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Original Research Article

Evaluation of Micro-Tensile Bond Strength of Indirect Resin Composite Inlay to Dentin

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ABSTRACT

Objective: To evaluate micro-tensile bond strength (µTBS) of indirect resin composite inlay to dentin after specimen's storage in distilled water and lactic acid.
Materials and Methods: Standardized MOD Class II cavities were prepared in 96 intact human molars and restored with SR Nexco resin composite inlay restorations. The specimens for each test (n=48) were assigned into three sets (n=16) according to the luting resin cement used (etch-and-rinse (Variolink N), self-etch (Panavia F2.0) and self-adhesive (RelyX Unicem)). Each set was subdivided into two equal subsets (n=8) relative to the storage media either distilled water or lactic acid. Half of the specimens of each subset were stored in each storage medium for 24h while the other half was stored for 168h (7 days).
Results: The outcome of µTBS to dentin evaluation showed that, SE Panavia F2.0 stored in distilled water for 24h revealed the highest values. Etch & rinse Variolink N stored in lactic acid for 168h showed the lowest values.
Conclusion: µTBS showed good results with SE resin cement strategy.

Keywords: Micro-tensile bond strength (µTBS), indirect resin composite, resin cement

INTRODUCTION

Due to the increased demands for esthetic dentistry and conservation of tooth structure, resin based composites are used as direct posterior restorations. Which have grown in popularity in combination with adhesive systems as the treatment of choice where esthetic is a primary concern. [1,2] Sometimes clinicians were confused when dealing with restoration of posterior teeth, especially during rehabilitation of severely damaged or fractured ones. Where they have to select which material and technique is more adequate for restoration. Direct composite restoration may be inadequate in the long term due to insufficient wear resistance, imperfect proximal or occlusal morphology and deficient mechanical properties. [3]

Other resin based composite materials and curing systems have been introduced, one category is the indirect composite restorations such as inlays, which defined as single-tooth restoration that compensates a proximal-occlusal lesion with minimal or moderate extensions. These alternative esthetic restorations are developed to overcome the limitations of direct posterior composite and ceramic inlays. Laboratory-processed resin composite inlays are characterized by superior mechanical properties, high wear resistance, excellent esthetics, low polymerization shrinkage, better proximal contact and occlusal morphology in comparison to direct restorations. Also, they are lower in cost, lower wear for opposing teeth and easier in repair than ceramic inlays. But, they require two appointments and provisional restoration between visits. [4,5]

A brand of indirect resin composites, SR Nexco has been introduced in the dental market in 2012. SR Nexco paste is a light-curing laboratory composite indicated in the fabrication of the framework-free dental restorations (inlays and onlays). The combination of microfillers plus prepolymer enables a very high filling ratio and excellent physical properties. The use of the prepolymer allows the advantages of large filler particles to combine with those of microfillers. This technology allows for a superior strength of resin composite that if only inorganic microfiller were used. [6]

Cementation process is considered a critical step in ensuring the longevity of resin composite inlays. It may be difficult for choosing the appropriate resin cement to be used, because many dentin adhesives have been introduced in order to achieve a good bonding between resin cement and dental substrate. [7-10] Resin cements can be classified according to tooth substrate pretreatment into: (1) etch-and-rinse, (2) self-etch, (3) self-adhesive. [11-18]

Etch & rinse resin cements are time consuming and sensitive to handling, efforts have been made to simplify the luting process and to provide a reliable as well as durable bond to dental tissues by producing self-adhesive resin cements. Which have attracted the interest of both manufacturers and clinicians, because they do not require any pretreatment of dentin surface. [19,20] The ideal resin luting cement should be impenetrable to oral fluids or acids produced by dental plaque and resist dissolution over the life time of restorations. In case of oral environment and presence of moisture or acids, there is increase risk of cement dissolution and bond degradation at the marginal gap leading to weakening and failure of restoration. [21,22]

