BONDING OF CONTEMPORARY ADHESIVES TO OZONE-TREATED DENTIN SURFACES

Adesão de adesivos contemporâneos em superfícies dentinárias tratadas com ozônio

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Abstract

OBJECTIVES: To evaluate the effect of Ozone application on the shear bond strength of different types of contemporary adhesive systems to dentin. **MATERIALS AND METHODS**: Three of the contemporary adhesive systems representing different generations (Exite, Adper Prompt L-pop and G Bond) were used to bond standardized Tetric Ceram composite build-ups (3mm in diameter and height) either to Ozone-treated (Groups I, IV and VI) or non-treated (Groups II, V and VII) flat sound dentin surfaces. Another test group (Group III) was also existed when the Ozone was applied against etched dentin before applying the Exite single-component adhesive. Some of the prepared specimens were tested for shear bond strength and others were sectioned for SEM examination. The debonding patterns of the fractured specimens were also assessed using steromicroscope. **RESULTS**: Ozone treatment of dentin surfaces showed no significant effect on the bond strength of different adhesives to dentin (Tukey's comparisons, $p<0.05$). The only exception was registered when the gas was applied to the already etched dentin surfaces and before the application of Exite single-component adhesive (Tukey's comparison, $p= 0.000917$). The microscopic finding announced that the adhesive debonding was the most common mode of adhesive/dentin bond failure. The existence of narrow adhesive junction was observed in the SEM images of specimens bonded with G-bond. Narrow hybrid layer and short resin tags characterized the adhesive junction of specimens subjected to Ozone treatment after dentin etching and before applying the Exite adhesive. **CONCLUSIONS**: Ozone application seems to have no effect on the bond strength of contemporary adhesives to dentin. However, in case of using 2-step adhesives, the application of this gas should be performed before the dentin-etching step.

Keywords: Ozone gas; Bonding to dentin; Adhesives; Composites; Bond strength.

Resumo

OBJETIVOS: *Avaliar o efeito da aplicação de ozônio na força de adesão de diferentes tipos de sistemas adesivos contemporâneos à dentina.* ***MATERIAL E MÉTODO***: *Três sistemas de adesivos contemporâneos, representando diferentes gerações (Exite, Adper Prompt L-pop e G Bond), foram usados na adesão de corpos de prova de compósito Tetric Ceram (3 mm de diâmetro e altura), em dois grupos: superfícies dentinárias tratadas com ozônio (Grupos I, IV e VI) e não tratados (Grupos II, V e VII). Em outro grupo de teste (Grupo III) o ozônio foi aplicado em dentina tratada antes da aplicação do adesivo mono componente Exite. Alguns dos espécimes preparados foram testados para resistência ao deslizamento e outros foram secionados para exame em microscopia eletrônica. Os padrões de descolamento dos espécimes fraturados foram determinados com o uso de estéreomicroscópio.* ***RESULTADOS***: *O tratamento com ozônio das superfícies dentinárias não demonstrou efeito significativo na resistência de adesão de diferentes adesivos à dentina (comparação de Tukey p<0.05). A única exceção foi registrada quando o gás foi aplicado nas superfícies previamente tratadas com ácido e antes da aplicação do adesivo monocomponente Exite. (Tukey p+ 0.000917). O achado microscópico demonstrou que os descolamentos foram o modo mais comum de falha de adesão à dentina. A existência de junção adesiva estreita foi observada na microscopia eletrônica em espécimes colados com G.bond. Camada estreita híbrida e tags curtos na dentina caracterizaram a junção de adesão de espécimes sujeitos ao tratamento com ozônio após tratamento da dentina e antes da aplicação do adesivo Exite.* ***CONCLUSÕES***: *A aplicação de ozônio parece não causar efeito na força de aderência de adesivos contemporâneos à dentina. Entretanto, no caso de uso de adesivos de duas etapas, a aplicação do gás deveria ser feita antes do tratamento da dentina.*

Palavras-chave: *Gás ozônio; Adesividade à dentina; Adesivos; Compósitos; Resistência de adesão.*

INTRODUCTION

The Ozone gas is a colorless form of oxygen with powerful oxidizing potential. Applying this gas in specified concentrations seems to have high killing effect against different strains of bacteria (1-3) and could be used to disinfect several items accordingly (4, 5). The use of Ozone has been recently introduced to the field of dentistry (6, 7). The gas was usually recommended to destroy caries-inducing organisms in root and incipient carious lesions, and following certain regimen, tooth remineralization could be happened (8-12). However, sterilizing root canals before final obturation and treating some suppurative lesions have recently been demonstrated using Ozone gas or even Ozonated water (2, 3, 13-16).

