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ORIGINAL ARTICLE / ÖZGÜN ARAŞTIRMA

Effect of Immediat Dentin Sealing on the Bonding State of Hybrid Ceramic CAD/CAM Restorative Material to Dentin

İmmediat Dentin Kapama Prosedürünün Hibrit Seramik CAD/CAM Restoratif Materyalinin Dentine olan Bağlantısına Etkisi

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Abstract

Objectives: Lately, computer-aided design (CAD) computer-aided manufacturing (CAM) systems with simplified procedures for indirect restoration have gained rapid improvements and commenced to be used in daily practice of dental clinicians. The aim of this pilot in vitro study was to analyze the bonding efficiency and observation of the failure mode of hybrid ceramic CAD/CAM block materials after termo-cycling, with different immediate dentin sealing (IDS) techniques.

Materials and Methods: Eighteen freshly extracted human molars were selected, and mounted with acrylic resin, fixed the root up to 2.0 mm under the cementoenamel junction. Standardized, flat, nonretentive, midcoronal dentin surfaces were prepared. Using the stratified random sampling process, all teeth (N=18) were divided into groups of three by approximately similar sizes; afterwards, these teeth were randomly distributed into the groups 1, 2 and 3 (n = 6). The specimens from groups 2 and 3 received IDS, whereas delayed dentin sealing was carried out for specimens of control group (Group 1). For IDS materials, universal adhesive system G-Premio Bond (GC, Tokyo, Japan) and highly filled flowable resin composite G-aenial Universal Flo (GC, Tokyo, Japan) were used. All block material specimens were prepared using the cutting instrument (IsoMet 1000; Buehler, USA) and were fitted with a standardized 3x3-mm^3 cubes cutted out of a CAD/CAM block of hybrid ceramic (Cerasmart, GC; Tokyo, Japan). Dual-polymerized resin cement G-CEM Link Force (GC, Tokyo, Japan) was utilized to lute restoration materials for all groups. To test the shear bond strength (SBS) each of the specimens was placed in a jig with 90^o to the vertical plane and tested a universal testing machine (Shimadzu AG-IS; Shimadzu Corp). One-way ANOVA was used to analyze the data of the SBS. Results were evaluated statistically significant for $p<0.05$.

Results: No statistically significant difference revealed by the statistical analysis between Group 1,2 and 3 (p=0,372). This means there is no significant difference in the sealing methods (p>0,05). Bond failure rates showed similar results in all groups, where the most frequent failure pattern detected was 'adhesive type'.

Conclusions: Within the limitation of this in vitro pilot study, the following conclusion was drawn: 2 Different IDS procedures tested does not statistically ($p<0.05$) effect the SBS of hybrid ceramic CAD/CAM material bonded to dentin with G-CEM Link Force.

Keywords: Immediate dentin sealing, hybrid ceramics, shear bond strength

Öz

Amaç: Günümüz diş hekimliği pratiğinde indirekt restorasyonların üretiminde CAD/CAM teknolojisinin kullanımı oldukça yaygınlaşmıştır. Bu pilot çalışmada dentin yüzeylerine uygulanan iki farklı IDS prosedürürünün, hibrit seramik CAD/CAM restoratif materyalin dentine bağlanma dayanımına etkisini değerlendirmek amaçlanmıştır.

Gereç ve Yöntemler: 18 tane yeni çekilmiş büyük azı dişi mine sement sınırının 2 mm altından akrilik rezin içerisine gömülerek sabitlendi. Tüm dişler (N=18) düz dentin yüzeyleri oluşturulacak şekilde prepare edildi. Birbirine yakın boyutlardaki dişler rastgele olmak üzere eşit sayıdaki 3 gruba (n=6) ayrıldı. Grup 2 ve Grup 3 örneklerine IDS tekniği uygulanırken Grup 1 (Kontrol grubu) örneklerine bu teknik uygulanmadı. IDS tekniğinde yüksek miktarda doldurucu içeren G-Premio Bond (GC, Tokyo, Japonya) universal adezivi ve G-aenial Universal Flo (GC, Tokyo, Japan) akışkan kompozit rezini kullanıldı. Hibrit seramik CAD/CAM blok materyal (Cerasmart, GC; Tokyo, Japan) örnekleri kesme cihazında (IsoMet 1000; Buehler, Lake Bluff, IL, USA) 3x3 mm^3 lük küpler halinde hazırlandı. Örneklerin simantasyonunda dual polimerize rezin siman G-CEM Link Force (GC, Tokyo, Japonya) kullanıldı. Makaslama bağlantı kuvvetlerinin hesaplanmasında universal test cihazı (Shimadzu AG-IS; Shimadzu Corp) kullanıldı. Veriler İstatistiksel olarak tek yönlü varyans analizi (ANOVA) kullanılarak değerlendirildi.. Sonuçlar $p<0.05$ için istatistiksel olarak anlamlı kabul edildi.

