RESEARCH ARTICLE

EVALUATION OF MICROTENSILE BOND STRENGTH AND NANOLEAKAGE AFTER DOUBLE LAYER ADHESIVE COATING APPLICATION USING BULK FILL COMPOSITE.

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Abstract

Aim: The present study used to evaluate nanoleakage and microtensile bond strength after double layer adhesive coating application. The tensile bond strength of an adhesive system is mostly influenced by the hybrid layer, followed by resin tags in the dentinal tubule and finally by chemical bonding. However, porosities can be observed at the bottom of hybrid layers. This phenomenon was termed “nanoleakage” Sano et al. They had seen incomplete penetration of hybrid layers during adhesive bonding techniques. Then nanoleakage effect has been discussed to be one factor negatively affecting the quality of dentinal bonding. Influence of nanoleakage on microtensile bond strength was determined using different visualization techniques. For instance, resin–dentin specimens were prepared and immersed in silver nitrate that penetrated into nanoleakage. Subsequently, specimens were broken at the adhesive interface (commonly using tensile testing), and nanoleakage was visualized indirectly on the exposed surface using scanning electron microscopy (SEM). It has been postulated that, between polymerization shrinkage and microtensile bond strengths (µTBS) a highly significant correlation was found because forces developed during the polymerization of dental restorative composites placed in a restricted setting, cause tension in the material, with possible subsequent distortion of the bond to the tooth.

Material and methods: 40 human molars stored in deionized water will used. Teeth will be divided into four equal groups according to application mode (self-etch vs. Etch-and-rinse mode). group A: using filtek™ bulk fill with single bond universal adhesive (self-etching mode), then application of hydrophobic resin coating (Heliobond).
group B: using filtek™ bulk fill with single bond universal adhesive (self-etching mode), without application of hydrophobic resin coating.
group C: using filtek™ bulk fill with single bond universal adhesive (etch and rinse mode), then application of hydrophobic resin coating.
group D: using filtek™ bulk-fill with single bond universal adhesive (etch-and-rinse mode), without application of hydrophobic resin coating.

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Keywords:-
Energy Dispersive X-ray spectrometry, Filtek Bulk fill composite resin, Heliobond, Scanning Electron microscope, Scotch bond universal.
**Results:** It was found that the filtekTM bulk fill composite with self-etch mode application of adhesive followed by coating application revealed the highest mean μ-TBS value (41.9 MPa) followed by filtekTM bulk fill composite with self-etch mode application of adhesive without coating application (39.7MPa). FiltekTM bulk fill with self-etch mode followed by coating, showed the highest Nanoleakage results (55.4%), while FiltekTM bulk fill with etch-and-rinse mode followed by coating, showed the lowest Nanoleakage results (39.9%). **Conclusion:** multiple consecutive coating during dentin bonding improved the bond strength for etch-and-rinse adhesive and reduced nanoleakage due to increases in the amount of resin impregnation within the hybrid layer. Nevertheless the better performance of the bulk-fill composites, all clinician should not consider that the bulk-fill composites are perfect substitutes for conventional composites.

**Introduction:**

The tensile bond strength at tooth restoration-interface is mostly influenced by the hybrid layer, followed by resin tags in the dentinal tubules [1-2]. However, porosities can be observed at the bottom of hybrid layers. This phenomenon was termed “nanoleakage” in which incomplete penetration of hybrid layers during adhesive bonding techniques [3]. Silver nitrate used to detect nanoleakage around composite restorations [4]. The low molecular weight silver nitrate tracer diffusion is similar to that of water and has been used to trace nanometer-sized water-filled spaces or “nanoleakage” within bonded interfaces, examination used scanning electron microscopy [5,6]. Several factors have a positive effect on the development of nanoleakage, including the type of solvent [7,8,9,10]. It has been pointed out that the defective dentinal sealing and fluid conductance across resin-bonded dentin may be significantly affected by the solvent used to saturate the acid-etched dentin [11,12]. New resin adhesives contain more acidic hydrophilic monomers, that already have higher amounts of water to increase monomer pollinate into wet dentin, this decreases the degree of polymerization of adhesive resin. This leadsto increased amount of silver uptake into the hybrid and adhesive layers (increased micropermeability). Different visualization techniques used to detect the Effect of nanoleakage on microtensile bond strength. Resin–dentin beams were prepared thin immersed non-destructively in silver nitrate that penetrated into nanoleakage. Then the specimens were broken at the adhesive interface and nanoleakage was analyzed on the exposed surface using scanning electron microscopy [5,6]. It has been revealed that, there is a highly significant correlation was found between polymerization shrinkage and microtensile bond strengths (μTBS) because forces developed during the polymerization of dental restorative composites placed in a restricted setting, cause tension in the material, with possible subsequent debonding at the tooth-restorement interface [13]. Furthermore, the quality of the bond between tooth and restorative materials could, also, be affected by the incompatibility between adhesive and restorative material as well as by the surface tensions of the two components coming into contact with each other [14]. The polymerization shrinkage of composites is also influenced by the geometric form of the cavity. When the ratio between the bounded to unbounded surfaces is higher than two, the stress generated by the composite shrinkage may exceed the bond strength to the cavity walls and produce marginal gaps [15]. When these problems are added to an incorrect placement technique and finishing mistakes, marginal leakage, inadequate anatomic form and proximal contacts occur clinically, which lead to a consequent reduction in the longevity of the restoration [16,17].