Caries recurrence is occasionally detected beneath different kinds of restorations. Several attempts have taken place to overcome that problem including the use of antimicrobial adhesives and cement materials. The application of Ozone has been recently proved as an efficient measure for eliminating the ecological niche of microorganisms (1) and accordingly its use before bonding such restoration was also postulated to minimize the chance of caries recurrence (17-18).

The advances in chemical technology have served in developing several generations of adhesive resin systems over a short period of time. Each of these generations has its specific characters those ensure strong and durable bonds to the tooth structure (19-21). At the same time, reducing the number of application steps of such adhesive was also considered aiming to save patients' and dentists' times and to minimize the sensitivity of the application technique (22).

The aim of the current study was to evaluate the effect of topical Ozone gas application before bonding final restorations on the shear bond strength of some contemporary adhesives to human dentin.

MATERIAL AND METHODS

Thirty-five caries-free human molars were collected at The Department of Oral Surgery, Gizan Dental Center, KSA. The teeth were subjected to thorough scaling (P5 Brooster suprasson, SATALEC, Merignac, France) and ultrasonic cleaning (Sultan Chemists, Englewood, NJ) to remove any hard deposits or soft tissue remnants then stored for less than 2 months in 0.1% Thymol solution (23) Both buccal and lingual surfaces of the collected molars were prepared using Edenta diamonds (# 837.012, Edenta GmbH, Lustenau, Austria) to expose flat dentin surfaces on which composite build-ups (TetricCeram, Ivoclar Vivadent, Schaan, Liechtenstein) were bond using different adhesive systems representing three successive generations as shown in Table 1.

TABLE 1 - Materials used

Material	Description	Composition	Manufacturer
I. Adhesive systems:			
G-Bond	7th generation, One-step Self-etch (no mix) adhesive	phosphoric acid ester monomer, UDMA, 4-MET, TEGMA, acetone, water, initiators	GC America, St. Alsip, IL
Adper Prompt L-Pop	6th generation, One-step Self-etch (no mix)	Compartment 1 : Methacrylated phosphoric acid esters, photoinitiator, adhesive Compartment 2 : Water, HEMA, polyalken oic acid, stabilizers	3M/ESPE Dental Products, St. Paul, MN stabilizers
Exite	5th generation, Alcohol-based, 2- step adhesive	HEMA, DMA, phosphoric acid acrylate , highly dispersed silicone dioxide, initiators and stabilizers in alcohol solution	Ivoclar Vivadent, Schaan, Liechtenstein
II. Restorative material:			
Tetric Ceram	Light-curing, fine-particle hybrid resin composite	BisGMA, UDMA, TEGDMA, ytterbium trifluoride, barium glass , pigments and initiators	Ivoclar Vivadent, Schaan, Liechtenstein

After cutting the roots off, teeth crowns were sectioned in mesiodistal direction resulting in 70 halves using diamond disks (Edenta, Lustenau, Austria) in low speed straight handpiece. Each half was then held with its flat dentin surface facing up in a plastic ring 3 cm in diameter by the aid of chemically polymerized acrylic resin (Duracrol, Sofa-Dental, Prague, Czech Republic). The dentin specimen were then classified into 7 groups (n=10) according to the study design shown in Table 2.

TABLE 2 - Levels of the study

Groups	Adhesive system	Dentin surface treatment
I (Control)	Exite	No Ozone treatment
II	Exite	Ozone treatment has been carried out before dentin etching
III	Exite	Ozone treatment has been carried out after dentin etching and before the application of liquid adhesive
IV	Adper Prompt L-Pop	No Ozone treatment
V	Adper Prompt L-Pop	Ozone treatment has been carried out before applying the self-etching, one-step adhesive
VI	G-Bond	No Ozone treatment
VII	G-Bond	Ozone treatment has been carried out before applying the self-etching, one-step adhesive

Treatment of dentin surfaces

The dentin surfaces of group I specimens were directly etched using 37% phosphoric acid (Vivadent, Schaan, Liechtenstein) for 15 s and left moist after washing the acid out. A custom-made silicone covers 3 mm in height with a central mold 3 mm in diameter were firmly adapted to the top surface of the mounting rings in order to confine both the adhesive resin and the further composite build up to the exact bonding area. The Exite adhesive was then applied using a brush for 10 s and cured for 20 s using Hawe-Neos light-curing device (Gentilino, Switzerland) that delivers intensity of 550 mw/ Cm^2.

