Micro-Tensile Bond Strength Of Three Different Dual Cure Resin Composites Bonded To Dentin

Atya A. Alzarouq[1], Rabab Mehesen[2], Ashraf I. Ali[3]

Abstract:

Objective: To evaluate the micro-tensile bond strength (μTBS) and micro-morphological analysis of composite dentin interface of dual-cure resin composites bonded to dentin. **Materials and Methods:** For the micro-tensile bond strength test, sixty sound human maxillary and mandibular molars were collected. After embedding the selected molars in an acrylic resin block, the occlusal enamel and superficial dentin of each tooth were removed, exposing the mid-dentin area. They were assigned randomly into three groups (n=20) according to the restorative system. Then both groups were further divided into two subgroups; subgroup 1 for immediately tested specimens after 24 h (n=10), and subgroup 2 for delayed tested specimens after 6 months (n=10). Resin composite build-ups were made incrementally 2mm up to reach full thickness. The blocks were sectioned and then subjected to a micro-tensile (μTBS) test at 24h and after 6 months. Two specimens from each subgroup were used for the micro-morphological analysis of the adhesive/dentin interface using a scanning electron microscope (SEM) (n=12) . **Results:** Regarding the (μTBS) test, the One-way ANOVA test showed a significant difference among all groups(p=0.002). Tukey post hoc multiple comparison test showed that All-bond universal adhesive had the highest mean bond strength, which was significantly different from Tetric N bond UA and Futurabond M+ UA. Also, Tetric N bond UA showed the lowest mean bond strength values. All-bond universal and Futurabond M+ universal adhesives produced hybrid layers with high resin tag infiltration. While the Tetric N-bond universal adhesive showed the typical micro-morphological pattern of the hybrid component of this material. **Conclusions:** The present study is based on the outcome of the (μTBS) test, for universal adhesives, the addition of a dual-curing activator did not influence bond strength to dentin, and there was no relation between the micro-tensile bond strength and the micro-morphological patterns in all tested adhesives. Aging affects negatively the bond strength.

Introduction:

The composite resin restoration materials have become very popular because they can be bonded to the remaining tooth structure to provide resistance and retention for the final restoration.[1] However, the restoration of ideal occlusal anatomical form and proper interproximal contact area in damaged teeth is always considered a clinical challenge.[2] Dual cure resin composite (DCRC) material was invented to address the issue of cure depth.[3] Polymerization reaction begins with light exposure to the superficial surface and continues with chemical activation in the deeper layers.[4,5] Self-cured (SC) and dual-cured (DC) resin composites are incompatible with certain bonding systems, and failure occurs at the adhesive interface.[6,7] This is associated with using simplified adhesives due to the residual uncured acidic monomers from the oxygen-inhibited layer of the cured adhesives that remain in direct contact with resin composite.[8] This reaction occurs between adhesives and the catalytic components (aromatic tertiary amines) of chemically-cured composite, resulting in a low polymerization rate.[9,10] There are few published studies until now that evaluated the use of universal adhesive with dual-cured resin composite. Furthermore, there is still a lack of knowledge on the effect of dual-cured core buildup composite material affected by the type of universal adhesive systems. Hence, the current study was conducted to assess the bond strength of dual-cure resin composite bonded with different universal adhesives.

Null Hypothesis: This study was designed to test the null hypothesis that neither the micro-tensile bond strength nor the micro-morphological analysis of dual-cured core buildup composite material would be affected by the type of universal adhesive system

Materials and Methods:

Materials: Three different dual-cure resin composite (DCRC) and universal adhesive restorative systems (Core flo DC lite /All-bond universal, Multicore flow/ Tetric N-Bond universal, and Repilda DC/ Futurabond M^+ universal) were used in this study.

Methods:

Selection of teeth: A total number of 60 sounds human maxillary and mandibular molars were collected from the surgery clinic (Faculty of Dentistry, Mansoura University). The teeth used for this study were collected according to the regulation of the Ethical Committee of the Faculty of Dentistry-Mansoura University. These teeth were extracted due to periodontal diseases. Teeth were cleaned of soft tissue and calculus deposits with an ultrasonic scaler (GUILIN WOODPECKER medical instrument Co, LTD). Then, they were disinfected for 24 hours in an aqueous solution of 0.5 % Chloramine-T and washed under running water.

