconds, air-dry for 5 seconds and then light cure for 10 seconds.	Dentsply Caulk, Milford, Del, USA
Beautifil II nano-hybrid resin based giomer material.	Matrix: 16.7wt% of resin (Bis-GMA & TEGDMA) Filler structure: Surface Pre-Reacted Fluoroboroaluminosilicate Glass Filler Nano Filler, Multi Fluoroboroaluminosilicate Glass Filler Filler Loading: 68.6 vol% and 83.3wt% Range of particle size: 0.01 ~ 4µm Average Particle size: 0.8µm	1. Dispense the necessary amount of material from the syringe. After dispensing, close the syringe cap immediately; rotate the handle 360-degrees counter-clockwise to release the pressure within the syringe. 2. After packing of the material into the cavity, light cure each increment for 20 sec.	SHOFU INC., Kyoto, Japan

2.2. Methods:

2.2.1. Scheme of the work:

After restoration of the cavities, each group was further divided into four subgroups (n=10): subgroup A; control, subgroup B; subjected to lateral load cycling, subgroup C; subjected to thermocycling and subgroup D; subjected to both lateral load and thermocycling.

The root and the restoration of each tooth that was assigned to be subjected to load cycling were covered

with an aluminum foil in order to remove the teeth easily from the moulds, and then a specially fabricated split cylindrical Teflon mould (20 mm heights and 15 mm internal diameter) was filled with self curing acrylic resin. Each tooth was then embedded into the self-curing acrylic resin in the mould such that the long axis of the premolar was approximately 45° to the plane of the acrylic resin base, Jang et al, (2001). Lateral load cycling was applied on the middle of the

buccal cusp incline using Lloyd universal testing machine (model LRX plus II, Freham, England).

The specimens were subjected to lateral load cycling of 90 N for 5000 cycles, Li et al, (2002) in the form of a sine wave at a rate of 1 Hz. The rate was used as equivalent to the average masticatory cycle of 0.8–1.0 seconds. The force was centered on the middle of the inner inclines of the buccal cusp of each tooth using a specially designed stainless steel loading rounded tip. The subgroups that assigned for thermal cycling were thermocycled in water between 5 and 55 °C for 500 cycles (with 20 seconds dwell time).

In order to assess nanoleakage, the root apices of each tooth were sealed with sticky wax and the entire tooth, except for the restoration and 1mm apart from the restoration margins, was coated with two layers of nail varnish then the teeth were immersed in 50% freshly prepared silver nitrate solution for 24 hours in Light proof container in total darkness, rinsed under running water for 5 minutes then immersed in a photo developing solution for 8 hours while being exposed to a fluorescent light in order to reduce the silver ions to metallic silver, finally, the teeth were rinsed under running water for 5 minutes to remove the photo developing solution. Each tooth was sectioned longitudinally in a bucco-lingual direction through the center of the restoration and prepared to be examined under electron microscopy.

2.2.2. Nanoleakage testing :

In this study, nanoleakage was tested by the use of Quanta Environmental Scanning Electron Microscope (QESEM). The QESEM is an analytical tool that provides exceptional depth of field and minimal specimen preparation since it allows examination of the specimens without being coated whether with gold or carbon as in other scanning electron microscopes. EDAX (Electron Dispersive Analytical X-ray) analysis was also carried out in parallel to identify the existence of metallic silver particles. The use of SEM in combination with EDAX had the ability to present both distinct images and sensitive quantification of silver ion penetration accurate. As it permits analysis for the element composition of the scanned square area. Thus, provides accurate identification for the presence or absence of metallic silver particles along the adhesive tooth / restoration interface. By this, both false negative and false positive results were excluded.

2.2.3. Scanning and EDAX quantification:

Scanning and EDAX quantification were performed at three points along both tooth/restoration interfaces (occlusal and gingival) of each specimen. It was performed:

- 1) At the middle of the tooth/restoration interface,
- 2) Midway between the middle of the tooth/restoration interface and the cavity margin, and
- 3) Near the cavity margin.

2.2.4. Statistical analysis:

The mean of the weight percentage of silver deposition along both the occlusal and gingival margins was calculated.

Means and standard deviations of the percentages of silver penetration data then were calculated for each group and statistically analyzed using Statistical Package for Scientific Studies for Windows SPSS 15.0 (SPSS Inc., Chicago, IL, USA). Regression model with two-way analysis of variance (ANOVA) and Tukey's post-hoc test ($p \leq 0.05$) were used

3. Results:

The results of the Two-way ANOVA showed that the regression model is fit to describe the relationship between the studied variables. Means and standard deviations of silver penetration percentages (as indicated by EDAX) are shown in tables (2 and 3) and figures (1 and 2). As shown in table 2 and figure 1, silver deposition was not present in the occlusal walls of all experimental subgroups. Meanwhile, it was present in the gingival walls of all experimental subgroups.

