CLINICAL STUDY ON THE INFLUENCE OF USING SEPARATING MEDIUM AND ITS TYPE ON INDIRECT RESIN COMPOSITE MICROTENSILE BOND STRENGTH TO DENTIN

Asmaa A. Yassen* and Mohamed F. Haridy**

ABSTRACT

Purpose: To evaluate the effect of using separating medium and its type on the microtensile bond strength of indirect resin composite restoration to dentin clinically.

Materials and methods: Standard 24 MOD cavities were prepared in the upper first premolars for the selected patients by one operator and restored using indirect resin composite (Grandio So Inlay System, VOCO, Germany). Inlays were constructed outside the patient mouth either on silicone die (according to manufacture instructions) or on stone die using two different separating media (glycerin or Vaseline). The fitting surface of the inlay was sandblasted, silanated and bonded before cementation. Teeth were treated with a dual cured adhesive (Futurabond DC, VOCO, Germany) and resin cement (Bifix QM, VOCO, Germany) was used for cementation. After the assigned time (either 24 hours or 3 months) the restored teeth were extracted atraumatically. They were prepared for microtensile bond strength testing which was performed using the universal testing machine. Each restoration yielded 3-4 beams with a total of 90 specimens (15 specimens for each group). Failure mode analysis was done by digital microscope. Collected data were statistically analyzed using One Way-ANOVA and Independent t-test.

Results: After 24h silicon die group showed the highest statistical mean microtensile bond strength (29.25±1.57 MPa) followed by stone die (Glycerin) (28.2±1.01 MPa) and then stone die (Vaseline) (24.88±1.17 MPa) at p≤0.001 with an insignificant difference between silicon die and stone die (Glycerin) after 24 h .After 3 months all the values were statistically lower than 24h at p≤0.001.Stone die specimens exhibited predominantly mixed failure and the other two groups commonly had cement/restoration adhesive failure .

Conclusion: Fabrication of indirect restoration on silicone die without using separating medium has a great influence on achieving high microtensile bond strength value to the dentin even after 3 months. Vaseline has a deteriorating effect on the microtensile bond strength when it is used as a separating medium for the stone die.

KEYWORDS: Separating medium- Indirect resin- Silicone die- Microtensile- Clinical study

* Associate Professor of Operative Dentistry, Faculty of Dentistry, Cairo University, Egypt
** Lecturer of Operative Dentistry, Faculty of Dentistry, Cairo University, Egypt
INTRODUCTION

Recently a lot of advancements have been introduced into the world of resin composite materials and techniques, bringing it to the upfront lines options for restoring defected hard tooth structure esthetically and functionally.\(^{(1)}\) Such restoration can be achieved by application of resin composite directly to the prepared cavity or indirectly to a specially constructed mold.\(^{(2)}\)

It is worth mentioning that the direct application of resin composite especially in the posterior region has many challenges including restoring the proximal contact, intraoral polymerization shrinkage, compromised marginal integrity, surface loss and great possibility for post-operative hypersensitivity. Hence the indirect technique has gained a great popularity among dentists. Nevertheless, it still has its shortcomings as being time consuming and requiring multistep fabrication with sensitivity in dealing with the impression and die materials.\(^{(3)}\)

Buildup of indirect restoration can be done either on a stone or silicone die (flexible model technique). Each technique has its merits and demerits. For the stone die we have to use a separating medium to facilitate removal of the restoration from the die. Many materials can be used for this purpose including petroleum jelly, which is being marketed under the name of Vaseline or Glycerin. Contamination of the fitting surface with such materials may affect the microtensile bond strength between the restoration and tooth structure.\(^{(4)}\)

Flexible model technique includes taking silicone impression and pouring it with the same silicone material to obtain the flexible die. Chemical similarity between the impression and die material necessitates using a separating medium in between which may contaminate the inlay fitting surface.\(^{(5)}\) A new system has been introduced and dictated taking alginate impression and pouring it with silicone material without applying separating medium.\(^{(6)}\) Such technique may decrease the risk for compromised bond due to contamination.

The aim of this study was to evaluate the influence of using separating medium and its type on the indirect resin composite restoration microtensile bond strength to dentin. Also studying the effect of time on the sustainability of the obtained bond was done. The null hypothesis is that there is no difference between the used techniques on the bond strength of the restoration.