The dentin surfaces of group II were first subjected to Ozone treatment by the aid of the Helozone delivery system (Kavo Dental Excellence, Bibrach/Riss, Germany) before performing the acid-etching procedure. The Helozone apparatus pulls air at 615cc/min and delivers ozone at a concentration of 2100 ppm ± 10%. The ozone gas was applied to the flat dentin surfaces for 40 s followed by 20 s of suction to recollect the gas and eliminate surface contaminants. The silicone cups attached to the Helozone hand piece helped in defining the area of dentin to be treated and provided the seal desired during the application-suction cycle. The Dentin surfaces were then etched and washed before applying the Exite adhesive as previously described. The dentin surfaces of group III were received their Ozone treatment after performing the acid-etching procedure and immediately before the applications of Exite (Vivadent, Schaan, Liechtenstein) single-component adhesive.

Adper Prompt Adper Prompt L-pop (3M ESPE, St. Paul, MN) one-step self etching adhesive was applied on the exposed dentin immediately after fixing the silicone mold with no Ozone treatment of group IV surfaces. The adhesive was left for 10 s then light cured for another 10 s. Exactly the same application procedures were followed using the same adhesive against the ozone-treated dentin surfaces of group V. G-Bond (GC America, St. Alsip, IL) one-step self-etching adhesive was applied against the exposed non-treated dentin surfaces of groups VI for 10 s and light cured for 10 s, while in group VII the same adhesive brand was applied against the Ozone-treated dentin following the former procedure.

Composite building up and shear bond strength testing

Tetric Ceram composite cylinders 3mm in diameter and height were incrementally built up against the cured adhesive by the aid of those silicone molds. Each composite increment was 1.5 mm high and light cured for 40 s. All specimens were finished using #15 carbon steel surgical blades (Wuxi Xinda medical Device) excising only the composite flashes (if any) existed at the bonding interface. Twenty four hours after preparation, all specimens' rings were held horizontally on the lower member of the Lloyd universal testing machine (Type 500, Lloyd Instruments, London, UK) and the bonding interfaces were stressed to failure under shearing force applied by the aid of knife edged plate running at crosshead speed of 2mm/min (Figure 1). The shear bond strength of each specimen was calculated by dividing the maximum force at failure by the bonding surface area (equivalent to the cross-section of the composite build-up) and the mean of each group was then calculated.

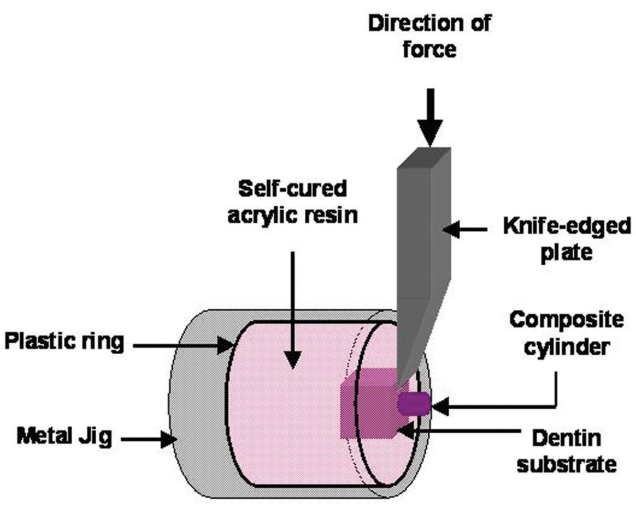

FIGURE 1 - Performing the shear test

BOTTOM JIG

Both parts of each debonded specimen were also assessed using a stereomicroscope (Olympus Zoom Stereomicroscope, Tokyo, Japan) at 30 X original magnification to detect the mode of bond failure. The detected modes of failure were classified as adhesive, when the fracture was entirely at the adhesive/tooth or adhesive composite interface; cohesive, when the fracture was exclusively within the resin composite or dentine; or mixed when the fracture site continued from adhesive into either dentine or composite.