Tukey's test indicated that, no statistically significant differences were observed between the control, thermal cycling, and lateral load cycling groups (table 3 and figure 2). This means that, both thermocycling and load cycling when applied separately had no statistically significant effect on the silver penetration percentages of all groups at the gingival tooth/restoration interface. Meanwhile, the adhesive type and the combination of thermal and lateral load cycling had a statistically significant effect on the mean percentage of silver penetration at the gingival tooth/restoration interface ($P \leq 0.05$). Fl Bond II subgroup that was subjected to lateral load cycling followed by thermocycling showed the highest means of silver penetration. This was followed by Prime & Bond subgroup that was subjected to lateral load cycling followed by thermocycling which showed lower values. However, the control group showed the lowest values ($P \leq 0.05$).

Table (2): Summary of the results of ANOVA:

Surface	Cycling Adhesive	FI Bond II		Prime & Bond NT		P-value
		Mean	SD	Mean	SD	
Occlusal	Control	0	0	0	0	1.000
	Thermocycling	0	0	0	0	1.000
	Load cycling	0	0	0	0	1.000
	Thermocycling + Load cycling	0	0	0	0	1.000
Gingival	Control	0.93	0.14	0.95	0.15	0.989
	Thermocycling	0.97	0.09	0.97	0.10	0.998
	Load cycling	0.98	0.12	0.98	0.08	0.996
	Thermocycling + Load cycling	6.73	0.50	4.32	0.43	0.013*

*: Significant at $P \leq 0.05$

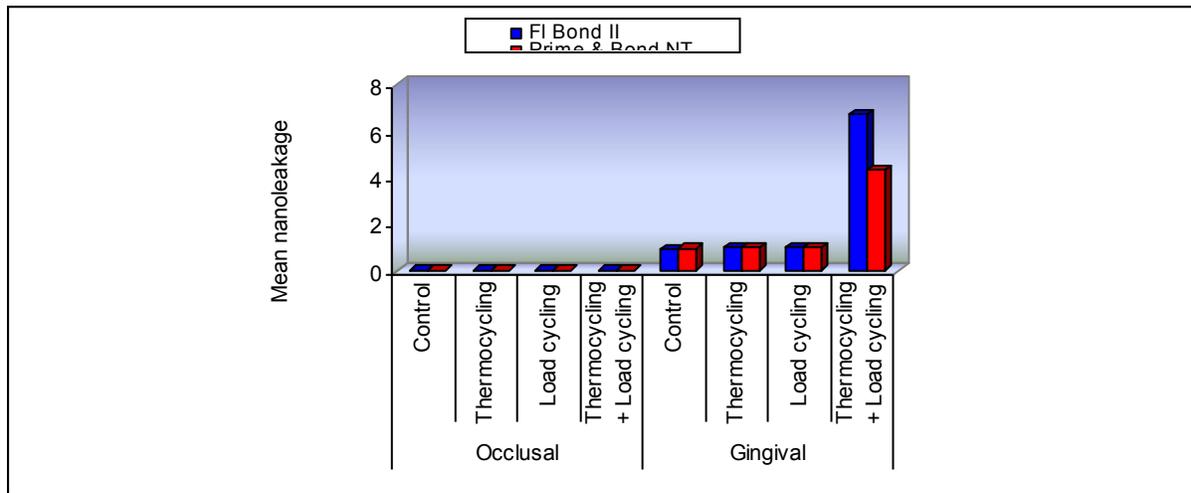


Fig. (1): Bar graph comparing the effect of main variables on the nanoleakage at the occlusal and gingival tooth/restoration interface

Table (3): Comparison between interactions of different subgroups at the gingival tooth/restoration interface.

Bar graph comparing the effect of main variables on nanoleakage at the occlusal and gingival tooth/restoration interface Interaction for Fig. 1	Mean	SD	P-value
FI Bond II x Control	0.93 ^c	0.14	<0.001*
FI Bond II x Thermocycling	0.97 ^c	0.09	
FI Bond II x Load cycling	0.98 ^c	0.12	
FI Bond II x Thermocycling + Load cycling	6.73 ^a	0.50	
Prime & Bond NT x Control	0.95 ^c	0.15	
Prime & Bond NT x Thermocycling	0.97 ^c	0.10	
Prime & Bond NT x Load cycling	0.98 ^c	0.08	
Prime & Bond NT x Thermocycling + Load cycling	4.32 ^b	0.43	

*: Significant at $P \leq 0.05$, Different letters indicate statistically significant differences according to Tukey's test.