Few studies evaluated the effect of acids produced by human dental plaque, and showed that lactic and other acids had detrimental effects on softening and surface degradation of polymeric resin materials. [21,22] Studies about the action of lactic acid on self-adhesive resin cement may increase the knowledge towards their durability in the oral environment. Therefore, it is necessary to evaluate the adhesion durability of indirect resin composite inlays both *in-vitro* and *in-vivo*. [23]

Micro-tensile bond strength is considered one of the main factors affecting the success of clinical performance of resin cements and the longevity of inlay restorations. Which in turn, is affected by tooth substrate pretreatment, mode of polymerization (light, chemical or dual-cure), depth of cure, degree of conversion and mode of resin cement handling. [24] All resin cements undergo dimensional changes during and after setting. During setting, the closer distance between the reacted molecules produces volumetric shrinkage, which results in developing tensile stresses at resin cement/tooth substrate interface, that make the adhesion at risk. [25-39] Considering this clinical scenario, the polymerization protocol of resin cement is of great importance for restoration performance. [40] The null hypotheses of this study was there is no difference between micro-tensile bond strength investigations of the three different resin cement strategies: etch-and-rinse Variolink N, self-etch Panavia F2.0 and self-adhesive RelyX Unicem for luting indirect resin composite inlays in MOD cavities.

MATERIALS & METHODS

The present study was performed using a laboratory resin composite, SR Nexco (Ivoclar Vivadent AGSchaan, Liechtenstein), cemented with three different resin composite luting cements: an etch-and-rinse dual-cured Variolink N (Ivoclar Vivadent AGSchaan, Liechtenstein), self-etch dual-cured Panavia F2.0 (Kurary medical, Okayama, Japan) and self-adhesive dual-cured RelyX Unicem (3M ESPE, ST Paul, MN USA).

Table 1: Indirect resin composite restorative system used in the study

Material	Composition	Manufacturer
SR Nexco liner	Dimethacrylates (48wt.%), barium glass filler, silicone dioxide (51wt.%), additional contents are catalysts, stabilizers and pigments (<1wt.%).	Ivoclar Vivadent AG Schaan, Liechtenstein
SR Nexco paste Layering materials (incisal & dentin)	Dimethacrylates (17-19wt.%), copolymer and silicone dioxide (82-83wt.%), inorganic filler (64-65wt.%), inorganic filler (64-65wt.%) (<1wt.%).	Ivoclar Vivadent AG Schaan, Liechtenstein
SR Nexco stain	Dimethacrylates (47-48wt.%), copolymer and silicone dioxide (49-50wt.%), additional contents are catalysts, stabilizers and pigments (2-3wt.%).	Ivoclar Vivadent AG Schaan, Liechtenstein
SR Gel	Glycerine, silicone dioxide and aluminium oxide	Ivoclar Vivadent AG Schaan, Liechtenstein

Table 2: Resin composite cements used in the study

Resin composite cement	Composition	Mechanism of adhesion	Manufacturer
N-Etch Syntac Heliobond Variolink N	Etchant: 37% phosphoric acid. -Syntac primer: 4% maleic acid, TEGDMA, PEGDMA, water, acetone. -Syntac adhesive: PEGDMA, glutaraldehyde, water. -Heliobond: Bis-GMA, UDMA, TEGDMA. -Variolink N base: Bis-GMA, UDMA, TEGDMA, fillers, ytterbium trifluoride, stabilisers, pigments. -Variolink N catalyst: Bis-GMA, UDMA, TEGDMA, fillers, ytterbiumtrifluoride, stabilizers, pigments, penzoyl peroxide.	Dual-cured 3-step etch-and-rinse	Ivoclar Vivadent AGSchaan, Liechtenstein
Panavia F 2.0	ED primer: -Primer A (HEMA,10- MDP, chemical initiator, water, 5-NMSA) - Primer B (5-NMSA, chemical initiator, water panavia F2.0) -A Paste (quartz, glass,10- MDP, methacrylate, photoinitiator) -B Paste (silanated barium glass, NaF, methacrylate, chemical initiator)	Dual-cure one step self-etch	Kurary medical (Okayama, Japan
Rely X Unicem	-Powder (silica, glass fillers, calcium hydroxide, chemical curing initiators, light curing initiators) -Liquid (methacrylated phosphoric esters, dimethacrylates, chemical curing initiators)	Dual-cure self-adhesive Translucent aplicap	3M ESPE (ST Paul, MN USA)