MATERIALS AND METHODS

A total of 12 patients were selected from the outpatient clinic of the orthodontic department in the faculty of oral and dental medicine (Cairo University) in accordance with the local ethical committee rules (approval date 17/3/2015). The selected patients were planned to extract their first maxillary premolars for orthodontic purposes. Accordingly, 24 human maxillary premolars were used in this study. The age range of the selected patients was 20-28 years. All the selected patients were free from any systemic condition, active carious lesions or periodontal disease and they had good oral hygiene. Presence of antagonists and contralateral for the treated tooth were other inclusion criteria. Before starting this study, all patients signed a computerized informed consent and agreed to participate in the study. One week before the preparation visit, all patients were subjected to full mouth scaling and were given special oral hygiene instructions and mouth washes in order to minimize, as much as possible, gingival bleeding during cavity preparation and impression steps. All the procedures were carried out by the same qualified operator.

The used 24 premolars were randomly divided into two main groups according to the type of die materials used to construct the indirect resin composite restoration either silicon die (8 teeth in 4 patients) or stone die (16 teeth in 8 patients).
The stone die group was further divided into two subgroups according to the separating medium used either glycerin (8 teeth) or Vaseline (8 teeth) using split mouth design. All the teeth of the previously assigned groups and subgroups were divided according to time elapsed after cementation of the restoration either 24 hours (act as a control) or 3 months.

**Cavity preparation**

Standard MOD cavities were prepared using straight fissure carbide bur no. 57 size 010 (Brassler, Savannah, Georgia, USA) rotating at high speed with air/water cooled hand-piece (PANA MAX, NSK, Japan). A new bur was used for every five preparations. All cavities were prepared according to the common principles for adhesive indirect resin composite restorations. The cavity dimensions were 4 ± 0.2 mm for the pulpal depth from the tip of the palatal cusp and 4 ± 0.2mm for the buccopalatal width. The proximal walls were parallel with slight occlusal divergence. The preparations were finished with 25-μm grit diamond stones with a slight taper of 6° (TR-13EF; MANI, INC). A single periodontal probe (Hu-Friedy Co., Rockwell St. Chicago) was used as a guide for all the cavities and no bevel was performed at the margins of the cavities.

**Impression taking and die fabrication**

In the stone die groups, 2-step impression technique, sectional impressions were taken with a polyvinyl siloxane impression material (Express Putty Regular Set Heavy Body and Express Light Body, 3M ESPE, St Paul, MN, USA) followed by rinsing with tap water and dried with air syringe and then impressions were disinfected by glutaraldehyde solutions for 10 minutes. Impressions were poured twice with type IV stone to obtain two casts, one with removable die and the other without a removable die. The margins were marked with a red pencil on the removable die. For the silicon die group, the impression was taken with sectional tray using alginate material (Hydrogum 5, Zhermack SpA, Italy) to be poured with model silicone material (Modellisilikon, VOCO GmbH, Germany, Cuxhaven) according to manufacturer’s instructions. Eugenol-free temporary restoration (Cavit G, 3M ESPE, St Paul, MN, USA) was inserted into the cavity as a provisional restoration.

**Composite inlay build up**

For the fabrication of stone die group, the die was soaked in water for one minute, and a very thin coat of separating medium either Glycerin (Phelopharm company, Cairo, Egypt) or Vaseline (Vaseline, Unilever) was applied with a brush to the preparation for easy removal of the restoration from the die without fracture or chipping. Using a gold plated plastic instrument (Hu-Friedy Co., Rockwell St.Chicago), the first increment of GrandioSo inlay system composite (VOCO GmbH, Germany, Cuxhaven) was added to the floor of the preparation till reaching half the cavity depth (2 mm), short of the margins, and then cured for 20s using a LED light curing unit (Elipar S10, 3M ESPE, St Paul, MN, USA) operating in standard mode at light intensity 1200 mW/cm² according to manufacturer instructions. The second increment including the contact areas both proximally and occlusally was applied and light cured for 20s. The inlay was removed and again light cured from inside for 20s. Finally the restoration was polished using Sof-Lex spiral wheel kit (3MESPE, St Paul, MN, USA).
Inlays try-in

Using rubber dam, the teeth to be restored were isolated, the provisional restorations were removed, and the prepared cavities were cleaned from any remnants using air water spray. Restorations were checked for occlusion, proximal contacts and marginal fit.

