SHEAR BOND STRENGTH AND SEM EVALUATION OF GLASS CERAMICS BONDED TO IMMEDIATELY SEALED DENTINE USING UNIVERSAL ADHESIVE

Mohamed Ellayeh*, Nessma Sultan** and Nesma Elgohary***

ABSTRACT

Immediate dentin sealing (IDS) has been proposed as an alternative to delayed dentin sealing (DDS), a procedure in which adhesive resin is applied immediately to freshly cut dentin following tooth preparation.

Materials and methods: The occlusal surfaces of 50 freshly extracted human teeth were prepared flat to expose the midcoronal dentin surface. Then, all prepared teeth were allocated randomly according to dentin sealing protocol into two main groups; Group (DDS): Delayed Dentin Sealing and Group (IDS) Immediate Dentin Sealing. For (DDS) group, the prepared occlusal surfaces were covered directly by a layer of provisional restoration. After 1 week, provisional restorations were removed and dentin was cleaned. For (IDS) group, universal dentine bonding adhesive was applied immediately to freshly cut dentine, then light cured, and temporarization was performed as in DDS group. Fifty glass ceramic discs were fabricated from Lithium disilicate block. After ceramic surface treatment, discs were bonded to dentine of both groups. Then, all specimens were thermo-cycled before being subjected to shear bond strength test. Scanning electron microscope (SEM) was used to examine dentin-cement-ceramic junction and mode of failure.

Results: Specimens with IDS protocol revealed higher shear bond strength than DDS protocol. SEM revealed that Adhesive failure was recorded as the most common pattern in IDS group, while in DDS group showed predominantly mixed failure at the dentine side.

Conclusion: The findings of current study suggest that, immediate dentine sealing with universal adhesive proved to be a reliable strategy for bonding glass ceramic restorations on prepared teeth with exposed dentin.

KEYWORDS: IDS, Glass Ceramic, Universal bonding adhesive

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INTRODUCTION

The field of dental materials technology has made significant advancements since the inception of partial adhesive restorations with introduction of CAD-CAM technology, indirect composite, resin strengthened dental ceramics and durable adhesives.1

The wide range of materials available for indirect restorations makes it possible to choose the best material according to the patient’s needs. Dental ceramics and indirect resin composites are the most widely used materials, as they proved relatively compatible survival rates. Moreover, the development of digital dentistry has tremendously revolutionized the way restorative procedures are performed today.2

The predictability of indirect restorations depends mainly on two main factors; selection of the right bonding strategy and understanding how to condition the restorations’ fitting surface to achieve the proper surface treatment for the variety of available restorative materials.3

Currently, advanced manufacturing techniques and materials are available to both practitioners and dental technicians for achieving minimally invasive restorations with minimal destruction of tooth structure.4 Although, dentinal tubules exposure is unavoidable regardless of how much tooth structure was removed. Moreover, inadequate sealing of provisional restoration would leave potential pathways for bacterial leakage to exposed dentine. In addition to mechanical and chemical stimuli that occurred through drying, rinsing, impression making and interim restorations removal and temporary cements.5

To mitigate the forementioned concerns and avoid potential pulp damage, Pashley et al.6 recommended immediate application of bonding agent to freshly cut dentine surface before impression making. This technique, which is also termed, dual bonding technique, prehybridization or resin coating technique, emerged with title of immediate dentin sealing (IDS) as an alternative to delayed dentin sealing (DDS) whereas bonding of dentin is performed just before the final restoration cementation. In IDS technique, the claimed advantages consist of reducing gap formation, bacterial microleakage, pulpal irritation and dentin hypersensitivity.7

A three-steps etch and rinse dentine bonding agent is advisable for IDS protocol.8 However, this system is very technique sensitive and cold be very confusing and time consuming with multiple application steps, which requires an exacting technique of controlled acid etching followed by two or more components (primer and bond) on both enamel and dentin.9

Universal adhesives has been introduced in adhesive dentistry as versatile multifunctional systems with reduced application steps, which is capable of achieving durable bond with all dental substrates and different restorative materials after proper surface treatments.10

Universal adhesive has considerably simplified and accelerated the adhesive protocol, and these systems actually represent a very important change in adhesive dentistry which has recently been used to improve IDS simplicity.11

A wide variety of CAD/CAM materials like ceramic, hybrid ceramic and indirect composite resin are available for fabrication of high-quality indirect restorations.12 Lithium disilicate (LS2) is glass ceramics category that is considered one of the world’s top selling CAD-CAM machinable ceramics owing to the outstanding properties and versatility of use either by heat-pressing technique, or CAD-CAM technologies.13

Since a firm bond between the tooth and restoration is crucial for long term success of final restoration, the hypotheses of this study was that, immediate dentine sealing using universal adhesive after teeth preparation will not significantly influence the shear bond strength of glass ceramic bonded to dentin.
MATERIALS AND METHODS

Fifty (n=50) caries-free permanent teeth were selected from outpatient clinic, Faculty of Dentistry, Mansoura University. Teeth were extracted as result of periodontal disease, mobility of teeth due to systemic disease as diabetic or extraction for orthodontic reason. This study was taken the approval of Local Research Ethics Committee, Faculty of Dentistry, Mansoura University, and followed its guidelines with approval no. M0103023FP.