[1]*Postgraduate Student, Department of Operative Dentistry, Faculty of Dentistry, Mansoura University35516, Mansoura, Egypt.*
Email: atyaomar238@gmail.com
[2] *Lecturer, Department of Operative Dentistry, Faculty of Dentistry, Mansoura University35516, Mansoura, Egypt.*
[3] *Associate Professor, Department of Operative Dentistry, Faculty of Dentistry, Mansoura University35516, Mansoura, Egypt.*

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Preparation of teeth: The teeth were fixed in polyvinyl chloride (PVC) cylinders of 1.8 cm diameter and 2 cm height to accommodate the molars with large roots. These cylinders were filled with auto-polymerizing acrylic resin. Then, in the dough stage of acrylic resin, each tooth was invested vertically in the center of the cylinder with a corresponding metal ring, and two opposing screws were used to fix teeth centralized in acrylic resin blocks. The screws were used to hold the tooth in place in a centralized position, parallel to the long axis of the mold, during the setting of acrylic resin. After that, the teeth were mounted in an automated diamond saw (Isomet 4000, Buehler Ltd., Lake Bluff, IL, USA).

Teeth Grouping: All the teeth were divided according to the type of restorative systems used into three main groups (n=20) as follows: Group I in which the teeth were restored with All-bond universal adhesive, and Core flotmdc Lite. Group II in which the teeth were restored with Tetric N bond universal adhesive, and Multicore*Flow. Group III in which the teeth were restored with Futurabond M+ universal adhesive, and Repilda DC. Then, each main group was subdivided into two subgroups according to storage period: Subgroup 1 in which teeth were not stored and tested immediately after 24h (n=10).Subgroup 2 in which teeth were stored for 6 months (n=10) in distilled water at 37±1ºc in an incubator (BTC, Model: BT1020, Egypt), and the storage medium was replaced with a new fresh one every week.

Restorative procedures: At first, a split plastic mold with a central space and dimensions of (5mm width, 7mm length, and 4mm height) was used for the insertion of RC on the dentin surface. The mold had an outer frame flanked by the inner split mold to make it easier to be attached to teeth, and prevent any damage to the RC during the removal of the mold. All the restorative systems procedures were performed according to manufacturers' instructions and all universal adhesives were used in self-etch mode (without phosphoric acid etching) as directed by the manufacturers.Regarding Group I, the teeth were removed from distilled water, and then the dentin surface was dried with gauze to keep the surface hydrated. Two separate coats of the adhesive were applied to the dentin surface using a Micro brush with continuous agitation for 10 seconds, and the adhesive layer was dried by gentle air using the tip of plastic air spray pressure in a dental unit for 5 seconds to remove any solvents. The adhesive was light cured for 20 seconds with an LED light-curing Unit (Eli par™ Deep Cure-S LED Curing Light).Following the bonding procedure, DCRC was Applied with an auto-mix dual-syringe to fabricate build-up incrementally with 2 mm thickness by Inserting the mixing tip into the mold and then injecting RC to the dentin surface. A small condenser (stainless steel condenser #2, slandered handle with a 1.7mm diameter and has a flat end. (A.G.M-Pakistan) was used for adaptation of RC into the dentin surface, and then light cured for 30 seconds. After that, the DCRC was applied to fabricated buildup in a second increment as mentioned before; to achieve a total thickness of 4mm. Then, a piece of transparent Mylar strip was pressed over the surface of the split mold to ensure a smooth superficial surface and ensure adaptation of the material and cured for 30 seconds. To confirm that the materials were completely set, the block was light cured all around for additional 10 seconds on each side after the mold was removed.

Regarding Group II, the teeth were removed from distilled water, the teeth were removed from distilled water, and then the dentin surface was dried as mentioned before. The adhesive was applied to the dentin surface using a micro brush with continuous agitation for 10 seconds, and the adhesive layer was dried by gentle air using the tip of plastic air spray pressure in a dental unit for 5 seconds to remove any solvents. The adhesive was light cured for 20 seconds with an LED light-curing unit. Following bonding procedures, DCRC was applied as mentioned before. The DCRC was then cured after the first and second increment for 40 seconds. To confirm that the materials were completely set, the block was light cured all around for additional 20 seconds on each side after the mold was removed. Regarding Group III, the teeth were removed and dried as mentioned before. Then, Futurabond M+ universal adhesive was used by mixing one drop with one drop of Futurabond M+ Dual Cure Activator in a mixing palette with a disposable applicator for 3 seconds. This mix was applied to dentin using a micro-brush with continuous agitation for 10 secs, and then the adhesive layer was dried by gentle air using the tip of plastic air spray pressure in a dental unit for 5 seconds to remove any solvents. The adhesive was light cured for 20 seconds with an LED light-curing unit. Following bonding procedures, DCRC was applied as mentioned before. The DCRC was then cured after the first and second increment for 40 seconds. To confirm that the materials were completely set, the block was light cured all around for 10 seconds on each side after the mold was removed.