**Materials and methods:**

The study was an in vitro study conducted in Al-Azhar University for three years. FiltekTM Bulk Fill, shade A1, it is a visible light-curing posterior restorative resin based composite. Scotchbond Universal Adhesive also known as Single Bond Universal, Hydrophobic resin coating (Heliobond).

**Specimen preparation:**

A flat occlusal dentin surface was exposed in all teeth. The exposed dentin surfaces were further polished to standardize the smear layer [18]. The adhesive systems were applied according to the respective manufacturer’s instructions. Resin composite was built up in one increment then light-cured for 40 s using a LED light-curing unit set at 1200 mW/cm² (Radical, SDI Limited, Bays water, Victoria, Australia). the specimens were sectioned.
longitudinally in mesio-distal and bucco-lingual directions across the bonded interface with a slow-speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA) to obtain resin–dentin beams with a cross sectional area of approximately 1 mm²(Fig A) measured with a digital caliper (Digimatic Caliper, Mitutoyo, Tokyo, Japan)(Fig B).

Regarding group A:
Regarding subgroup A: (self-etching mode), Apply single bond universal adhesive to the entire preparation with a micro brush (3M, ESPE,USA) and rub it in for 20s. Air spread gently over the liquid for about 5s till no longer moves and the solvent is evaporated completely. Light-cure for 10s, then apply a very thin layer of Heliobond with a microbrush on the dental surface. Apply air to achieve an optimally thin layer. Light-cure applied for 10s. Then filtek™ bulk fill composite resin crowns were built up in one increment and cured using a light-cured for 40 s using a LED light-curing unit.

Regarding group B:
The same technique used in group A with one exception Heliobond will not applied.

Regarding group C:
(etch-and-rinse mode) using etchant (37% phosphoric acid gel) for 15s, rinse for 10s, Air dry 5s, Apply single bond universal adhesive, Heliobond and filtek™ bulk fill composite resin crowns were built up in one increment as mentioned before.

Regarding group D:
The same technique used in group C with one exception Heliobond will not applied.

Testing procedures:
For microtensile bond strength (µTBS):
The resin–dentin bonded beam tested under tension (Model5565, Instron Co., Canton, MA, USA) at 0.5 mm/min until failure. The µTBS values were calculated by dividing the load at failure by the cross-sectional bonding area [19,20]. The µTBS values (MPa) of all beams from the same tooth were averaged for statistical purposes.

For nanoleakage analysis:
The beams were placed in an ammonical silver nitrate solution in darkness for 24 h [21], and immersed in photo developing solution for 8 h under a fluorescent light to decrease silver ions into metallic silver grains. Resin–dentin interface were analyzed with a scanning electron microscope (Philips, XL 30, Eindhoven, The Netherlands) also analyzed using Energy dispersive X-ray spectrometry (EDX Philips, XL 30 W/TMP, Eindhoven, The Netherlands) The micrograph was taken in the center of the beam [21]. The mean NL (%) of all beams from the same tooth was averaged for statistical purposes. Comparison between the twelve different groups was made using four-dimensional mapping which was performed over 100 mm x 100 mm areas across the resin–dentine bonded interface, these areas covered the adhesive layer. The hybrid layer and unaffected dentine were visualized at 1000x magnification. Amount of silver grains that was penetrated at resin–dentin interface was calculated and statistically analyzed through energy levels of EDX analysis [22].

Statistical analysis:
Data analysis was performed in several steps. Initially, descriptive statistics for each group results. Multi-factorial ANOVA was used to detect effect of each variable (composite groups, adhesive type, application mode and resin coating). One way analysis of variance ANOVA was done between all subgroups. Student t-test was performed to detect significance between coat subgroups. Statistical analysis was calculated using software for Windows (Campina Grande, Paraiba state, Brazil). P values ≤0.05 are considered to be statistically significant in all tests.

Results:
µ-Tensile bond strength (µ-TBS):
Comparison of µ-tensile bond strength results between all groups as function of adhesive type, resin coating with self-etching application mode are summarized in Table/Fig Representatethe column chart of µ-tensile bond strength mean values for all groups as function of adhesive type, resin coating with self-etching application mode. It was found that the filtek™ bulk fill composite with self-etch mode application of adhesive followed by coating application (SE with coating) revealed the highest mean µ-TBS value (41.9 MPa) followed by filtek™ bulk fill composite with self-etch mode application of adhesive without coating application (SE without coating)(39.7 MPa). It
was found that the filtek™ bulk fill composite with self-etch mode application of adhesive followed by coating application revealed the highest mean µ-TBS value (41.9 MPa) followed by filtek™ bulk fill composite with self-etch mode application of adhesive without coating application (39.7MPa).

Filtek™ bulk fill (A2): it was found that group with Resin coating recorded statistically non-significant (P>0.05) higher µ-tensile bond strength mean value than groups without Resin coating as indicated by paired t-test.

| Composite type | Bulk fill (B) | P value |
|----------------|--------------|---------|
| Application mode & coating application | Self-etch with coating | 41.9±5.4 | 0.2127 ns |
| | Self-etch without coating | 39.7±3.5 | 0.002* |
| | Etch and rinse with coating | 38.6±4.7 | 0.0276* |
| | Etch and rinse without coating | 34.1±4.9 | 0.0299* |
| Mean ± SD | | 38.6±5.5 | |

*; significant (p < 0.05) ns; non-significant (p>0.05)

Etch-and-rinse application mode
With Resin coating: it was found that Filtek™ bulk fill recorded statistically significant (P<0.05) highest µ-tensile bond strength mean value.