Cementation procedures

The fitting surface of the inlay was sandblasted, silanated and bonded before cementation. A dual cured adhesive (Futurabond DC, VOCO, Germany) was used according to manufacturer’s recommendations. Liquid 1 & liquid 2 were mixed with 1:1 ratio till homogenous mix was obtained. The mix was applied in a medium thickness and rubbed into the tooth surface for 20 seconds then dried for at least 5 seconds with an air syringe. Finally the adhesive was light cured for 10 seconds. Resin cement (Bifix QM, VOCO, Germany), was dispensed into the preparation; the inlays were firmly placed in the prepared cavities and checked for full seating. The extruded excess cement was light cured for only 5 seconds to facilitate its removal with an explorer. Then light curing was applied from all directions for 40 seconds to achieve the final set. The inlay margins were finished with finishing stone, polished with rubber points in a low-speed handpiece (NSK, Japan) and checked using an explorer. The proximal contacts were checked with dental floss, the occlusion was checked and adjusted using articulating paper and diamond stones. Finally, the inlays were polished with Sof-Lex spiral wheel kit (3MESPE, St Paul, MN, USA). The patients were instructed to clean their teeth regularly at least for two times per day using toothpaste and medium type tooth brush. The restorative materials used are presented in table (1).

Atraumatic extraction of the prepared teeth

According to the assigned time for extraction either (24 h or 3 months), the restored teeth were

<table>
<thead>
<tr>
<th>Materials</th>
<th>Category</th>
<th>Composition</th>
<th>Manufacturer and Batch number</th>
</tr>
</thead>
<tbody>
<tr>
<td>GrandioSo</td>
<td>Nanohybrid resin composite</td>
<td><strong>Fillers:</strong> Glass ceramic filler with an average particle size 1µm. Functionalised silicon dioxide nano-particles fillers with an average size 20-40 nm. 89% w/w filler content. Pigments (iron oxide, titanium dioxide). <strong>Resin:</strong> Bis GMA, Bis EMA and TEGDMA</td>
<td>(VOCO GmbH, Cuxhaven Germany) REF 2642 LOT 1351154</td>
</tr>
<tr>
<td>Future Bond DC</td>
<td>Dual cure self etch adhesive</td>
<td><strong>Liquid 1:</strong> Acid modified methacrylate (methacrylate ester), HEMA, Camphorquinone. <strong>Liquid 2:</strong> Water, Ethanol, Silicium dioxide</td>
<td>(VOCO GmbH, Cuxhaven, Germany) REF 2642 LOT 1351154</td>
</tr>
<tr>
<td>Bifix QM</td>
<td>Dual cure resin cement</td>
<td>Monomer matrix: Bis GMA Inorganic fillers</td>
<td>(VOCO GmbH, Cuxhaven, Germany) REF 2642 LOT 1351154</td>
</tr>
</tbody>
</table>

**Bis-GMA:** bisphenol A glycol ether dimethacrylate, **Bis-EMA:** ethoxylated bisphenol A glycol ether dimethacrylate, **TEGDMA:** triethylene glycol dimethacrylate, **HEMA:** hydroxethylmethacrylate
extracted using posterior periotome instrument (Hu-Friedy Co., Rockwell St.Chicago) to avoid fracture and deterioration of the restoration. These Periotomes have thin sharp blades to facilitate the removal of teeth with minimal damage by severing the periodontal ligament. Several strokes were applied from all directions to loosen the tooth. Then the tooth was extracted using the upper premolar forceps (Hu-Friedy Co., Rockwell St.Chicago). The extracted teeth were cleaned under running tap water and put in distilled water for 24h before testing.