Abbreviations: TEGDMA=triethylene glycol dimethacrylate; PEGDMA=polyethylene glycol dimethacrylate; UDMA=urethane dimethacrylate; Bis-GMA=bisphenol A di glycidylmethacrylate; HEMA=2-hydroxyethyl methacrylate; MDP=10-methacryloyloxydecyl dihydrogen phosphate; 5-NMSA=N-methacryloyl 5-aminosalicylic acid; NaF=sodium fluoride

Teeth Selection

Freshly extracted human molars from healthy individuals free from caries, or restorations were selected for µTBS tests. Teeth were cleaned from the adherent soft tissues with a hand scaler (Zeffiro, Lascod, Florence, Italy) then stored and disinfected in 2% sodium azide solution for three days. After that, they were cleaned using a rubber cup and fine pumice water slurry then examined by binocular Stereomicroscope (30X magnification, SZ TP, Olympus, Tokyo, Japan) to exclude the cracked ones. Forty-eight molars were selected and finally kept in distilled water at 4^{o}C, which was changed periodically every 5 days throughout the study to avoid their dehydration, and teeth were removed only before their use.

The roots of selected teeth were embedded in a cylindrical polyvinyl chloride (PVC) rings up to 2 mm below the cementoenamel junction, using autopolymerizing acrylic resin (Acrostone, Cairo, Egypt) to complete stabilization of the teeth. A cylindrical Teflon mold, with a corresponding metal ring and two opposing screws at its top was used to hold the tooth in a centralized position, parallel to the long axis of the mold, during the setting of acrylic resin.

Cavity Preparation

At first, an impression was taken for each tooth before any preparation was done. Using equal amounts of the base and catalyst of high-viscosity impression paste (SHERATWIST 60) that were mixed together and seated in a sectional tray. A standardized MOD cavity was prepared using special kit for inlay preparation (Komet, Brasseler GmbH & Co. KG, Lemgo, Germany) and high speed handpiece with water coolant. To ensure high cutting efficacy, a new diamond instrument was replaced every five preparations.

The cavity dimensions were strictly standardized during preparation by securing the handpiece in a specially designed appliance that was constructed at Production Engineering and Mechanical Design Department, Faculty of Engineering, Mansoura University, Egypt. This device allowed accurate movements of the handpiece, resulting in approximately a standard divergence of cavity walls with a standard depth and width. The dimensions of the cavity preparation were 4mm buccolingually, 3 mm deep at the isthmus; 4 mm deep at the mesial and distal surfaces and the boxes were 1.5 mm at the base towards the pulp. The cavities were prepared 1-1.5 mm above the cemento-enamel junction. All the internal line angles were smoothed and rounded so as to reduce the possibility of stress concentrations.

Inlays Fabrication

Final impression was taken for each cavity using mix of light-viscosity impression paste (SHERATWIST 60), which syringed into the prepared cavity and over the high-viscosity primary impression which seated in the sectional tray. Then, every impression was sent to dental laboratory to be cast into a die stone. The technician fabricated all the restorations following the manufacturer’s instructions.

Inlays Cementation

After try-in procedure, the 48 specimens were assigned into three groups (from group I to III and n=16) in relation to the resin cement used for luting the inlays. The cementation, finishing and polishing steps were followed according to the manufacturer’s instructions. Each group was subdivided into two equal subgroups (n=8) relative to the storage media used (0.01M buffered lactic acid of pH 4 or distilled water of pH 7). Finally, half of the specimens of each subgroup (n=4) was stored in each storage medium for 24h while the other half was stored for 168h.