Without Resin coating: It was found that Filtek™ bulk fill recorded statistically significant (P<0.05) highest µ-tensile bond strength mean value.

| Composite type | Bulk fill with coating | Etch & rinse (2) | P value |
|----------------|------------------------|-----------------|---------|
| Application mode & coating application | Self-etch(1) | 41.9±5.4 | 38.6±4.7 | 0.169 ns |
| | Bulk fill without coating | 39.7±3.5 | 34.1±4.9 | 0.035* |
| Mean ± SD | | 37.42±7.2 | 32.0±8.7 | |

*; significant (p < 0.05) ns; non-significant (p>0.05)

For Nanoleakage:
Nanoleakage results (%) for all composite groups as function of adhesive type, application mode and resin coating are summarized in Table/Fig 2represent the column chart of total nanoleakage mean values as function of application mode. Regarding different composite groups the Nanoleakage results revealed non statistical significant difference. For filtek bulk fill composite the group C (etch-and-rinse with coating) showed the lowest results (39.9%) while the group A (self-etch with coating) showed the highest results (55.4%).

Effect of composite material:
Irrespective of different adhesive (type, application mode) or resin coating, totally it was found that Filtek™ bulk fill (recorded statistically significant (P<0.05) highest nanoleakage mean value (46.3±5.49 %) as indicated by chi square test.

| Composite type | Bulk fill (B) | Mean± SD | Chi square test (P value) |
|----------------|--------------|----------|--------------------------|
| application mode & coating application | Self-etch with coating | 55.4% | 39.94±21.86 | 0.06 ns |
| | Self-etch without | 48.25% | 48.03±0.32 | |
| | Etch and rinse with coating | 39.9% | 23.4±23.33 | 0.101 ns |
Etch and rinse without coating | 41.78% | 44.84±4.33
Mean± SD | 46.3±5.49
Chi square test (P value) | 0.4679 ns

*: significant (p < 0.05) ns; non-significant (p>0.05)

Effect of application mode on Nanoleakage:
The results (%) of Nanoleakage with different application mode regarding of adhesive coating application and composite type are summarized in the following table:

| Application mode | Self-etch(1) | Etch-&-rinse(2) | Mean± SD | Chi square test (P value) |
|------------------|--------------|----------------|----------|-------------------------|
| Composite type& coating application | | | | |
| Bulk fill with coating | 55.4% | 39.9% | 46.95±9.97 |
| Bulkfill without coating | 48.25% | 41.78% | 45.02±4.57 | 0.201 ns |
| | 40.76±8.48 | 26.26±7.7 |
| Mean± SD | 0.001* |

*: significant (p < 0.05) ns; non-significant (p>0.05)

Nanoleakage (SEM and EDX analysis):
Nanoleakage patterns for all composite groups as function of adhesive type, application mode and resin coating are summarized as a following: The silver nitrate penetration method, combined with high magnification SEM by means of secondary electron or backscattered electron mode was used to provide much better information concerning the sealing ability of the restorations and the quality of the hybrid layer. In this study a comparison was performed between different four groups and their corresponding adhesive system by the use of Energy-dispersed X-ray (EDX) spectrum that recorded the amount of silver grains (wt%) presented in area (100-100 µm) at resin dentin interface and demonstrated it by Ag peaks. A thin layer of resin composite was used to avoid the possible effects of polymerization shrinkage. The length of silver penetration along the interface was not recorded, since this study was focused to determine if a variation in the nanoleakage patterns occurred among the materials tested. The results in this study demonstrated different leakage patterns depending on the dentin bonding systems tested. However, silver ion accumulations were often noted at the base of the hybrid layer for all materials.

Group A;
Table/Fig 3 a,b exhibited a highest amount of nanoleakage, with highest peak of Ag and the amount of Ag shown in Table4/Fig-4 was the highest one (55%) compared to the other samples.

Regarding Group B;
Table/Fig 4a,b, the granules of silverdepositions on the top of the hybrid layer showed that silver uptake was smaller than group A. This accommodated with amount of silver (48 wt%). The EDX spectrum demonstrated and identified those granules as silver by the sharp Ag peak.

Regarding Group C;
Table/Fig 5 a,b, the granules of silverdepositions on the top of the hybrid layer showed that silver uptake was very smallest, when compared with the other groups this accommodated with amount of silver (39 wt%). The EDX spectrum of the granules demonstrated and identified those granules as silver by the sharp Ag peak.
Regarding Group D:
Table/Fig 6 a,b the granules of silverdepositions on the top of the hybrid layer showed that silver uptake was slightly decreased than group C. This accommodated with amount of silver (41wt%). The EDX demonstrated analyze those granules of silver by the sharp Ag peak. Overall microtensile bond strength (MPa) and overall Nanoleakage values (%) are summarized in the (Table/Figure 7). Column chart of the mean values of µ-tensile bond strength measured in mega Pascal (MPa) with different composite restoration regarding application mode of adhesive and coating application. Also this chart show, the results (%) of Nanoleakage with different composite restoration regarding application mode of adhesive and coating application. The group A showed the highest µTBS values and the highest Nanoleakage values. The group C revealed the lowest µTBS values and high Nanoleakage value.