SEM examination

One molar from each group with its composite build-ups was randomly selected and half-sectioned bucco-lingually across the bonding surface. The cut surfaces were then lightly finished using a sequence of 600, 800, and 1000-grit abrasive papers (SAITAC Abrasive, Torino, Italy) in presence of water-cooling followed with light polishing with diamond paste. The specimens were then immersed in 6N (mol/L) HCL for 45 seconds, rinsed with distilled water, dried for 3 h at 37 ± 0.1°C in Binder Incubator (Type B28, Tuttlingen, Germany) and then left at room temperature for 24 hours. The dried specimens were sputter coated with gold-palladium (JCF, 1100e, Sputter Coating Device, Tokyo, Japan) before assessing the dentin/adhesive interface of each nearly at 2000 X original magnification using a scanning electron microscope (JEOL, 5300, 19kV, Tokyo, Japan).

RESULTS

Mean shear bond strength values of different test groups are presented in Table 3. Statistical analysis of the results indicated a difference between the test groups (ANOVA, p = 1.906 E-5).

TABLE 3 - Shear bond strength in MPa of different adhesives to dentin surfaces before and after Ozone application

Test groups	I (cont)	II	III	IV	V	VI	VII
Bond strength	24.69	23.8	20.84*#	21.78*	20.12*	22.18	22.04
S.D.	± 0.97	± 0.74	± 1.03	± 3.35	± 2.77	± 1.48	± 1.74

One-way ANOVA table

	S Sq	DF	M Sq	F-ratio	Probability
Between groups	151.381	6	25.2302	6.599	1.906 E-5
Within groups	240.865	63	3.82325		
Total	392.246	69			

* Significantly different values from the control (Tukey's comparisons, p< 0.05)
\# Significantly different values from those registered for the same adhesive system under different test conditions (Tukey's comparisons, p< 0.05)

Tukey's comparisons revealed no significant difference (p>0.05) between Group II values (Exite bonded to Ozone-treated dentin) and the control (Group I, Exite bonded to untreated dentin surface). Group III specimens (Exite bonded to receiving Ozone treatment after etching) showed significant lower values than those of both groups I and II values (P= 0.000917 and 0.02351). Groups IV and V (Adper Prompt L-Pop adhesive bonded to non-treated and Ozone–treated dentin) showed significantly lower shear bond strength values in comparison to the control (Group I) (p= 0.02351 and 0.000166). The shear bond strength of group VI and VII (G-Bond bonded to dentin with and without ozone–treatment) showed no significant differences from that of the control (Group I).

Microscopic bond failure analysis (Figure 2) revealed adhesive kind of failure for the majority of cases.