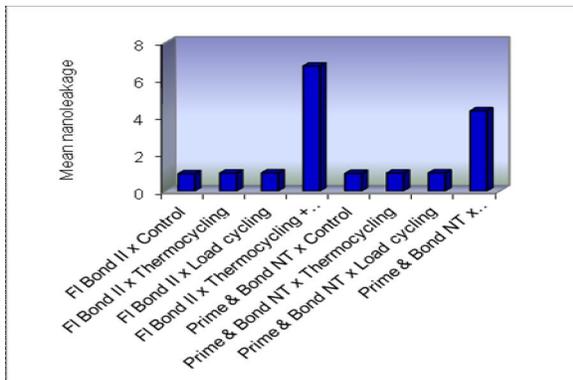
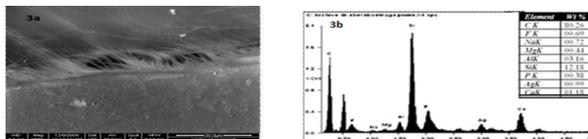


Fig. (2): Bar graph comparing nanoleakage of different subgroups at the gingival tooth/restoration interface.

Figures 3-8 represent SEM micrographs with their corresponding EDAX spectrum. Many shiny spots were observed in all micrographs, however, they did not correspond with silver percentages when analyzed using EDAX. These shiny areas were found to be totally free from silver and comprised only of silica and calcium. The ultra structure of Prime and Bond (fig 2, 4, 6) generally showed a distinctive hybrid layer with long resin tags formation. While, in the FI Bond II subgroups (fig 4, 6, 8), shorter resin tags were observed along the tooth restoration interface.



used, with 5000 cycles being the median value, Abdallah and Davidson(1996); Toledano et al.,(2006). The load was applied on the buccal cusp incline to ensure the same type of stresses that induced on lesions at the cervical area, Jang et al.,(2001).

Pooling of the data from occlusal and gingival walls revealed that, the nanoleakage pattern of each of the tested bonding systems was not influenced by the cycling load (table 2 and Figure 1). These results were in agreement with Li et al.,(2002), who used the same load and the same number of load cycles but with different direction. Similarly, although Mitsui et al.,(2003) and Bedran-de-Castro et al.,(2004), used different load values, different number of cycles and different load direction, they found that load cycling did not affect the microleakage or the nanoleakage pattern. This could be attributed to that, the effect of mechanical loading in adhesive restorations may be transient, taking place only at the moment of load application. After that, cusps would recover their initial configuration, thus resulting in marginal sealing that do not differ from those of the unloaded teeth, Mitsui et al.,(2003). Frankenberger et al.,(2005) also stated that the incidence of nanoleakage remained stable or was even reduced with increasing load cycles. Since, with increasing the number of cyclic stresses, the initial water that diffused into the free volume of the resins may squeeze out of the interface over time, resulting in a general reduction in nanoleakage.

However, the results of the present study were in contradiction with other researches which recorded an increased microleakage under load cycling. Such variation in findings may be due to difference in load direction or difference in materials used as some adhesive systems were adversely affected by load cycling, while others could withstand such a force, Abdalla and Davidson (1996). It is also important to note that most studies used axial compressive loading to stress on margins of restorations, Davidson and Abdalla(1994) ; Abdalla and Davidson(1996). While others applied load on the buccal side to ensure the same type of stresses that induced on lesions at the cervical area, Jang et al(2001). Also, many studies used different value and number of load cycles.

The bonded specimens were subjected to thermocycling, in order to evaluate the durability of the bond. Thermocycling is applied to introduce an artificial aging parameter that would allow evaluation of the temperature dependent degradation of the materials, Uno et al.,(2004). It has been reported that hot water may accelerate hydrolysis of the interface components with subsequent water uptake and leaching of the breakdown products or poorly polymerized resin oligomers. In addition, thermal cycling may induce stresses between the tooth substrate and the restorative material, Asaka et al.,(2007). These stresses may lead

to cracks that propagate along bonded interfaces, accelerating chemical degradation of the bonds. Numerous investigations have used this technique for providing laboratory simulations of oral conditions. The concept is to subject the bonded interface to mechanical stresses generated by the different thermal conductivities and coefficient of thermal expansion of the substrates and bonded materials. Thermocycling of 500 cycles (5-55°C) was selected for this study. According to the ISO TR 11450 standard (1994), this regimen is considered an appropriate artificial aging test.