Bulgular: Farklı immediat dentin kapama prosedürleri arasında dentine bağlantıda istatistiksel olarak anlamlı bir fark

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bulunamamıştır (p>0,05). Bütün örneklerin kopma yerleri optik mikroskopta değerlendirilmiş ve tüm gruplarda en sık 'adeziv kopma' gözlemlenmiştir.

Sonuç: Bu in vitro pilot çalışmanın sınırları dahilinde, düz dentin yüzeylerine farklı immediat dentin kapama prosedürleri uygulanması, tümü G-CEM Link Force ile simante edilen hibrit seramik CAD/CAM materyalinin dentine olan makaslama bağlantı kuvvetini etkilememiştir.

Anahtar Kelimeler: İmmediat dentin kapama, hibrit seramik, makaslama bağlanma dayanımı

Introduction

Lately, computer-aided design (CAD) computer-aided manufacturing (CAM) systems with simplified procedures for indirect restoration have gained rapid improvements and commenced to be used in daily dental clinical practice. In addition, adhesive dentistry has revolutionized our clinical everyday practice in dentistry [1]. The advances in adhesive materials along with progresses in CAD/CAM technologies have increased the durability and reliability of indirect restorations. Indirect restorations are usually the preference of the dental clinician in large cavities, as they are more minimally invasive than full coverage crowns. From a biomimetic perspective, minimally invasive approach is paramount in maintaining the subtle equilibrium between functional, biological, mechanical and aesthetic parameters [2]. By taking all these parameters into account, a harmonious and natural restorative treatments could be accomplished [3].

The immediate dentin sealing (IDS) technique is a succesfull bonding procedure used for indirect restorations [4]. This procedure was introduced by Japanese clinicians in the early 1990s who were professionals in adhesive dentistry [4,5]. IDS procedure has been recommended to enhance the adhesion of indirect restorations to dentin. This procedure provides to decrease post operative sensitivity compared to conventional adhesive luting also called as delayed dentin sealing (DDS) [5,6]. The IDS suggested that a bonding agent be applied when remarkable dentin tissue has been exposed during preparation for indirect restorations (crown, onlay/inlay and veneer preparations) before taking the impression [7]. Dentin adhesive can combined with a flowable composite when sealing exposed dentin surfaces. However, the effectiveness of IDS with dentin bonding agents (DBA) and flowable resin composites on hybrid ceramic CAD/CAM block materials has not been investigated. The main difference between the DDS and IDS procedure lies in the fact that with the IDS, DBA is applied freshly prepared dentin, while in the DDS, the DBA is applied just before inserting the final restoration [8]. IDS has many favorable results starting with the lesser gap formation, enhancement bond strength, maximum conservation intact tooth structure and better adaptation of the restoration [9-13]. In addition, it prevents bacterial leakage or any dentin contamination and thus induces decreased postoperative sensitivity [14-16].

The first null hypothesis was that the application of IDS procedure will not improve the SBS of hybrid ceramic luted to dentin. The second null hypothesis was that the application of bonding agent combining with flowable composite for IDS procedure will not effect the SBS. The aim of this pilot study was to investigate the SBS and observation of the failure mode of hybrid ceramic CAD/CAM block materials after termo-cycling, with/without IDS (combined with/without flowable composite).

Materials and Methods

Eighteen freshly extracted free of restorations, fractures, caries and root canal treatment sound human molars were stored in 0.1% thymol solution less than 6 months after extraction during the test period (< 6 months). The Ethics Committee of the Marmara University Faculty of Dentistry approved this study (2018-232).

Specimen preparation

The information about the materials used in this study was listed in Table 1. Teeth were mounted with acrylic resin, fixed the root up to 2.0 mm under the (CEJ). The same operator performed all the procedures. Using the stratified random sampling process, all teeth (N = 18) were divided into groups of three by approximately similar sizes; afterwards, these teeth were randomly distributed into the test groups 1, 2 and 3 (n = 6). Standardized, flat, nonretentive, deep dentin surfaces were prepared without opening the pulp chamber using a round-ended tapered diamond rotary cutting instrument (IsoMet 1000; Buehler, Lake Bluff, IL, USA).