Discussion:-
The evaluation of relationship between microtensile and nanoleakage is an important procedure, further more development of adhesive systems that increase microtensile hence minimizing Nanoleakage to optimize dentin bonding also is more important. But the influences in marginal discoloration, recurrent caries, post-operative symptoms and the longevity of the composite restoration cannot be excluded.

For microtensile:
Bonding degradation typically occurs with hydrophilic simplified adhesives [23,24]. The demineralized collagen fibrils are vulnerable to time-dependent hydrolytic degradation by water, regardless of the bonding strategy. When the exposed collagen is not fully encapsulated by the polymerized adhesive monomers, nano-channels are left within the hybrid layer and/or demineralized dentin [25], disclosing areas of NL. Adhesive interfaces formed by simplified ER and SE adhesives behave as permeable membranes [26,27]. Once cured, SE adhesives allow transudation of dentinal fluid to the sur-face where it accumulates as droplets. However, degradation is less frequent when a more hydrophobic solvent-free adhesive coating is used [28,29]. HC has been shown to reduce the potential for hydrolytically degradation of the bonds and increase the longevity of resin–dentin interfaces both in vitro and in vivo [30,31].

Regarding hydrophobic resin coating vs. non coating, in our study, the use of HC may improve the mean (µTBS) for any of the adhesives in ER mode. However, when HC was omitted, this may reduce significantly mean TBS for all adhesives. This finding highlights the protective role that a HC may have on the adhesive interface when dentin is etched with phosphoric acid. In fact, adhesive for which the respective manufacturer recommends the ER strategy, SBU and GBP, resulted in lower NL when HC was used. Perdigão et al. speculated that the SBU have low variable nature, this may be due to its water content (10–15% by weight) [32]. The absorbed water may form hydrogen bonds with the hydrophilic and ionic domains (i.e. hydroxyl, carboxyl and phosphate groups) present in these hydrophilic adhesives [33]. Water is an important component of 1-step self-etch adhesives as it allows their ionization and permeation through the smear layer and underlying mineralized dentin [34]. However, the intrinsic hydrophilicity of self-etch adhesives jeopardizes their in vitro and clinical performance [35]. The application of HC over SBU improved mean (µTBS) for the SE strategy. However, no differences were observed between 24h and 6m mean (µTBS) for the ER strategy when compared to the group without HC. SBU is an “ultra-mild” self-etch adhesive (pH 2.7), with a two-fold bonding ability: (1) mechanical interlocking on dentin surface and (2) chemical interaction [36]. Stable calcium-methacryloyloxydecyl dihydrogen phosphate (MDP) complexes [37,38] formed within the partial demineralized dentin through nanolayering [39]. A second additional chemical mechanism is associated to the interaction of a polyalkenoic acid co-polymer (also known as Vitrebond co-polymer, or VCP) with calcium in hydroxyapatite [39]. This self-adhesiveness may be responsible for the excel-lent long-term performance of polyalkenoic-based materials [40]. It is believed that the composition and the resulting mechanical strength of the adhesives are better predictors for the immediate bond strength than the acidity of the adhesive. But chemical composition may be responsible for extent of diffusion-induced water movement and the amount of water within resin–dentin interfaces in the long-term [41]. HC resulted in greater mean µTBS of SBU/SE. The thickness of the adhesive layer may have increased [61] allowing the formation of a more densely packed hybrid layer with improved mechanical properties. The HC also increased the mean µTBS of SBU/SE, which may have been a result of enhanced adhesive layer hydrophobicity. The adhesive layer becomes less permeable to water movement, and less susceptible to water degradation [41]. Coating with a hydrophobic layer may couple more unsolvated hydrophobic monomers to the adhesive interface through copolymerization with the uncured adhesive surface, decreasing the relative concentration of retained solvent and unreacted monomers, thus enhancing the in situ degree of conversion. The monomer conversion continues after the polymerization process has finished, due to continuous propagation of free radicals, independently of the water storage conditions [42]. The hydrophobic layer may have protected the post-polymerization process. In a previous report, a 2-stepmild self-etch adhesive (Opti Bond XTR, Kerr Co., Orange,
CA, USA) resulted in higher mean TBS after 6 month water aging, in the same magnitude than the self-etch “golden standard” Clearfil SE Bond [43]. This may have been a result of the presence of the hydrophobic resin step in the 2-step self-etch adhesive, besides and a potential chemical bonding between a functional monomer molecule (GPDM, glycerol phosphate dimethacrylate) with calcium in hydroxyapatite.