Sample preparation

The extracted teeth were divided into two main groups (control and experimental group/25 each). A low-speed saw machine (Isomet 1000, Buehler, Lake Bluff, IL, USA) was used for cutting of occlusal half of crowns to expose the midcoronal dentin surfaces. Then All teeth were embedded in the centre of auto polymerizing acrylic resin blocks (Acrostone, Egypt).

Group (DDS) Delayed dentin sealing:

Exposed dentine surface was covered by a layer of provisional restorative material then immersed in saline solution at room temperature for 1 week.

Group (IDS) Immediate dentin sealing:

Exposed dentine surfaces were air dried for 5 seconds, then immediately sealed using universal adhesive system (Beautibond, shofu dental corporation, Japan) with the aid of a microbrush. universal bonding agent was applied onto the entire adhesive surface and remained undisturbed for a period of 10 seconds, air dried gently for 3 sec. Then, dried with stronger air to dry dentine surface. Light curing for 20 seconds was performed using LED curing light (BlueLex; Monitex, Taiwan) with power density of 1000 mW/cm2. A layer of interim restoration was applied on all teeth surfaces after protection of IDS layer with petroleum gel to prevent potential adhesion.

Ceramic disc fabrication

A total of 50 lithium disilicate discs were fabricated from (IPS™ e.Max CAD; Ivoclar Vivadent) with a dimension of (diameter: 4 mm, height: 2 mm). All ceramic blocks were subsequently crystallised in a Programat ceramic furnace (P500; Ivoclar Vivadent) according to the manufacturer’s instructions. Low speed diamond polisher (OptraGloss; Ivoclar Vivadent) was used for polishing of all blocks. Then, all specimens were cleaned in ultrasonic bath for 10 minutes in distilled water and dried for 60 seconds with oil-free air.

Ceramic disc conditioning

The intaglio surfaces of ceramic discs were treated using single-component self-etch primer (Monobond Etch and Prime, Ivoclar Vivadent). A microbrush was used to apply MEP to specimens’ surface for a period of 20 seconds and left to react for another 40 seconds. Then, specimens’ surface was washed using air water spray for 30 seconds before being air dried for 30 seconds.

Ceramic discs cementation

DDS group: After 1 week, temporary restorations were removed and dentinal surface was cleaned using pumice slurry. Then, ceramic discs were cemented in its place using adhesive resin cement (Breeze, Pentron, USA) under a 5 Kgm load using custom-made cementation device. Excess resin cement was removed using microbrushe. LED light-curing unit (BlueLex; Monitex, Taiwan) was used for light curing the assembly for 20 sec. Glycerin gel was applied to the exposed margins to guarantee complete polymerization and the assembly was light cured additionally for 10 seconds, and the load was continued constantly for 10 min.

IDS Group: After 1 week, the temporary restoration was removed. The sealed surfaces were refreshed by air-borne particle abrasion using 29μm aluminum-oxide powder (AquaCare Single,
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Velopex international, UK) for 10 mm distance with 2 bar pressure. Then, ceramic discs were cemented the same manner used in DDS group.

Thermocycling of the specimens

Teeth with bonded specimens were stored for 1 month in distilled water at 37°C and subjected to 5000 thermocycles between 5°C - 55°C using thermocycling device (thermocycler, ROBOTA, Alexandria, Egypt). Each thermal cycle composed of a 1-minute cold bath followed by a 1-minute hot bath with a 30 second dwell time.

Shear bond strength test

The SBST was conducted by applying compressive load at the ceramic/dentine interface using universal testing machine (Model 3345; Instron, USA). Teeth/ceramic assembly were fixed to the lower compartment while 0.5 mm thickness mono-bevelled chisel with a width of 6 mm was fixed to upper compartment moving at a cross-head speed for 0.5 mm/min until bonding failure occurred (Fig.1). Debonding load of failure was presented in Newton (N) and then transformed into Megapascals (MPa).