Immediate Dentin Sealing

Once the preparation was completed, the specimens from groups 2 and 3 received immediate dentin sealing, whereas delayed dentin sealing was carried out for specimens group 1.

Group 1: Delayed dentin sealing (Control Group)

Group 2: IDS with all in one adhesive

Group 3: IDS using all in one adhesive combined with flowable composite

For IDS materials, universal adhesive system G-Premio Bond (GC, Tokyo, Japan) and highly filled flowable resin composite G-aenial Universal Flo (GC, Tokyo, Japan) were used. In the Group 1 the IDS technique was not used and dentin was not sealed after the preparation, whereas in Group 2 and Group 3 an IDS technique was used and freshly cut dentin surface was immediately sealed after the preparation. Group 2 and Group 3 differed in the IDS protocol. Group 2 specimens sealed with only universal adhesive, whereas Group 3 specimens sealed with combination of universal adhesive with flowable resin composite. In the Groups 2 and 3, dentin surfaces were etched 15 seconds with 37% phosphoric acid (GC Etchant, GC; Tokyo, Japan), rinsed with water for 15 seconds and gently air dried without desiccation. The universal adhesive G-Premio Bond was applied on the etched dentin surfaces according to manufacturer's instructions: after application of the bonding agent, waiting for 10 seconds and using maximum air pressure for 5 seconds with oil-free air spray. Bonding agent, was then 10 seconds light-cured at 1400 mW/cm^2 (Valo; Ultradent, USA) with an extra 10 seconds under the glyserine gel for air blocking to diminish the oxygen-inhibition layer. Thereafter for the Group 3 specimens, highly filled flowable resin composite G-aenial Universal Flo was applied to the pretreated dentin surface; it was spread with a small brush to maintain a uniform thickness and then all the specimens light-cured for 20 seconds at 1400 mW/cm^2 (Valo; Ultradent, USA) with an extra 10 seconds under the glyserine gel to diminish the oxygen-inhibition layer. Glyserine gel was rinsed until the surface was clean (Johnson&Johnson, France).

All specimens were restored with temporary restoration material (GC Revotek LC; Tokyo, Japan) and then stored in distilled water at 37^{o}C for 1 week.

Preparation of the CAD/CAM block materials

All specimens were prepared using the cutting instrument (IsoMet 1000; Buehler, USA) and were fitted with a standardized 3x3-mm^3 cubes cutted out of a CAD/CAM block of hybrid ceramic (Cerasmart, GC; Tokyo, Japan).

Adhesive cementation

Following the 1-week storage, the temporary restoration was removed with an ultrasonic tip and a scaler. The dentin surface and indirect restoration should be pretreated individually according to manufacturer's indicated method.

A slurry of pumice and water were used to clean all tooth surfaces.. In Group 1 dentin surfaces were etched with 37% phosphoric acid (GC Etchant, GC; Tokyo, Japan) for 15 seconds, rinsed for 15 seconds and gently air dried without desiccation. In Groups 2 and 3 the IDS layers were checked for stability using magnification after tribochemically treated (CoJet Sand, 3M ESPE) for 4 s with noozle angle of 45^o, 2 bar pressure from distance of 10 mm using a chairside air abrasion device (Dento-PrepTM, Daugaard, Denmark), but no detrimental effects were occured in any of the specimens. IDS layers was chemically cleaned with GC Etchant for 15 seconds, rinsed and air-dried. Subsequently, for dual cure mode of the G-CEM Link Force, the universal adhesive G-Premio Bond and G-Premio DCA were mixed in 1:1 portions and applied to the all preparation surfaces using a microbrush according to manufacturer's instructions: after application of the dentin bonding agent waiting for 20 seconds and using maximum air pressure for 5 seconds with oil-free air spray.

All Cerasmart (GC, Tokyo, Japan) restoration materials were sandblasted with 50-µm aluminum oxide particles approximately 10 mm from the surface at 3 bar pressure for 15 sec (Korox, Bremeen, Almanya). Afterward, GC Multi Primer was applied for silanization of the sandblasted restoration surfaces and thinned with dry air spray.