For Nanoleakage:
Regarding analysis of NL results for ER and SE, we were unable to find a cause–effect relationship from the application of HC, as it occurred for µTBS. Some reductions were observed within groups with HC (SBU/ER). NL may be more related to the adhesive infiltration and sealing capability. It is well known that the quality of the resin–dentin bonds is affected by the extent of resin infiltration into the exposed collagen [44,45]. For ER, peritubular hybridization of the resin tags may not occur. For SE, the weakest zone in aged specimens is below the hybrid layer, due to poorer polymerization of the monomers within the bottom of the hybrid layer [46]. These findings corroborate our NL pattern observations for both ER and SE modes. Even if resin hydrolysis may negatively affect the long-term bonding stability, collagen depletion may also occur due to enzymatic degradation. Adhesive chemical formulations on both mineralized and demineralized dentin, cause induction and activation of matrix metallo-proteinases (MMP’s) regardless of the bonding strategy [47]. However, MMP’s activation that subsequently cause degradation, is believed to be more destructive for mild SE hybrid layers, than for ER hybrid layers as SE adhesives bond to dentin with less demineralization [48]. Regarding effect of bulk-fill composite, the lower shrinkage observed in FBF can be explained by the exclusion of the commonly added TEGDMA (286 g/mol), which has approximately half the molecular weight of the aforementioned dimethacrylates. Regarding the modulus development, higher modulus values were obtained in the high-viscosity composites. This finding is in agreement with the generally reported observation that the modulus of composites, which is in direct contrast with the shrinkage, increases with increased filler content [49,50,51,52]. The Acoustic emission analysis technique was used by Rayan Kim et al, evaluate the debonding behaviour of composites in human teeth. The quality of the bonded interface can also be assessed by confocal microscopy, [53] dye penetration, [54] microtensile bond strength, [55] and SEM, [56]. However, these methods only provide post-evaluation of the interface after degrading has already occurred by polymerization shrinkage stress. Recent studies using this technique have confirmed that a slower rate of polymerization [57], better bonding surface, low-shrinkage composites, and lower C-factor [58,59,60,61], are strongly associated with better resistance to interfacial debonding as evidenced by fewer Acoustic emission analysis (AE) events. The high-viscosity bulk-fill composites may not have advantages over the high-viscosity conventional composite. In contrast, compared with the low-viscosity conventional composite, the low-viscosity bulk-fill composites showed better performance in terms of shrinkage stress and debonding behaviour [62,63,64]. Therefore, provided that low-viscosity bulk-fill composites have the mechanical properties required to replace dentine for clinical function, the low-viscosity bulk-fill composites would save clinical time for filling deep cavities, assuming a 2-mm occlusal space is left for subsequent capping with a conventional high-viscosity composite. Recent evidence from a clinical study by van Dijkenet al. [65,66] has shown that the bulk-fill composite demonstrated comparable clinical outcomes to a conventional incrementally placed composite, when the specific bulk-fill composite was placed in bulk, followed by a 2 mm capping with a hybrid composite, as per manufacturer’s instruction [67,68,69,70]. However, despite the benefits of the bulk-fill composites, since their mechanical properties appear to be lower when compared with conventional composites, clinicians should keep in mind that bulk-fill composites cannot entirely replace conventional composites.

Significance:
This study was done to improve the clinical performance of adhesive application and composite restoration at the tooth restoration interface. This had achieved by increase the microtensile bond strength also decrease the Nanoleakage at the bottom of the hybrid layer, hence increase the longevity of the restoration which is the main goal of each dentist nowadays.

Limitation:
The Microtensile and nanoleakage measurements might be done by two methods to compare the results and evaluate the methodology used in this study.

Conclusion:
Under the conditions of this study and with respect to the materials used, following conclusion could be drawn:
1. The application of hydrophobic coating enhances the microtensile bond strength (µTBS). 2. Nanoleakage is positively affected by the application of hydrophobic coating. 3. The types and composition of the composite filling material produces different effects on microtensile bond strength (µTBS) and Nanoleakage. 4. Adhesive application
mode has positive effect on microtensile bond strength (µTBS). 5. Although Nanoleakage (NL) pattern is material-dependent, it seems not to be related with the adhesive strategy. 6. The degree of Nanoleakage (NL) and its location depends on the adhesives used.

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Conflicts of interest:
There are no conflicts of interest.

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Abbreviations:
SEM= Scanning Electron Microscope. EDX= energy dispersive X-ray spectrometry. FBF=filtek bulk fill. HB= heliobond. SBU= single bond universal. MTBS= microtensile bond strength. NL= Nanoleakage. SE= self-etch mode. ER= etch-and-rinse mode.

Trade names:
The materials, single bond universal and filtek bulk fill composite (3M ESPE, St. Paul, MN, USA also Heliobond (IvoclarVivadent, Schaan, Liechtenstein)

Resin dentin beam measured by caliper

Figure 1
Figure 2

Fig 3

Fig 4

Fig. 5a
Representative backscattered SEM images of resin–dentin interfaces (Co = composite resin; HL = hybrid layer; De = dentin). For IEC, a slight decrease in NL was observed when HC was omitted (white arrows). Backscattered electron image of SEM 29(a) and corresponding EDX spectrum 29(b) of the fractured surface of resin dentin beam side. The granules of silver depositions at the bottom of the hybrid layer show that silver uptake was more than ISC, due to absence of hydrophobic coating. This accommodated with amount of silver (47.8 wt%) in table 15 at energy level (L).

Representative backscattered SEM images of resin–dentin interfaces (Co = composite resin; HL = hybrid layer; De = dentin). For BSC, a slight decrease in NL was observed (white arrows) when compared to BSC. Backscattered electron image of SEM 31(a) and corresponding EDX spectrum 31(b) of the fractured surface of resin dentin beam side. The granules of silver depositions on the top of the hybrid layer, showed that silver uptake was highest among the all tested groups and this accommodated with amount of silver (48.25 wt%) in table 17 at energy level (L).
Representative backscattered SEM images of resin–dentin interfaces (Co = composite resin; HL = hybrid layer; De = dentin). For BEC an increase in NL was observed when HC was used (white arrows) Backscattered electron image of SEM 32(a) and corresponding EDX spectrum 32(b) of the fractured surface of resin dentin beam side. The granules of silver depositions at the bottom of the hybrid layer, showed that silver uptake was low when compared with BSC, BSC₀ tested groups and this accommodated with amount of silver (39.9 wt%) in table 18 at energy level (L).