Group I: 16 inlays were cemented by Variolink N resin cement using etch-and rinse adhesive system.

Group II: 16 inlays were cemented by Panavia F2.0 resin cement.

Group III: 16 inlays were cemented by RelyX Unicem resin cement.

After samples storage in both media for both periods of time, the 48 teeth in all groups were sectioned by an automated diamond saw (Isomet 4000, Buehler Ltd., Lake Bluff, IL, USA) under copious water coolant (Cool 2 water-soluble anticorrosive cooling lubricant, Buehler Ltd., Lake Bluff, IL, USA), with a concentration of 1:33, lubricant: water. Every tooth had repeated longitudinal sections in buccolingually and then mesiodistaly direction so as to obtain beams with an approximate surface area of $1mm^2$, each consisting of resin composite, resin cement and dentin.

For the longitudinal sectioning to be perpendicular to the occlusal surface of restored teeth, a specially designed gripping attachment was used to hold the acrylic blocks with mounted teeth firm in place parallel to the sectioning direction, thus maintaining the perpendicular relation between the cutting disc and the occlusal surface of tooth. The L-shaped attachment which is composed of a cylindrical metal ring soldered at its base to a metal rod that is used to mount the attachment into the diamond saw machine. Two axial grooves, perpendicular to each other were made on top surface of metal ring to facilitate accurate positioning and rotation of acrylic blocks inside the gripping attachment. The final components are two screws in-line with each other's so as to fix the acrylic blocks in place with minimal movement during sectioning.

After fixing in the gripping attachment, the teeth were sectioned by 0.3 mm diamond coated disc (Buehler, IL, USA) at 2050 rpm, 8.8 mm/min under copious coolant. Repeated sections were done in buccolingual direction then rotated 90^o clockwise and sectioned in mesiodistal. A final horizontal cut at level of cementoenamel junction was made so as to obtain beams. Four resultant beams were obtained from the central region of each tooth, where each beam was 0.9±0.1 mm in thickness. A digital caliper (Mitutoyo, Tokyo, Japan) was used to check the thickness of all beams.

Geraldeli's jig was used to mount the tested beams onto the universal testing machine (Instron, MA, USA). Each beam was aligned in the central groove of the jig and glued in place by its ends using cyanoacrylate based glue (Zapit, DVA Inc, USA). The jig was in turn mounted into the universal testing machine (Instron, MA, USA) with a load cell of 500 N. Tensile load was applied, at a cross-head speed of 0.5 mm/min, until bonding failure of the specimen occurred. Bond strength between inlay-resin cement and cavity floor which is dentin was calculated in MegaPascal (BluehillLite software, Instron, MA, USA). Specimen fragments were carefully removed from the jig with a scalpel and stored in their corresponding labelled plastic cones until examination of failure mode using Stereomicroscope, which can be classified as an adhesive failure (at resin cement/dentin or at resin cement/composite inlay), a cohesive failure (in composite or in resin cement or in dentin), and mixed failure. [41] Then, these fragments were gold sputtered (SPI Module-Sputter Carbon/Gold coater, EDEN instruments, Japan) and observed under a Scanning Electron Microscope (SEM) (JSM-6510LV, JEOL, Japan).

Statistical Analysis

Data were collected and analyzed using a statistical package (SPSS ™ Software, V.21, IBM, NY, USA). The data were normally distributed using Shapiro-Wilk Test, then analyzed using a Three-Way ANOVA Test to examine the effect of three variables (resin cement type, storage media and storage time) and the interaction of these factors on the μTBS. Tukey's post hoc multiple comparison Test was performed to compare the μTBS means between the tested groups, with a statistical significance set at α=0.05.