Dual-polymerized resin cement G-CEM Link Force was used for luting the restoration materials for all specimens. Resin cement applied to the sandblasted restoration surface. Soon after, the restoration material was pressed firmly on preparation surface of the tooth. Slightly hardened excess cement was carefully cleaned with microbrushes and cotton pellets, and then photo-polymerized from the four directions of cube material for 20 s each, using 1400 mW/cm^2 (Valo; Ultradent) and this polymerization process was repeated after the application of glyserine gel.

Thermo-cycle Procedure

All specimens were thermocycled with 30 seconds dwell time at 5^{o}C to 55^{o}C temprature and a total of 5500 thermal cycles at a frequency of 2.4 Hz was used (SD Mechatronik Thermocycler, Germany).

Shear Bond Strength Test

To test the SBS, each of the samples was individually placed in a jig with 90° to the vertical plane and tested a universal testing machine (Shimadzu AG-IS; Shimadzu Corp) utilizing a 1 kN load cell with a range set at 0-100 newtons. 1mm/min test speed was used. The hybrid ceramic restoration materials were subjected to shear forces applied at 1 mm front from the tooth/restoration material limit. Recorded shear bond force in newtons was converted to megapascals (MPa) to symbolize bond strength.

After testing the restorative material and dentin surface of whole samples were evaluated under 100x light magnification (Leica DC-100, Meyer Instruments, USA). All samples photographed to evaluate the failure mode. According to the failure location, it was classified as cohesive in dentin, adhesive, mixed or cohesive in the hybrid ceramic block.

Statistical analysis

One-way ANOVA was used to analyze the data of SBS. Results were evaluated statistically significant for $p<0.05$. Kolmogorov-Smirnov test was used to control whether the data distribution was normal and the homogenity of variance was controled by Levene test.

Table 1. Information about the materials used in this study

Brand	Type	Manufacturer	Composition
G-Premio Bond	Universal adhesive	GC Corporation, Tokyo, Japan	MDTP, 4-MET, MDP, acetone, photoinitiators, water, dimethacrylate monomers, silicon dioxide
G-ænial Universal Flo	High filled flowable resin composite	GC Corporation Tokyo, Japan	Dimethacrylate monomers, pigments, bis-EMA, silicon dioxide, UDMA, fillers, photoinitiators
G-CEM LinkForce	Dual-cure resin cement	GC Corporation, Tokyo, Japan	Paste A:UDMA, dimethacrylate monomers, bis-GMA, fillers, photoinitiators, pigments Paste B : fillers, bis-EMA, UDMA, photoinitiators, dimethacrylate monomers
G-Multi Primer	Primer for alumina, glass ceramics, metal bonding, hybrid ceramics, composites, zirconia	GC Corporation, Tokyo, Japan	Phosphoric ester monomer, Ethanol, Methacrylate monomer, γ-Methacryloxypropyl trimethoxysilane
GC Etchant	%37 phosphoric acid etching gel	GC Corporation, Tokyo, Japan	Silicon dioxide, phosphoric acid (37%), colorant
GC Cerasmart	Hybrid ceramic CAD/CAM block	GC Corporation, Tokyo, Japan	UDMA, pigments, bis – EMA, dimethacrylate monomers, silicone dioxide, initiator, barium glass powder

Results

No statistically significant difference revealed by the statistical analysis between Group 1,2 and 3 (p=0,372). This means there is no significant difference in the sealing methods (p>0,05). The results of the SBS test are shown in Table 2.

Bond failures were determined by light optical microscope. Bond failure rates showed similar results in all groups (Table 3), where the most common failure pattern detected was ‘adhesive type’. Chi-Square test was used to analyze the relationship between the groups and failure types. In addition, no significant interaction was found between the groups and failure types.

Table 2. SBS values of all tested groups with the mean and the standard deviation (SD)

	Group 1 (n:6) Mean±SD	Group 2 (n:6) Mean±SD	Group 3 (n:6) Mean±SD	p (sig.)
Shear Bond Strengths (MPa)	14,22 ± 3,58	14,36 ± 2,53	17,06 + 4,94	0,372

p>0.05 indicates no significant difference

Table 3. Results showing the failures for each group, with the failure type

	Adhesive	Mixed	Cohesive in dentin	Cohesive in restoration material
Group 1	4	0	0	2
Group 2	3	0	0	3
Group 3	4	0	0	2

Discussion and Conclusion

This pilot study was planned to investigate the effect of IDS technique on the SBS of resin cement for luting hybrid ceramic material to dentin surface. Two different IDS procedures were used to treat the dentin surfaces. The first null hypothesis was that the application of IDS procedure will not improve the SBS of hybrid ceramic luted to dentin and the second null hypothesis was that the application of bonding agent combining with flowable composite for IDS procedure will not increase the SBS. According to the results of this pilot study, both of the null hypotheses were accepted .