Representative backscattered SEM images of resin–dentin interfaces (Co = composite resin; HL = hybrid layer; De = dentin). a slight decrease in NL was observed when HC was omitted (white arrows) Backscattered electron image of SEM and corresponding EDX spectrum of the fractured surface of resin dentin beam side. The granules of silver depositions at the bottom of the hybrid layer show that silver uptake was more than BEC, due to absence of hydrophobic coating. This accommodated with amount of silver (41.78 wt%) at energy level (L).
Fig 1: Mitotoya digital caliper
Fig 2: Resin dentin beam after specimen preparation
Figure legend 3: Microtensile bond strength column chart (Mpa)
Figure legend 4: Nanoleakage column chart
Figure legend 5: SEM & EDX analysis for group A
Figure legend 6: SEM & EDX analysis for group B
Figure legend 7: SEM & EDX analysis for group C
Figure legend 8: SEM & EDX analysis for group D
Figure 9: Column chart represents the total effect of both Nanoleakage and microtensile

Table 1: Results of microtensile bond strength test

| Composite type | Bulk fill (B) |
|----------------|---------------|
| Application mode & coating application | |
| SE with coating | 41.9±5.4 |
| SE without coating | 39.7±3.5 |
| ER with coating | 38.6±4.7 |
| ER without coating | 34.1±4.9 |

Table 2: Results of Nanoleakage analysis

| Composite type | Bulk fill (B) |
|----------------|---------------|
| Application mode & coating application | |
| SE with coating | 55.4% |
| SE without coating | 48.25% |
| ER with coating | 39.9% |
| ER without coating | 41.78% |

Table 3: Amount of silver ions at energy level L between different groups

| Groups | element | Amount of silver | Energy level |
|--------|---------|------------------|--------------|
| Group A | Ag | 55.4% | L |
| Group B | Ag | 48.25% | L |
| Group C | Ag | 39.9% | L |
| Group D | Ag | 41.78% | L |

Table 4: Overall amount for Nanoleakage & microtensile

| Composite type | Bulk fill (B) |
|----------------|---------------|
| Application mode & coating application | |
| SE with coating | 55.4% |
| SE without coating | 48.25% |
| ER with coating | 39.9% |
| ER without coating | 41.78% |
| Bulk fill (B) | |
| Application | SE with coating | 41.9±5.4 |
| Mode & Coating Application | SE without coating | ER with coating | ER without coating |
|---------------------------|--------------------|----------------|-------------------|
|                           | 39.7±3.5           | 38.6±4.7       | 34.1±4.9          |

**References:**

1. Guzman-Armstrong S, Armstrong S, Qian F. Relationship between nanoleakage and microtensile bond strength test at the resin–dentin interface. Oper Dent 2003;28:60–6.

2. Van Meerbeek B, Van Landuyt K, De Munck J, Hashimoto M, Peumans M, Lambrechts P, et al. Technique-sensitivity of contemporary adhesives. Dent Mater J 2005;24:1–13.

3. Sano H, Shono T, Takatsu T, Hosoda H. Microporous dentin zone beneath resin-impregnated layer. Oper Dent 1994;19:59–64.

4. Wu W, Cobb E, Dermann K, Rupp N. Detecting margin leakage of dental composite restorations. J Biomed Mater Res 1983;17:37–43.

5. Sano H, Takatsu T, Ciucchi B, Horner JA, Matthews WG, Pashley DH. Nano-leakage: leakage within the hybrid layer. Oper Dent 1995;20:18–25.

6. Dorfer C, Staehle H, Wurst M, Duschner H, Pioch T. The nanoleakage phenomenon: influence of different dentin bonding agents, thermocycling and etching time. Eur J Oral Sci 2000;108:346–51.

7. Kanca J. Effect of resin primer solvents and surface wetness on resin composite bond strength to dentin. Am J Dent 1992;5:213–5.

8. Nakabayashi N. Bonding of restorative materials to dentin: the present status in Japan. Int Dent J 1985;35:145–54.

9. Nakabayashi N, Ashizawa M, Nakamura M. Identification of a resin–dentin hybrid layer in vital human dentin created in vivo: durable bonding to vital dentin. Quintessence Int 1992;23:135–41.

10. Bouillaguet S, Duroux B, Ciucchi B, Sano H. Ability of adhesivesystems to seal dentin surfaces: an in vitro study. J Adhes Dent 2000;2(3):201–8.

11. Pashley DH, Tay FR, Carvalho RM, Rueggeberg FA, Agee KA, Carrilho M, et al. From dry bonding to water-wet bonding to ethanol-wet bonding. A review of the interactions between dentin matrix and solvated resins using a macromodel of the hybrid layer. Am J Dent 2007;20:7–20.

12. Nikaido T, Kunzelmann K, Ogata M, Harada N, Yamaguchi S, Cox C, et al. The in vitro dentin bond strengths of two adhesive systems in class I cavities of human molars. J Adhes Dent 2002;4:31–9.

13. O’Keefe K, Powers J. Adhesion of resin composite core materials to dentin. Int J Prosthodont 2001;14:451–6.

14. Felliżer A, Gee A, Davidson C. Setting stress in composite resin in relation to configuration of therestoration. J Dent Res 1987;66:1636–9.