Micro-tensile bond strength test: The immediate subgroups were sectioned and tested after 24h of the restorative procedure. The delayed subgroups were sectioned and tested after 6month of aging. Each tooth was sectioned longitudinally in both "x" and "y" directions through the bonded interface with the Isomet cutting machine (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.

Statistical Analysis: All the data of this study were collected, tabulated, and statistically analyzed using version 22 of IBM SPSS Statistics software for NY). After testing the normality using Shapiro–Wilk test

Table 1: Comparison between μTBS means of immediate and delayed subgroups for each restorative systems

Mpa	G 1 All-bond universal, coreflo lite dc composite	G2 Tetric-N bond universal, Multicore flow composite	G 3 Futurabond M+ universal, Rebilda dc composite	Test of significance
Immediate	24.43±10.71 ^{AB}	14.96±8.23^{A}	20.40±8.02^{B}	F=6.85P=0.002*
Delayed	19.04±8.38^{A}	13.91±6.05^{A}	17.84±8.32	F=3.07 P=0.053

F: One Way ANOVA test, parameters described as mean ±SD, similar superscripted letters denote significant difference between groups by Post Hoc Tukey test. *Statistically significant. AB: There was a significant difference when compared between groups.

quantitative variables were summarized as mean±SD for normally distributed data, and median, range for non-normally distributed ones. For normally distributed data, the Student t-test was used to compare 2 independent Groups, and the One Way ANOVA test was used to compare more than 2 independent Groups with the Post Hoc Tukey test to detect pair-wise comparison. Mann-Whitney test was used to compare 2 or more independent Groups for non-normally distributed data. The significance of the results was judged at a P value ≤ 0.05.

Micro-morphological analysis of the adhesive interface: Two extra teeth from each subgroup were selected, prepared, restored, and grouped as mentioned in the microtensile test (n=2) for evaluation of hybrid layer ultra-morphology. The teeth were sectioned bucco-lingually into two halves along the long axis of the teeth in a direction perpendicular to the resin-dentin interface using a water-cooled diamond disc at low speed (IsoMetTM 4000, Buehler Ltd; Lake Bluff, IL, USA). Each half was polished with coarse (600 grit), medium (800 grit), fine (1000 grit), and extra fine (1200grit, 2000grit, 2500grit) silicon carbide papers (SIA Brand Switzerland). Final polish was obtained with fine diamond pastes with particle size (3μm, 1μm, 0.5μm) respectively (ENA polishing system, Micerium S.p.A.) with a polishing brush (ENA hri polishing brushes, Micerium S.p.A.). Then, the samples were cleaned in an ultrasonic bath for 10 min (XH-E412 ultrasonic cleaner, Xinghua, China). After that, the specimens were exposed to a 10% orthophosphoric acid solution for 5 sec. to demineralize dentin collagen fibers and then to a 5% sodium hypochlorite solution for 5 min to remove organic components. This technique demineralizes all dentin that was not infiltrated with resin so that the specimen surface could be dehydrated and exposed to RT for imaging.

Results:

Micro-tensile bond strength (μTBs)

a. Immediate subgroups: Regarding the data obtained from the μTBS of all groups in this study, Shapiro-Wilk test showed a normal distribution pattern of all values (p>0.05). A descriptive statistic was calculated in the form of mean and standard deviation (SD). Then, the significance of the difference between groups was tested using One-way ANOVA to compare the groups. The Largest Significant Difference (LSD) Post-hoc test was used to detect the difference between groups and showed that All-bond universal adhesive had the highest mean bond strength, which was significantly different from Tetric N bond UA and Futurabond M+ UA. Also, Tetric N bond UA showed the lowest mean bond strength values. One-way ANOVA showed a statistically significant difference among all tested groups (p=0.002). Tukey post-hoc test was used for multiple comparisons and showed that there was a significant difference between the All-bond universal adhesive system and the Tetric N bond. Also, there were significant differences between; the All-bond universal adhesive system and Futurabond M+ universal group, and the Tetric N bond group and Futurabond M+ group as presented, in Table.

b. Delayed subgroups: Similarly, the One Way ANOVA test was used to compare among studied groups and showed no statistically significant difference among the studied groups (P=0.053). The Largest Significant Difference (LSD) Post-hoc test was used to detect the difference between groups and showed that All-bond universal adhesive had the highest mean bond strength and Tetric N bond UA showed the lowest Mean bond strength values as shown in Table 1.