Hybrid ceramic block materials consist of highly filled ceramic particles in their organic matrix [17]. Hybrid ceramics can be seperated into various subgroups [18] according to their inorganic composition. Resin interpenetrating matrix with glass ceramics (e.g., Vita Enamic), resin nanoceramics (e.g., CeraSmart) and resin interpenetrating matrix with zirconia – silica ceramics (e.g., Shofu Block). These hybrid ceramics are less fragile than glass ceramics and resist cracking and chipping during milling. They can be polished easily and provide easier finishing of restorations [19]. Thats why we choosed this material in our study.

Since the early 90s, it has been well established by some authors [7,14,20,21] that the resin coating application on freshly cut dentin using a three-step etch and rinse system (IDS) reduces bacterial leakage and dental sensitivity, protects the pulp by sealing the dentinal tubules, avoids contamination by temporary cements [22] and gap formation, allows the DBA and the adhesive layer to be polymerized in two steps [14,23] and then prevent the collapse of the uncured dentin-resin during placement of the restoration with pressure [7,14,24,25].

However, in this pilot study, the control and experimental groups shear bond strengths had no significant difference. Nevertheless, Magne et al, [7,15,26] reported that IDS procedure has greater bond strength values than the other sealing procedures [10,27,28]. Universal adhesives include a hydrophobic and hydrophilic monomer mixture, in which residual water could cause phase separation. The reason for these divergence may be associated with the application of all in one adhesive system, which may cause degredation of hybrid layer in temporary phase. An additional potential cause may be the differing bond strength testing procedures. In this pilot study, SBS testing was choosed to test the bonding efficiency of resin cement to dentin.

The choice of an adequate adhesive system has a crucial role in IDS. Using filled DBA such as G-Premio Bond can be more useful for immediate dentin sealing than an unfilled DBA because of its capacity to provide a more stable resin coating [29]. Self-etching systems higher quantitative and qualitative function of penetration provide to reduce postoperative sensitivity more than total-etch or milder etching systems [30]. Another study has showed that universal adhesives show higher bonding efficiency than two-step self-etch adhesives [31]. Conversely, other studies reported no significant difference [32,33] or that the two-step self-etch bonding agents was higher than universal adhesives in bonding efficiency [34,35]. In Group 3, IDS layer application was done with combination of the bonding agent with flowable resin composite. Jayasooriya et al reported that the application a DBA combined with a flowable resin composite on a prepared dentin significantly increased mTBS of resin cement [27].

Mine et al reported that, creating a micro retentive surface by either blasting or etching with hydrophloric acid followed by silanization to promote chemical adhesion [36]. Cerasmart already have SiO2 filler particles in their structure, thereby silica coating is not necessary for them. Additionally, the luting process takes an important role on effecting the results. G-Multi Primer includes three main functional agents (phosphate and silane or thiophosphate monomers), which help adhesion to various substrates. Silane adding to primer substance improves the adhesion stability. Silane ensures adhesion to hybrid ceramics, glass ceramics and resin based composites. Goldberg et al reported that, under extreme conditions dual-polymerizing luting cement seems to show better results [37]. A dual-cure (G-CEM Link Force, GC) was applied as an adhesive cement to lute all of the samples in whole groups in this study.

Appropriate cleaning of the teeth surface is crucial to the luting of the final restoration regardless of the using any cement type. According to the literatures they were

reported that silicoated aluminium oxide and glycin [38], aluminium oxide abrasion [7,15,30], fluoride-free pumice paste [10,14,39], soft-air abrasion [40] and using low speed rotary cutting instruments [15] are some of the effective methods of treating the immediate dentin sealing surface. Dillenburg et al reported that, extra H3PO4 etching was useful for IDS surface conditioning and provide to clean contaminants [41].

The application of the tested DBA and the flowable composite to dentin as an IDS technique has no statistically significant effect ($p > 0.05$) on the SBS. Adhesive failure rate was higher in whole groups and these findings described in the literature [10,26,41]. There was no correlation found between the groups and failure types in this study.

Within the limitation of this pilot study, this conclusion was drawn: Application of bonding agent with and without flowable composite for IDS procedures tested does not statistically ($p<0.05$) effect the SBS of hybrid ceramic CAD/CAM material luted to dentin with G-CEM Link Force.

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