15. Llena P, Navarro L, Llacer V, Ferrandez A. Composite resin inlays: a study of marginal adaptation. Quintessence Int 1993;24:429–33.

16. Leinfelder KF. New developments in resin restorative systems. J Am Dent Assoc 1997;128:573–81.

17. Pashley DH, Tao L, Boyd L, King GE, Horner JA. Scanning electron microscopy of the substructure of smear layers inhuman dentine. Arch Oral Biol 1988;33:265–70.

18. Hipólito VD1, Alonso RC, Carrilho MR, Anauate Netto C, Sinhoreti MA, Goes MF. Microtensile Bond Strength Test and Failure Analysis to Assess Bonding Characteristics of Different Adhesion Approaches to Ground versus Unground Enamel. Braz Dent J 2011;22:122-28.

19. Ding PG1, Matzer AR, Wolff D, Mente J, Pioch T, Staehle HJ, Dannenwitz B. Relationship between microtensile bond strength and submicron hiatus at the composite–dentin interface using CLSM visualization technique. Dent Mater 2010;26:257-63.

20. Tay FR, Pashley DH, Yoshiyama M. Two modes of nanoleakage expression in single-step adhesives. J Dent Res 2002;81:472–6.

21. Santini A and Miletic V. Comparison of the hybrid layer formed by Silorane adhesive, one-step self-etch and etch and rinse systems using confocal micro-Raman spectroscopy and SEM. J Dent Res 2008;36:683-91.

22. Hashimoto M, Fujita S, Kaga M, Yawaka Y. In vitro durability of one-bottle resin adhesives bonded to dentin. Dent Mater 2007;26:677–86.