RESULTS

For all groups, Shapiro-Wilk Test showed that μTBS data followed a normal distribution pattern ($p>0.05$). Three-Way ANOVA Test revealed that μTBS was significantly ($p<0.05$) affected by the 'type of resin cement', 'storage media' and 'storage time'. Tukey's post hoc multiple comparison Test revealed that, SE Panavia F2.0 specimens stored in distilled water for 24h showed the highest μTBS values in comparison with the other groups. Conversely, the Variolink N (etch-and rinse-based luting system) specimens stored in lactic acid for 168h showed the lowest μTBS. In both storage media (distilled water and lactic acid) the 168h storage time for specimens showed a significantly lower μTBS compared to the 24h storage time ($p<0.05$).

Table 3. The mean μTBS results of tested groups and results of Tukey's post hoc multiple comparisons Test

Groups	N	Mean±SD
TE-W-24	16	11.87 ± 4.48^{e}
TE-W-168	16	10.32 ± 3.64^{e}
TE-W-24	16	11.44 ± 3.46^{e}
TE-W-168	16	8.54 ± 2.13^{f}
TE-W-24	16	23.35 ± 6.49^{a}
TE-W-168	16	17.32 ± 3.39^{c}
TE-W-24	16	20.73 ± 4.27^{b}
TE-W-168	16	11.66 ± 4.56^{e}
TE-W-24	16	17.79 ± 5.37^{c}
TE-W-168	16	14.45 ± 6.02^{d}
TE-W-24	16	14.16 ± 4.95^{d}
TE-W-168	16	10.42 ± 3.32^{e}
Total	192	14.34±6.19

N=Number W=Distilled water
SD=Standard deviation L=Lactic acid
TE=Total-etch (Variolink N) 24=24h
SE=Self-etch (Panavia F2.0) 168=168h
SA=Self-adhesive (RelyX Unicem)

Modes of Failure

The fractured specimens were examined using Stereomicroscope then SEM so as to determine the fracture pattern of tested resin cements to dentin substrate. In all groups, the predominant failure was adhesive failure (at resin cement/dentin), compared to cohesive failure (within resin cement) and mixed failure (dentin, resin cement and resin composite).