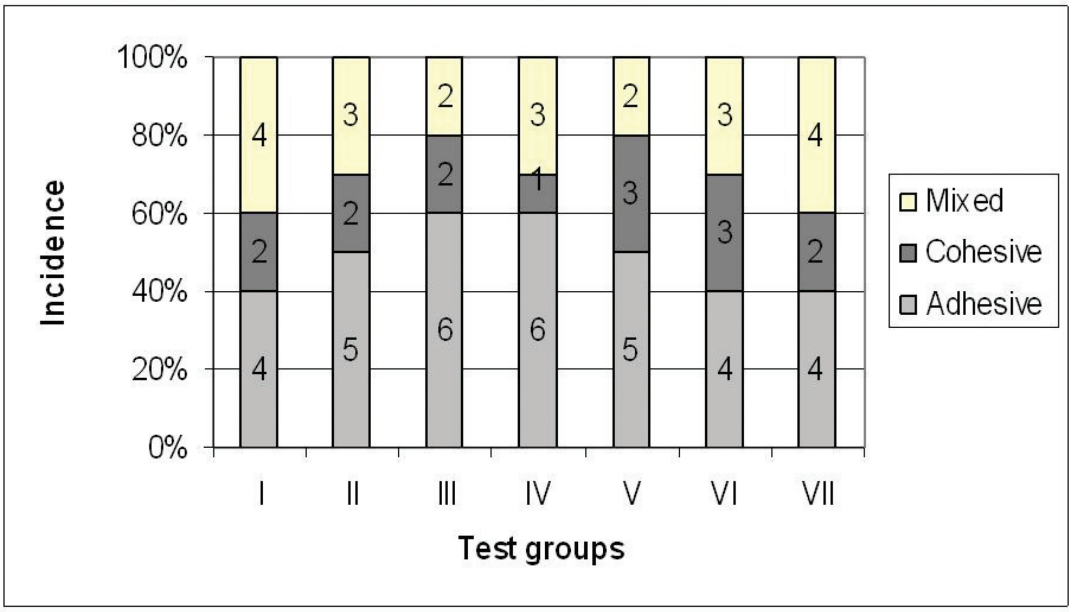

FIGURE 2 - Incidence of modes of bond failure

However an increased incidence of mixed type of failure seemed coincide with the increase in bond strength. SEM images indicated the formation of regular hybrid layer in specimens bonded either with Exite or Adper Prompt Adper Prompt L-pop adhesive (Groups I, II, IV and V) (Figure 3a and 3c). However, shortening in the resin tags together with the reduced thickness of hybrid layer were characterizing for the adhesive junction of Group III specimens those subjected to Ozone treatment after performing the etching procedures and before the application of Exite adhesive (Figure 3b). The existence of narrow adhesive junction (Figure 3d) was characteristic in the SEM images of specimens bonded with G-bond adhesive system (Groups VI and VII).