**Microtensile bond strength test**

The crowns were sectioned along the buccolingual and mesiodistal planes using a diamond disk (MTI Corporation, Richmond, CA, USA) in a low speed micro-slicing machine (Isomet, Buehler, Lake Bluff, IL, USA) under water-cooling, to produce beam-shaped specimens (bonding areas approximately 1 mm²). Centralized 3 to 4 specimens were taken from each tooth and so a total of 90 specimens were obtained from 24 teeth, 15 specimens for each subgroup. The bond strength test was performed immediately after cutting. The beam specimens were attached with cyanoacrylate gel (Zapit; Dental Ventures of America, Corona, CA, USA) to the testing customized microtensile jig. This jig is designed to transmit tensile forces to the specimen purely without any torque and designed to fit the µTBS Instron Universal testing machine (Bisco Inc. Schaumburg, IL, USA). The tensile load was applied at a cross-head speed of 0.5/minute until specimen failure occurred. At this point the failure load in Newton was recorded. The bond strength was calculated as the ratio between the failure load and the beam area that was checked with a digital caliper before testing.

**Failure mode analysis**

The fractured specimens were examined using a digital microscopic (Scope Capture Digital Microscope, Guangdong, China) at 50X magnification and photographed using image analysis software (Scope Capture 1.1.1.1. Ltd Co.) to determine the mode of failure either (1) adhesive failure along the cement–dentin interface, (2) adhesive failure along the cement–composite interface, (3) cohesive failure within resin cement, (4) cohesive failure within the resin restoration, (5) mixed failure of 1 and 3 and (6) mixed failure of 2 and 4.

**Statistical analysis**

Data were presented as mean and standard deviation (SD) values. They were explored for normality using Kolmogorov-Smirnov and Shapiro-Wilk tests. Micro tensile bond strength showed normal distribution, One Way-ANOVA was used to study the effect of using separating medium and its type on mean micro tensile bond strength within each evaluation period. Independent t-test was used to study the effect of different follow-up periods. The significance level was set at P ≤ 0.05. Statistical analysis was performed with IBM® SPSS® (SPSS Inc., IBM Corporation, NY, USA) Statistics Version 22 for Windows.

**RESULTS**

Mean and standard deviation (SD) for the microtensile bond strength (MPa) for different separating medium used and follow-up periods are presented in table (2) and figure (1).

Silicon die without separating medium showed the highest statistical mean microtensile bond strength (29.25±1.57 MPa) followed by stone die (Glycerin) (28.2±1.01 MPa) and then stone die (Vaseline) (24.88±1.17 MPa) at p≤0.001 with an insignificant difference between silicon die and stone die (Glycerin) after 24 h.

After 3 months silicon die without separating medium showed the highest statistical mean microtensile bond strength (23.75±1.29 MPa) followed by stone die (Glycerin) (22.89±1.33 MPa) and then stone die (Vaseline) (19.57±1.29 MPa) at
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Figure two showed failure mode analysis after microtensile bond strength in %. Silicone die specimens commonly exhibited mixed type including adhesive failure between the resin cement and restoration and cohesive failure in the restoration 50% after 24h and 30% after 3 months. Stone die with glycerin separating medium exhibited most commonly the adhesive failure either between the cement and restoration (40% after 24h and 50% after 3months) or between dentin and cement (30% after 24h and 3months). The same was occurred with the stone die with Vaseline separating medium. It showed adhesive failure either between the cement and restoration (50% after 24h and 60% after 3months) or between dentin and cement (30% after 24h and 3months).

TABLE (2) Mean and standard deviation (SD) values for microtensile bond strength for different die and separating medium used within each follow-up period

<table>
<thead>
<tr>
<th>Group</th>
<th>Silicon die</th>
<th>Stone die (Glycerin)</th>
<th>Stone die (Vaseline)</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean</td>
<td>SD</td>
<td>Mean</td>
<td>SD</td>
</tr>
<tr>
<td>Microtensile bond strength (MPa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>24 h</td>
<td>29.25a</td>
<td>1.57</td>
<td>28.20a</td>
<td>1.01</td>
</tr>
<tr>
<td>3 Ms</td>
<td>23.75a</td>
<td>1.29</td>
<td>22.89a</td>
<td>1.33</td>
</tr>
<tr>
<td>p-value</td>
<td>≤0.001*</td>
<td>≤0.001*</td>
<td>≤0.001*</td>
<td></td>
</tr>
</tbody>
</table>

Means with the same letter within each row are not significantly different at p=0.05. *= Significant