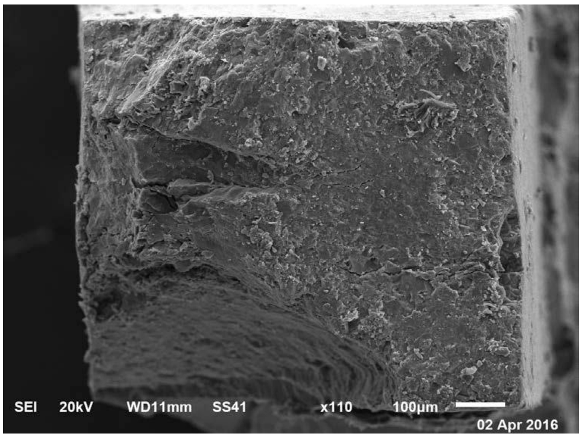

Fig 1. An adhesive failure at resin cement/dentin interface

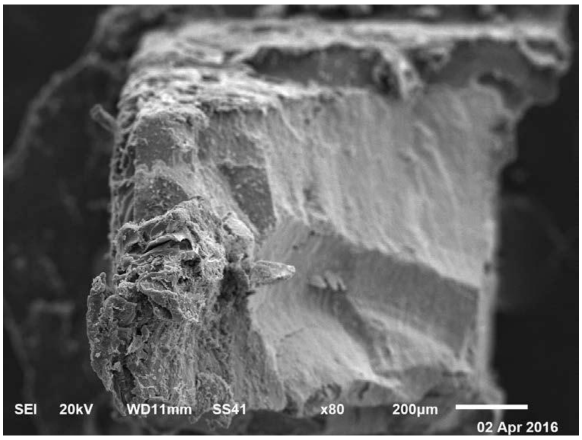

Fig 2. A cohesive failure within resin cement

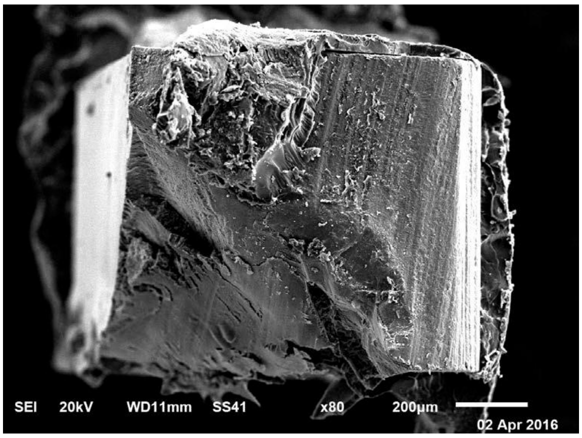

Fig 3. Mixed failure

DISCUSSION

In the current study, Large MOD cavities prepared in molars were chosen because, they are considered to be the least durable design, due to high loads exerted in this region and the extensiveness of restoration. [42] A standardized cavity preparation was made using special inlay diamond instruments, fixed in a high-speed handpiece which attached to specially designed appliance in order to avoid bias and incorrect interpretation of the results. [43] Laboratory resin composite was chosen because of its relatively low cost, easy fabrication, ability to absorb loads and its improved physical and mechanical properties, that allow it to be used as an alternative to esthetic dental prosthetic treatment where masticatory load could be required. [44] Moreover, indirect resin composite exhibits less polymerization shrinkage, which is still the main drawback for direct composite restorations. [45]

Micro-tensile bond strength test is more labor-intensive than tensile and shear strength tests. It was developed in order to overcome the non-uniform stress created in the conventional tensile bond strength test in laboratory studies. In principle, µTBS test uses specimens with a cross-sectional area of approximately 1mm^2. Decreasing the cross-sectional area results in a greater uniformly of the stress distribution, larger number of specimens can be produced with a reduced volume of material. In addition, µTBS test tends to provide a precise observation of the tooth/restoration bonding. Moreover, it allows less cohesive failure in the substrates and more adhesive failure at the bonding interface. This is thought to offer more accurate results for evaluating the real bonding potential between resin luting cement and composite inlays. [46,47]

Etch & rinse Variolink N resin cement showed the lowest µTBS, as acid etching of dentin surface with phosphoric acid is responsible for removing the smear layer and smear plugs, that results in increasing dentinal tubule diameter and dentin permeability. Also, rinsing step with water probably results in retention of a substantial volume of water within the widened tubule entrances. Such water may not be completely removed, which may contribute to blister growth at adhesive/resin interface. These blisters may have the effect of reducing the contact between resin cement and dentin surface that already affect the bond strength. [48, 49]

In brief, the multi-step etch & rinse approach is a rather aggressive procedure on dentin substrate, because of phosphoric acid etching which dissolves the natural protection of collagen and also removes them through rinsing, thereby producing a resin–collagen complex that is vulnerable to degradation upon water sorption, possibly enhanced by the documented enzymatic degradation process. [50] Moreover, when dentin is luted with conventional resin cements, the use of hydrophilic monomers as HEMA/BisGMA in the adhesive systems creates bonding interfaces more prone to degradation. Exposed collagen fibrils at the base of the hybrid become more susceptible to degradation. The aging protocol using water or acids, the calcium ions produce ideal conditions for endogenous Matrix Metalloproteinases (MMPs) present in dentin to degrade collagen. [51]

In case of SE Panavia F2.0 resin cement, which contains ED Primer that is responsible for only dissolving the smear layer, and does not remove the dissolved calcium phosphates as there is no rinse phase. So, producing a submicron hybrid layer with substantial hydroxyapatite-crystals (HAp) that still protecting the collagen fibrils and occlude the dentinal tubules. Moreover, the presence of functional monomers, in particular like 10-MDP (10-methacryloyloxy decyl-dihydrogen phosphate), have been proven to interact with this residual HAp through primary ionic bond. The resultant retention depends on twofold: micro-mechanical and chemical bonding mechanism closely resembles that of glass-ionomers. The formed ionic bond is stable in an aqueous environment so, the chemical bonding promoted by 10- MDP appeares not only more effective, but makes the interface more hydrophobic and thus better sealed. It can possibly achieve the direct benefit of bond durability; this may be due to its superior and hydrolytically stable bonding effectiveness. [52, 53]