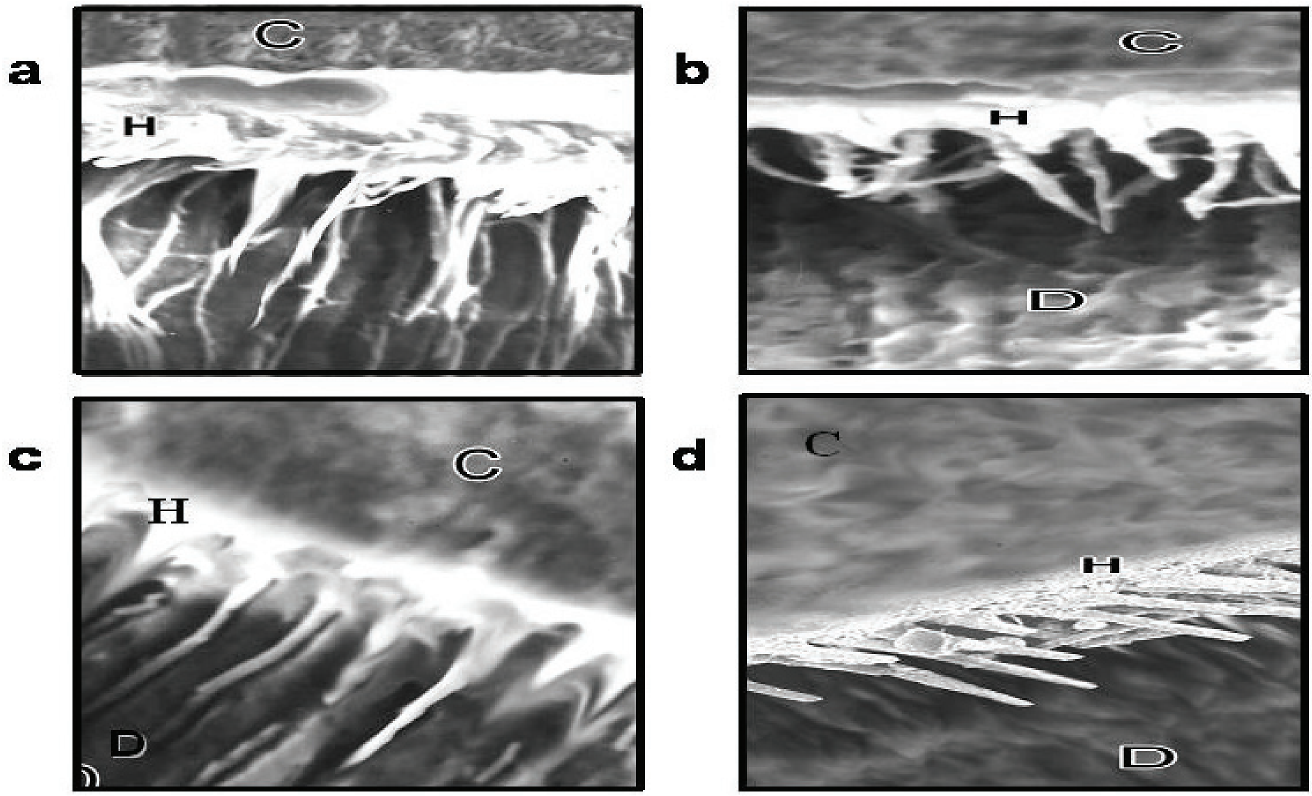

FIGURE 3 - SEM photomicrographs 3a, 3b e 3c

DISCUSSION

Caries recurrence could be detected beneath different kinds of dental restorations (14-16) and accordingly application of Ozone gas has been recently suggested before restoring the prepared tooth cavities (13). Testing the bond strength either in shear or tension usually provide useful information on the adhesion between different materials , however the shear bond test is mainly used to compare the relative effect of materials' properties and microstructure, and the treatment conditions on the performance of the formed bond at the time of load application (24-26).

Accordingly, the current in vitro study aimed to evaluate the shear bond strength of contemporary adhesives to Ozone -treated and non-treated dentine surfaces. The results indicated some variation in the bond strength values of different adhesives. Adper Prompt L-pop adhesive system appeared to be of lower bond strength in comparison to the control (Group I, Exite-no treatment). This finding could exist because of the impurity of the etching component, as they mixed with the other resin component of the system at the time of application, and the insufficient time contacting the sound dentin surfaces (21). Those contributing factors might result in poor etching and therefore improper interaction zone (adhesive junction) (Figure 3c). Moreover remaining of both smear and etching debris within the structure of the polymerized adhesive resin could reduce its mechanical properties and accordingly weaken the adhesive junction (21-27). Probably the fancy chemical structure of the G-bond and its ability to form a nano-interaction zone (Figure 3d) are responsible for the recorded insignificant difference in bond strength values when compared to the control (Group I) (28). The existence of the very thin adhesive junction provides a negligible effect of the adhesive's mechanical characteristics on the success of the adhesive junction (24) and this was very obvious from the results of failure mode assessment (Figure 2).

It is also obvious that Ozone application has no significant effect on the shear bond strength of all adhesives under investigation when it is applied before performing the dentin etching step. This finding coincides with that of Schmidlin et al. (18) and could be explained as follows; applying the etching acid alone or in combination with other adhesive ingredients (as in case of Adper Prompt L-pop and G-bond) cleans the tooth surfaces in contact and improves their critical surface tension (21). These actions accordingly improve the wettability of the hydrophilic resin adhesive to dentin. At the same time, the etching process creates micro-irregularities and opens the orifices of dentinal tubules which increase the bonding surface area and provide a sort of micro-mechanical attachment of adhesive to dentin. Washing and gentle drying of etched surface (in case of Exite) is going to leave moist collagen fibril network that allow the inter-penetration of adhesives and the creation of what is known by hybrid layer (Figure 3a)(21, 27).

On the other hand, application of Ozone after etching could enrich the area with oxygen that normally inhibits the polymerization of the subsequently applied adhesive resin (27). At the same time, suction of the applied Ozone and the derbies back to the machine could cause collapse of the collagen fibrils. These postulations could explain the significant reduction in Exite's bond strength values for Group III specimens when Ozone was applied to the etched dentin surface.

Evaluation of the effect of both cyclic loading and thermocycling on the adhesive performance of contemporary adhesives to Ozone-treated enamel and dentin surfaces is recommended in further investigations. The assessment of leakage around and within the formed adhesive junction for sure will be of great value.

CONCLUSION

Both Exite and G-bond adhesive systems exhibit comparable shear bond strength values. Ozone application seems to have no effect on the bond strength of contemporary adhesives, used in this study, to dentin. However, in case of using 2-step adhesives, the application of the Ozone gas should be performed before the dentin-etching step.

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Received: 09/20/2007
Recebido: 20/09/2007

Accepted: 10/25/2007
Aceito: 25/10/2007