Micro-morphological observation of adhesive/dentin interface under SEM: All groups' SEM micrographs of the adhesive/dentin interface were examined at magnifications of X 500. In Group 1, resin tags were very distinct, with a long, thick, anastomosed funnel-shaped configuration penetrating the dentin substrate with no sign of separation through the interface, and thick HL was also observed (Figure1).

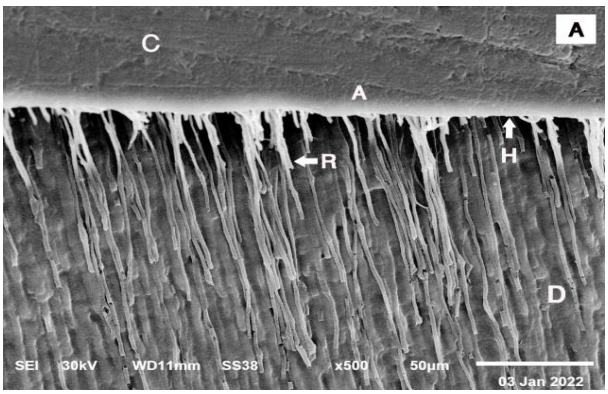

Figure1: Group1; Representative SEM micrographs of the resin-dentin interface of specimens bonded with All-bond universal using the SE technique at magnification X500 (A) adhesive, (R) resin tag, (C) resin composite, (H) hybrid layer, (D) dentin.

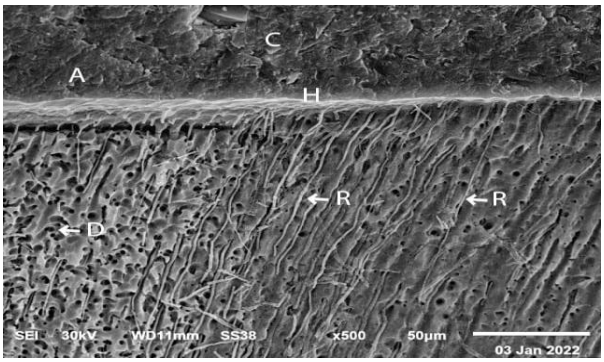

Figure2:Group2; Representative SEM micrographs of the resin-dentin interface of specimens bonded with Tetric N-bond universal using the SE technique at magnification X500. (A) adhesive, (R) resin tag, (C) resin composite, (H) hybrid layer, (D) dentin

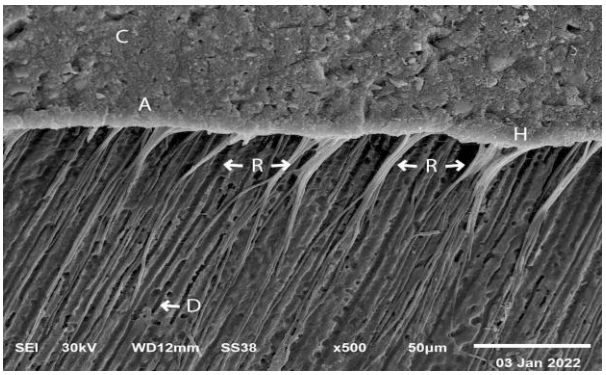

Figure3: Group 3; Representative SEM micrographs of the resin-dentin interface of specimens bonded with futurabond M$^+$ universal adhesive using the SE technique at magnification X500

Regarding restorative system in Group 2; showed thick and long RTs and packets-shaped infiltrations dentin substrate, SL and smear plugs appeared, and open dentinal tubules appeared due to the cut section (Figure 2). Regarding restorative system in Group 3; exhibited few thin and fracture RTs (Figure 3).