Regarding to SA RelyX Unicem resin cement, the presence of the hydrophilic phosphoric acid monomers that simultaneously demineralized and infiltrate the tooth substrate, producing very superficial micromechanical retention and chemical reaction with the calcium ions of the hydroxyapatite present in dentin substrate forming ionic bond. As a result of its limited ability to demineralize and, to infiltrate the dentin substrate and its relative high viscosity contributes to low monomer diffusion, so reducing its micromechanical retention and depends only on chemical bonding. Thus, the bonding mechanism of SA resin cements to dentin is based on chemical reaction between dentin and resin cements. [16,54]

During cement setting, calcium atoms present in the dentin substrate/smear layer act as electron acceptors enhancing chemical reaction between the acidic resin monomers and the hard dental tissues. Calcium phosphates are mostly formed and do not exhibit a high bonding energy. The ability of this chemical bonding mechanism to overcome the limited diffusion of resin cement into dentin and reduced micromechanical retention is questionable since because chemical bonds involve calcium atoms present in low-adhered smear layer. The concept of using the smear layer as a bonding substrate might be the weak link for obtaining high bond strength. This bonding mechanism may explain the reduced effectiveness of the tested self-adhesive resin cement in bonding to dentin in the present study. [54- 56]

Although, there are significant improvements, but the adhesive interface remains the weakest link of restorations especially when dentin surface is involved. Two major mechanisms are included in the loss of bond strength over time 1. Hydrolytic degradation of hydrophilic resin within the hybrid layer and 2. Deterioration of dentin collagen fibrils. SA resin cements (RelyX Unicem) differ from conventional etch & rinse or SE resin cements for their interaction with dentin is only superficial due to limited decalcification, low diffusion and partial exposure of collagen fibrils at the base of the adhesive interface. Meanwhile, the use of conventional resin cements (Variolink N) create a discrepancy between the etching depth and resin penetration into dentin. As a consequence, a zone of exposed non-infiltrated by resin monomers at the base of hybrid layer is formed. Deterioration of such unprotected collagen fibrils plays an important role on the degradation of the adhesive interface resulting in loss of bond strength and consecutively reducing adhesive restorations durability. [57-60]

When different dentin depths were evaluated using the micro-tensile bond strength test, indirect restorations must reside for long periods in the oral cavity. As a consequence, water uptake plays an important hole in the long-term *in-vivo* resin–dentin bond degradation. Storage in aqueous media is a valid method to simulate aging of resin–dentin bonded restorations. It is still debatable whether the hydrolysis of the resin components is the principal mechanism of resin–dentin bond degradation. Most likely, the process occurs simultaneously for subsequent resin elution from hydrolytically unstable polymers within the hybrid layer leaving the collagen fibrils unprotected and liable to degradation. Even with a low initial pH, no demineralization or infiltration of the dentin surface below the smear layer is noticed with only mild/partial exposure of collagen fibrils. [52]

This study revealed that, SE luting cement strategy showed the highest value of μTBS to dentin. While, etch-and-rinse resin cement showed the lowest values of μTBS. So, hypothesis is rejected. A study by Aguiar et al showed agreement with this study where, μTBS that obtained by SA resin cements to dentin were higher than conventional ones after storage. [61] Also, Cetin et al agreed with the present study where, resin/dentin bond strength reduced after water storage. [62] But, Suzuki et al disagreed with this study where, etch & rinse resin cement exhibited the highest μTBS to dentin in comparison to SE and SA resin cements. [63] Results obtained by Bacchi and his colleagues were different from this study where, etch & rinse dual cured resin cement showed higher μTBS than SE and then SA resin cements. [64]

CONCLUSION

Conclusion Based on the results of the current study, and despite of the limitation of small sample size, it seems reasonable to conclude that micro-tensile bond strength showed good results with SE luting resin cement strategy.

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