Scanning electron microscopy (SEM)

One specimen was selected from each subgroup after debonding for SEM examination using scanning electron microscopy (JEOL.JSM.6510LV) to examine the changes in microstructures of both the dentinal surface side and ceramic side and to investigate the mode of failure which was presented as (1) adhesive, (2) cohesive or (3) mixed failure.

Statistical Analysis

Data analysis was performed by SPSS software, version 26 (SPSS Inc., PASW statistics for windows version 26. Chicago: SPSS Inc.). Quantitative data were described using mean± Standard deviation for normally distributed data after testing normality using Shapiro Wilk test. Significance of the obtained results was judged at the (0.05) level. Student t test was used to compare 2 independent groups for normally distributed data.

RESULTS

Shear Bond Strength

The mean shear Bond Strength (SBS) and standard deviation for all tested group are presented in. (Table 1)

<table>
<thead>
<tr>
<th>TABLE (1) Shear bond strength among studied groups</th>
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<tr>
<td>DDS Sample</td>
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<td>Shear bond strength</td>
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<td>PST</td>
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*statistically significant

Fig. (1) Showing diagram for study specimen and SBS test using a universal testing machine
Paired t-test showed that there was a high statistical significant differences between SBS of (IDS) glass ceramic samples bonded to immediately sealed dentine (16.73±1.56) and (DDS) samples bonded to delayed sealed dentine (10.71±1.51) (P < 0.001)

Failur mode

Debonded specimens were examined by SEM to identify the type of the failure which is presented in (Table 2). Adhesive failure was recorded as the most common pattern of failure in (IDS) debonded specimens, while mixed failure was the most common type of failure in (DDS) group.

<table>
<thead>
<tr>
<th></th>
<th>Adhesive failure</th>
<th>Cohesive failure</th>
<th>Mixed failure</th>
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<tbody>
<tr>
<td>DDS</td>
<td>9</td>
<td>4</td>
<td>12</td>
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<tr>
<td>IDS</td>
<td>15</td>
<td>2</td>
<td>8</td>
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Scanning electron microscopy (SEM)

SEM of the dentinal surface in (DDS) group showed predominantly mixed failure with areas of both hybrid layer and/or resin cement covering areas of cohesively failed dentin showing various blocked hybridized resin plugs with few open ends of dentinal tubules (fig.2a,2b).

Also, some specimens showed adhesive failure between exposed dentin with cut blocked hybridized resin plugs and ragged collagen fibrils in between which suggest complete detachment of hybrid layer (fig.2c).

While SEM of the dentinal surface in (IDS) group showed undisturbed interface of resin cement on sealed dentinal surface and some cracks propagating through the adhesive resin cement which explained that dentin was totally covered with adhesive resin cement and bonding agent, proposing that failure does not occur between immediately sealed dentinal surface and resin cement (fig.3a).

Other SEM micrographs showed discrete, distinct areas of exposed IDS layers and resin cement. (fig.3b,3c)

SEM of deboned ceramic side in (DDS) group showed detached areas of resin cement and hybrid layer on the top of etched ceramic surface (fig.4a). Some other specimens suggested total adhesive bond failure at the dentine/bonding agent interface as ceramic was completely sealed with hybrid layers and endings of resin tags that detached from their former dentinal tubules (fig.4b).
SEM of deboned ceramic side in (IDS) group showed adhesive failure at ceramic/resin cement interface as the surface of specimen showed no remnants of resin cement. (fig.4c,4d)

SEM showed that universal bonding adhesive was able to establish a durable bond by means of good dentine hybridization (i.e. resin tags and hybrid layer) through micromechanical interlocking in both DDS and IDS groups. (fig.5a,5b)

Fig. (3) SEM of IDS treated dentinal surfaces at x1000 magnification showing: (a) Adhesive failure with crack propagate through the adhesive resin cement (RC). (b,c) discrete, distinct areas of exposed IDS layers and resin

Fig. (4) SEM of deboned ceramic side for DDS group with detached areas of resin cement (RC) and hybrid layer (HL) on the top of etched ceramic (C) surface (a and b) and IDS group showing conditioned ceramic surface with few remnants of resin cement (c and d).
DISCUSSION

Despite various advancements in clinical adhesive strategies, adhesion to dentin remains challenging. Over the last decade, the significance of micromechanical bonding to dentin has been recognised.\(^\text{15}\) According to current research, adhesion to dentin is mostly dependent on adhesive monomers infiltrating the collagen fibres exposed after acid etching. The dentin substrate exposed for bonding has a significant impact on the bonding strategy’s effectiveness.\(^\text{16}\) The IDS technique is purported to offer benefits such as improvement of bond quality, reducing gap formation, microleakage, dentinal hypersensitivity and pulpal irritation.\(^\text{7}\)

Since acid etching was first introduced into clinical practice, a variety of dentin bonding agents has been introduced to improve quality of adhesion to dentine substrate.\(^\text{9}\) In 2014, Magne et al.\(^\text{17}\) recommended using IDS protocol with either utilizing 3-step total etch technique or 2-step self-etch filled adhesive resin. However, the complexity and time-consuming nature of these systems, due to the numerous bottles and application steps, led to confusion. As a result, clinicians started seeking a more straightforward adhesive system.\(^\text{18}\) Universal adhesives are the latest generation of dentine boning agents, introduced in response to the growing demand for simpler and more user-friendly options.