Discussion:

Achieving a dependable, strong bond between resin composite systems and tooth structure is critical to the longevity of resin composite restorations. Manufacturers have reduced the number of steps required for bonding application to simplify the bonding procedure, reduce technique sensitivity, and shorten clinical procedure time.[11] In this regard, self-etching adhesives were introduced to the market, which used the self-etching monomer to dentin. After that, the universal adhesives were introduced to make self-etching adhesives, and used with different adhesion strategies, depending on the operator's preference; 'self-etching,' 'etch-and-rinse,' and 'selective enamel etching'.[12] The results of this study showed, that Core-flo DC resin composite and All-Bond universal adhesive- restorative system had the significant highest bond strength immediately and after aging. This may be due to lower acidity (pH 3.1) which is regarded as an ultra-mild etching type. Ultra-mild adhesives superficially preserve hydroxyapatite sound in the interaction zone, with many calcium ions available for a strong chemical bond with MDP functional monomers.[13,14] According to the adhesion-decalcification concept, the MDP-Ca salt complex is highly insoluble, and stable, and forms strong molecular bonds to hydroxyapatite-based substrates.[15] Some of these bonds are stable, even in an aqueous environment, so that the interface can better withstand the hydrolytic breakdown of its components. This mechanism is supposed to prolong the clinical lifetime of restorations.[16] Additionally, this adhesive is applied in double coats, which may contribute to increasing bond strength by enhancing the smear layer dissolving and boosting the functional monomers (10 MDP) in direct contact with dentin, allowing for more chemical interactions.[17] This result was in agreement with Gutierrez et al.,[18] who reported that the All-bond universal adhesive had the highest bond strength due to the low hydrophilicity of All-bond universal adhesive was responsible for this high bond strength. The low concentration of HEMA within the adhesive may improve the chemical bonding of the adhesive. For Muli-core flow DCRC and Tetric N bond universal adhesive- restorative system results; it showed significantly lowest tensile bond strength immediately and after aging. This could be explained due to higher acidity (pH 2.5) that demineralizes dentin HA decreasing the chance for chemical bonding.[19,20] Moreover, Tetric N-bond contains a high amount of bisphenol A diglycidyl methacrylate (Bis-GMA 25-50%) that is a highly viscous monomer that may limit resin monomers penetration into moist dentinal tubules and intertubular dentin.[21] The result of this study was in agreement with Jain et al. [22] who reported that the Tetric N-Bond had low bond strength in self-etch mode. They concluded that the reduced bond strength could be due to the phase separation of the hydrophilic (HEMA) and hydrophobicity which further opens up the polymer network, resulting in the micro void formation and increased free water uptake. On other hand, the result was in disagreement with Cardoso et al.[23] who reported that the Tetric N bond had high bond strength. This was applied in the etch-and-rinse mode. Another study by Sezinando et al.[24] reported that Tetric N-bond universal adhesive had high bond strength as it was applied in self-etch mode. Based on the results of the Repilda DCRC- Futurabond M$^+$ restorative system, there was showed a significant high bond strength compared to Tetric N-bond UA systems immediately and after aging it could be attributed to its acidity (pH 2) is regarded as a mild etching type which partially demineralizes dentine, leaving a substantial amount of hydroxyapatite crystals around the collagen fibrils.[7] In Addition, this adhesive is mixed with a dual cure activator, which may contribute to promoting the mechanical properties of the adhesive. Under acidic conditions, the catalyst reacts with the amines of the dual-polymerized composite, making them more reactive to the benzoyl peroxide activator (BPO), which is responsible for the chemical polymerization

process in resinous materials.[25]According to SEM Observations in the All-bond universal adhesive Produced a thick HL with little variable length of RT with the higher bond strength. Its acidity causes the dissolution of smear plugs and opening of tubular permeability and facilitates penetration, impregnation, polymerization, and entanglement of monomers with the underlying dentin to form hybridized complexes. Regarding HL of the Tetric N bond universal adhesive system had a thin, long, and numerous RT, but it was the weakest bond strength of the three groups in this study. Regarding HL, the Futurabond universal adhesive had thin, short RTs with relatively good bond strength. The thin HL formation may be explained by the inability of the adhesive acidity to dissolve the smear plugs within the tubules and is based on dentin surface roughness.

Conclusions:

Within the limitations and according to the results of the current study, the following conclusions can be drawn:

1- There are significant differences in micro-tensile bond strength among the three types of universal adhesives. However, there was an increase in bond strength value in All-bond universal adhesive compared with the other types in both periods of testing.

2- There was no relation between the micro-tensile bond strength and the micro-morphological patterns in all tested adhesives.

3- Aging affects negatively the bond strength between all groups.

4- When the futurabond universal adhesive was used in conjunction with dual-cured resin composite, the use of the dual-cure activator improved bond strength.

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