23. Osorio R, Pisani-Proenca J, Erhardt MC, Osorio E, Aguilera FS, Tay FR, et al. Resistance of ten contemporary adhesives to resin-dentine bond degradation. J Dent 2008;36:
25. Sano H, Shono T, Takatsu T, Hosodo H. Microporous dentinzone beneath resin-impregnated layer. Oper Dent 1994;19:59–64.
26. Tay FR, Pashley DH, Suh BI, Carvalho RM, Itthagaruna A. Single-step adhesive are permeable membranes. J Dent 2002;30:371–82.
27. Tay FR, Pashley DH. Have dentin adhesives become too hydrophilic? J Can Dent Assoc 2003;69:726–31.
28. Gamborgi GP, Loguercio AD, Reis A. Influence of enamel border and regional variability on durability of resin-dentin bonds. J Dent 2007;35:371–6.
29. Abdalla AI, Feilzer AJ. Four-year water degradation of atotal-etch and two self-etching adhesives bonded to dentin. J Dent 2008;36:611–7.
30. Reis A, Leite TM, Matte K, Michels R, Amaral RC, Geraldelli S, et al. Improving clinical retention of one-step self-etching adhesive systems with an additional hydrophobic adhesive layer. J Am Dent Assoc 2009;140:877–85.
31. Reis A, Albuquerque M, Pegoraro M, Mattei G, Bauer JR, Grande RH, et al. Can the durability of one-step self-etching adhesives be improved by double application or by an extralayer of hydrophobic resin? J Dent 2008;36:309–15.
32. Perdigão J, Sezinando A, Monteiro PC. Laboratory bonding ability of a multi-purpose dentin adhesive. Amer J Dent 2012;25:153–8.
33. Van Landuyt KL, Snaauwaert J, De Munck J, Coutinho E, Poitevin A, Yoshida Y, et al. Origin of interfacial droplets with one-step adhesives. J Dent Res. 2007;86:739–44.
34. Perdigão J. New developments in dental adhesion. Dent Clin North Am 2007;51:333–57.
35. Perdigão J, Dutra-Corrêa M, Anauate-Netto C, Castilhos N, Carmo AR, Lewgoy HR, et al. Two-year clinical evaluation of self-etching adhesives in posterior restorations. J Adhes Dent 2009;11:149–59.
36. Yoshida Y, Yoshihara K, Nagaoka N, Hayakawa S, Torii Y, Ogawa T, et al. Self-assembled nano-layering at the adhesive interface. J Dent Res 2012;91:376–81.
37. Perdigão J, Reis A, Loguercio AD. Dentin adhesion and MMPs: a comprehensive review. J Esthet Restor Dent 2013;25:219–41.
38. Yoshihara K, Yoshida Y, Hayakawa S, Nagaoka N, Torii Y, Osaka A, et al. Self-etch monomer-calcium salt deposition on dentin. J Dent Res 2011;90:602–6.
39. Yoshida Y, Yoshihara K, Nagaoka N, Hayakawa S, Torii Y, Ogawa T, et al. Self-assembled nano-layering at the adhesive interface. J Dent Res 2012;91:376–81.
40. Trairatvorakul C, Itsaraviriyakul S, Wiboonchan W. Effect of glass ionomer cement on the progression of proximal caries. J Dent Res 2011;90:99–103.
41. Hashimoto M, Tay FR, Sano H, Kaga M, Pashley DH. Diffusion-induced water movement within resin-dentin bonds during bonding. J Biomed Mater Res B Appl Biomater 2006;79:453–8.
42. Ye Q, Spencer P, Wang Y, Misra A. Relationship of solvent to the photopolymerization process, properties, and structure in model dentin adhesives. J Biomed Mater Res 2007;80:342–50.
43. Sezinando A, Perdigão J, Regalheiro R. Dentin bond strengths of four adhesion strategies after thermal fatigue and 6-month water storage. J Esthet Restor Dent 2012;20:345–55.
44. Hashimoto M, Tay FR, Sano H, Kaga M, Pashley DH. Diffusion-induced water movement within resin-dentin bonds during bonding. J Biomed Mater Res B Appl Biomater 2006;79:453–8.
45. Tay FR, Pashley DH, Yoshiyama M. Two modes of nanoleakage expression in single-step adhesives. J Dent Res 2002;81:472–6.
46. Kim J, Mai S, Carrilho MR, Yiu CK, Pashley DH, Tay FR. An all-in-one adhesive does not etch beyond hybrid layers. J Dent Res 2010;89:482–7.
47. Marchesi G, Frassetto A, Mazzoni A, Apolonio F, Diolosà M, Cadenaro M, et al. Adhesive performance of a multi-modal adhesive system: 1-year in vitro study. J Dent 2014;42:603–12.
48. Zheng P, Zaruba M, Attin T, Wiegand A. Effect of different matrix metalloproteinase inhibitors on microtensile bond strength of an etch-and-rinse and a self-etching adhesive to dentin. Oper Dent 2015;40:80–6.
49. Masouras K, Silikas N, Watts DC. Correlation of filler content and elastic properties of resin-composites. Dent Mater 2008;24:932–9.
50. Davidson CL, Feilzer AJ. Polymerization shrinkage and polymerization shrinkage stress in polymer-based restorative. J Dent 1997;25:435–40.
51. Lee JH, Um CM, Lee IB. Rheological properties of resin composites according to variations in monomer and filler composition. Dent Mater 2006;22:515–26.
52. Fok AS. Shrinkage stress development in dental composites—an analytical treatment. Dent Mater 2013;29:1108–15.
53. Sauro S, Mannocci F, Toledano M, Osorio R, Thompson I, Watson TF. Influence of the hydrostatic pulpal pressure on droplets formation in current etch-and-rinse and self-etch adhesives: a video rate/TSM microscopy and fluid filtration study. Dent Mater 2009;25:1392–402.
54. Leeveiloj C, Cochran MA, Matis BA, Moore BK, Platt JA. Microleakage of posterior packable resin composites with and without flowable liners. Oper Dent 2001;26:302–7.
55. Takemori T, Chigira H, Itoh K, Hisamitsu H, Wakumoto S. Factors affecting tensile bond strength of composite to dentin. Dent Mater 1993;9:136–8.
56. Ciucchi B, Bouillaguet S, Delaloye M, Holz J. Volume of the internal gap formed under composite restorations in vitro. J Dent 1997;25:30–12.
57. Cho NY, Ferracane JL, Lee IB. Acoustic emission analysis of tooth-composite interfacial debonding. J Dent Res 2013;92:76–81.
58. Gu JU, Choi NS. Evaluation of marginal failures of dental composite restorations by acoustic emission analysis. Dent Mater J 2013;32:398–404.
59. Kim RJ, Choi NS, Ferracane J, Lee IB. Acoustic emission analysis of the effect of simulated pulpal pressure and cavity type on the tooth-composite interfacial de-bonding. Dent Mater 2014;30:876–83.
60. Li H, Li J, Yun X, Liu X, Fok AS. Non-destructive examination of interfacial debonding using acoustic emission. Dent Mater 2011;27:964–71.
61. Liu X, Li H, Li J, Lu P, Fok AS. An acoustic emission study on interfacial debonding in composite restorations. Dent Mater 2011;27:934–41.
62. Van Dijken JW, Pallesen U. A randomized controlled three year evaluation of ‘‘bulk-filled’’ posterior resin restorations based on stress decreasing resin technology. Dent Mater 2014;30:245–51.
63. Bucuta S, Ilie N. Light transmittance and micro-mechanical properties of bulk fill vs. conventional resin based composites. Clin Oral Investig 2014;18:1991–2000.
64. Ilie N, Bucuta S, Draenert M. Bulk-fill resin-based composites: an in vitro assessment of their mechanical performance. Oper Dent 2013;38:618–25.
65. Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, Leloup G. Physico-mechanical characteristics of commercially available bulk-fill composites. J Dent 2014;42:993–1000.
66. Zorzin J, Maier E, Harre S, Fey T, Belli R, Lohbauer U, et al. Bulk-fill resin composites: polymerization properties and extended light curing. Dent Mater 2015;31:293-301.
67. Fukuda R, Yoshida Y, Nakayama Y, Okazaki M, Inoue S, SanoH, et al. Bonding efficacy of polyalkenoic acids to hydroxyapatite, enamel and dentin. Biomater 2003;24:1861–7.
68. Reis A, Grande RH, Oliveira GM, Lopes GC, Loguerchio AD. A2-year evaluation of moisture on microtensile bond strength and nanoleakage. Dent Mater 2007;23:862–70.
69. Sano H, Yoshiyama M, Ebisu S, Burrow MF, Takatsu T, Ciucchi B, et al. Comparative SEM and TEM observations of nanoleakage within the hybrid layer. Oper Dent 1995;20:160–7.
70. Van Meerbeek B, Yoshida Y, Lambrechts P, Vanherle G, Duke E, Eick J, et al. A TEM study of water-based adhesive systems bonded to dry and wet dentin. J Dent Res 1998;77:50–9.