In this study, universal or multimode adhesive was used to provide simplicity of IDS protocol as it offers a technique with less sensitivity by eliminating the etch and rinse step. Moreover, some of these generations have capability to bond with other different substances used for both indirect and direct techniques including ceramics.\(^\text{18}\)

Harden et al.\(^\text{8}\) claimed that regardless the adhesive strategy used, IDS protocol provide improved bond strength with freshly cut dentine to resin-based indirect restorations. However, limited information exists regarding the effectiveness of the IDS technique when employing universal adhesive systems.\(^\text{19}\) Hence, this current study investigated the impact of IDS protocol of freshly cut dentin using universal adhesive on shear bond strength of lithium disilicate glass ceramic luted to dentin surfaces in comparison with delayed dentine sealing protocol. The hypotheses of this research was rejected as glass ceramic samples bonded to IDS treated dentine showed higher significant difference compared to DDS samples (\(P > 0.001\)). In agreement with these findings, Choi et al. also reported that the IDS protocol achieved higher shear bond strength (SBS) values than that of DDS technique for indirect ceramics.\(^\text{20}\) Another in-vitro study reported that, utilizing IDS approach with universal adhesive found to be an efficient method to increase the ultimate bonding strength and decrease dentin permeability of CAD/CAM restorations.\(^\text{21}\)
The literature defined at least 2 compelling reasons that validate the effectiveness of IDS in enhancing dentin bond strength. **First,** freshly cut dentinal surfaces serve as the optimal substrate for dentin bonding as reported by Magne et al.\(^7\) In this study, the SBS values of DDS group could be attributed to contamination of dentinal surface with provisional cements before bonding step. **Second,** it could be attributed to the repolymerization of the universal adhesives which in turn increase the indirect restorations bonding strength to immediately sealed dentine. Frankenberger et al.\(^22\) reported that higher values of bond strength could be achieved by early polymerization of DBA to avoid the anticipated collapse of unpolymerized dentin–resin hybrid layer resulted from pressure of seating of final restorations.

Tooth preparation results in a residual organic and inorganic component which form a smear layer. This layer fills the dentinal tubules to form smear plugs which is considered a barrier that should be removed before resin application to the dentin substrate.\(^23\) Based on that consideration, in DDS group, the dentinal tubules were obliterated with smear plugs resulting in weak bond strength at the dentinal side. SEM of the dentinal surface in (DDS) group showed exposed dentin with ragged collagen fibrils in between cut blocked hybridized resin plugs which suggest complete detachment of hybrid layer.

IDS produced better bond strength than DDS which might be attributed to the application of the universal adhesive on freshly cut dentin with subsequent dissolution of the smear layer formed after preparation, exposing more dentinal tubules that were penetrated with the bond, resulting in better bond strength. SEM analysis of debonded surface mostly revealed undamaged interface of overlaying resin cement in IDS surface. It was confirmed that, dentinal surface was entirely covered with universal adhesive and resin cement, proposing that failure did not occur between resin cement and IDS surface.

In this in vitro study, IDS group specimens had the highest bond strength and the failure mode was adhesive pattern, whereas DDS group exhibited mixed failure pattern as reported by SEM investigation. This was in accordance with Nakazawa et al.\(^24\) who found that IDS protocol using single application of universal adhesive functioned substantially better than DDS protocol.

The surface of dentin, following the IDS protocol, is subjected to oral conditions, where fatigue can affect the mechanical and physical properties of dentin-resin bond strength.\(^25\) Hence, all tested samples in this study were thermo-cycled before being subjected to shear bond strength test. Taking into account the laboratory design of this study and recognizing that oral clinical conditions; such as variations in pH level, masticatory stress, moisture and saliva, may accelerate the breakdown of bonding interface. It is recommended that additional studies should be conducted with greater simulation of oral conditions.

**CONCLUSION**

The results of this study suggest that, Immediate dentine sealing with universal adhesive proved to be a reliable strategy for bonding glass ceramic restorations on prepared teeth with exposed dentin.

**REFERENCES**