In the present study, the use of thermocycling alone did not significantly affect the degree of silver penetration at the tooth/restoration interface regardless of the adhesive type. These results were in agreement with Dörfer et al.,(2000) and Bedran-de-Castro et al.,(2004) although they used different number of thermal cycles. The lack of change in the nanoleakage after the introduction of thermal stresses in this study can be attributed to the low C-factor since the giomer was bonded to wedge-shaped Class V cavities and the use of oblique incremental packing technique, resulting in slight contraction stress at the bonding interface. When examining the effect of thermocycling on bond strength, Asaka et al.,(2007) found that, 10000 thermal cycles had no influence on the bond strengths of single-step self-etch adhesive systems. Meanwhile, they found that, raising the number of thermal cycles up to 20000 decreased the bond strength significantly. On the other hand, Holderegger et al.,(2008) found that, thermocycling affected the bonding performance of tested materials. Such variation in their findings may be due to difference in materials used as some adhesive systems were adversely affected by thermal cycling, while others could withstand such thermal stresses.

Regardless of the significant nanoleakage in the gingival walls of both bonding systems, the significantly highest silver penetration showed by FI Bond II compared to Prime & Bond NT adhesive when subjected to lateral load cycling followed by thermal cycling suggests the inadequacy of the bond provided by this adhesive. It may have resulted from weak self-adhesive ability of the self-etch adhesive. Since, the FI Bond adhesive system belongs to the category of "mild" self-etch primers, Deliperi et al.,(2006). The tensile strains that were created by lateral loading, concentrated at the line angle of CEJ, Palamara et al.,(2006) may result in mechanical failure of cervical restorations, Ichim et al.,(2007) due to weakening of the bond at the gingival margins. Subsequent application of thermal cycling may lead to deterioration of the filler-resin matrix interface which may occur rapidly following water sorption, Shin and Drummond (1999) upon the subsequent formation of siliceous hydrogels around the ion-leachable glass fillers,

Ikemura et al.,(2003). This may explain the silver uptake within the interfaces, which can be attributed to lack of frank resin tags in the self-etch adhesive seen in fig. 3,5 and 7.

The overall results revealed that, silver deposition was found only in the gingival walls of both tested adhesives, whereas, no silver deposition was found along the occlusal walls of tested adhesives. This can be attributed to the presence of adequate thickness of enamel in the occlusal margins which might be the contributing factor for successful bonding. Since, there was difference in composition between enamel, dentin and cementum. Whereas enamel is highly mineralized tissue, dentin is a hydrated tissue due to the presence of interconnected maze of fluid filled dentinal tubules and cementum have higher organic and water content than dentin. Therefore, bonding to enamel is a relatively simple process without major technical problems, thus providing adequate marginal sealing with almost no nanoleakage. While bonding to dentin or cementum, on the other hand, presents a much greater challenges, thus demonstrating highest leakage, Pioch et al.,(2001).

Regarding Prime & Bond, etching enamel using the etching time that was recommended by the manufacture was enough to produce decalcified area in order to give adequate resin tags. Meanwhile, the tested self-etch adhesive, although it has “mild” self-etch primer and the resin tags is not as deep as that with phosphoric acid etching, it has no silver deposition occlusally. This can be explained that, nano-retentive interlocking is created between enamel and resin resulting in increased bonding effectiveness. The bonding effectiveness may result from micromechanical and chemical interaction with tooth substrate; the chemical component may be able to compensate for the decreased micromechanical interlocking, Van Meerbeek et al.,(2001and2003). The Fl Bond system contained 4-AET (4Acryloxyethyltrimellitic acid), which can interact with the calcium cations of hydroxyapatite to form 4-AETCa, a relatively insoluble calcium salt that improved durability of the adhesive system, Ikemura et al.,(2002). Accordingly, this restorative material is capable of bonding chemically, as well as, micromechanical to enamel and dentin.

The results of the present study was in agreement with Dörfer et al.,(2000), who found that, no specimen found without dye penetration within the hybrid layer at dentin, while no penetration of the fluorescent dye was found at the enamel/composite interface. Furthermore, Owens et al.,(2006), revealed that the tested adhesive systems showed significantly higher leakage at the dentin margins.

Under the circumstances of this study, the following conclusions could be derived:

The type of the tested adhesive system affected the sealing ability of the giomer restorations. Nanoleakage was significantly increased when combining lateral-load and thermal cycling, in an attempt to simulate oral conditions.

5.References:

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