Fig. (1) Bar chart showing the mean microtensile bond strength for different die and separating medium used within each follow-up period
DISCUSSION

Separating media are the materials used for filling porous surfaces for easy separation from other materials which are later poured against them.\(^7\) It is used in the fabrication of resin composite inlay to facilitate its removal from the stone die. Polymerization rate as well as the optical and physical properties of the resultant restoration can be greatly affected by the used separating medium type.\(^8\)

Glycerin has been used for heat cure and autopolymerizing resin.\(^9\) It is a simple poly oil compound, colorless, odorless, viscous liquid that is widely used in medical, pharmaceutical formulations and personal care preparations.\(^10\) It is an alcohol that is strongly hydrophilic (water-attracting). The viscosity of glycerin which is 1.4/2 Pas permits it to close any voids or porosity found. Also the high coefficient of penetration of this material permits its penetration.\(^{9,12}\)

Oils such as Vaseline were also used as a separating medium.\(^{13-15}\) It is the commercial name of Petroleum jelly which is a non-polar hydrocarbon hydrophobic (water-repelling) and insoluble in water. It is soluble in dichloromethane, chloroform, benzene, diethyl ether, carbon disulfide and oil of turpentine.\(^{16,17}\) It is very sticky and hard to remove from non-biological surfaces with the usual and customary cleaning agents typically found at home. It can be dissolved with paint thinner or other petroleum solvents such as acetone, which dissolves most plastics.\(^{18}\) However, the reason behind using it as a separating medium is its surface tension which occurs due to the strong adhesion force found between its molecules. The higher the surface tension of separating medium, the higher the sealing and separation between the restorative material and stone die.

Contamination of the inlay fitting surface with such separating medium may greatly affect the bond strength between the restoration and the tooth structure. In a trial to minimize such effect a recent technique was introduced, it is termed silicone die technique. In this technique an alginate impression is taken and poured with silicone impression material without using any separating material. The buildup of the inlay is performed directly on the silicone die. Thus possibility of separating medium contamination is omitted.\(^{19}\)

Therefore, this study was conducted to investigate clinically the effect of using separating medium, either Glycerin or Vaseline, in comparison with the silicone die technique, which did not involve using separating medium, on the microtensile bond strength of indirect resin restoration to dentin.
It was found that using separating medium during fabrication of resin inlay had a detrimental effect on its microtensile bond strength to dentin. This effect was much more evident in the Vaseline group. This might be due to its substantivity and great difficulty of its removal from the fitting surface. Worsening the scenario is the hydrophobic nature of Vaseline, which requires special solvents for its removal. Glycerin exhibited lesser effect due to its easy removal referring to its hyrophilicity. This findings can also be documented by the obtained failure mode. Majority of the stone die specimens either with Glycerin or Vaseline separating medium showed adhesive failure which indicated weak bonding.

Manufacturing of indirect composite restorations using the GrandioSO Inlay System (VOCO GmbH, Germany, Cuxhaven) seems to be a good alternative to direct composite restorations to reduce the polymerization shrinkage effect. Also for the indirect restorations which are built up on stone die with a great possibility for fitting surface contamination with the used separating medium. Pott et al. in 2014 reported better internal and marginal fit, without any fitting surface contamination, for this system. This finding was approved clinically in this study by higher bond strength values obtained in the silicone die group. Mixed type of failure was the common mode observed in this group indicating strong bond.

All groups showed a statistically significant decrease in the bond strength values after 3 months. This result is attributed to the oral environmental factors which include oral fluids, pH and thermal fluctuation and occlusal stresses. The durability of the bonded joint is threatened by hydrolysis and degradation inside the patient mouth.

Therefore, null hypothesis was rejected as there is significant difference between the bond strength values in the Vaseline, Glycerin and silicone die groups. Further studies to verify the findings of this study are required.

**CONCLUSION**

Within the limitations of this clinical study, the following conclusions can be drawn:

1- Using Vaseline as a separating medium had a deteriorating effect on the inlay microtensile bond strength to dentin.

2- Glycerin showed minimal damaging effect on bonding to dentin.

3- Silicone die technique is a good alternative to other techniques which dictated using separating medium during inlay fabrication procedures.

4- The inlay microtensile bond strength to dentin deteriorated by time.

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