

THE IMPACT OF APPLICATION MODE OF THREE UNIVERSAL ADHESIVES WITH DIFFERENT CHEMICAL COMPOSITIONS AND CURING TECHNIQUES ON THEIR MICRO SHEAR BOND STRENGTH TO DENTIN

Mohamed Amr Kamel^{*ID}, Mennatallah A. Aziz Meheba^{**ID}
and Zainab M. Diaan El-Din Soliman^{***ID}

ABSTRACT

Statement of problem: Different types of universal adhesives with different curing techniques are present in the market. The effect of their application modes on their performance is still unknown. Objectives: To test three universal adhesives with different curing techniques and their bonding ability to dentin when applied in different application modes.

Methods: Self cure Palfique Universal Bond (PUB), light cure One Coat 7 Universal Bond (1C) with and without One Coat 7.0 Activator (+/- SCA) and light cure All-bond universal (ABU) and Filtek supreme flowable composite were used. The three adhesives were used in etch-and-rinse (ER) and self-etch (SE) mode to bovine dentin. Microshear bond strength (SBS) was tested. For each group 3 teeth were prepared with 15 Microshear bond specimens. Data was collected and analyzed using R statistical analysis software version 4.1.3 for Windows.

Results: Among groups, 1C showed the highest SBS values irrespective to application mode while PUB showed lower SBS in both modes compared to other used adhesives. In the ER mode only, PUB showed significantly lower SBS values compared to other tested adhesives, no significant differences were present between the 1C +/- SCA and ABU. In SE, both 1C + SCA and PBU presented significantly lower SBS compared with other groups, while 1C showed significant higher SBS.

Conclusions: All adhesives performed better in ER mode compared to SE mode except for PUB. Significance: ER mode did not negatively affect the bonding performance to dentin and the variation of results in both application modes was adhesive dependent.

KEYWORDS: Universal adhesives, curing techniques, application modes

* Assistant Professor, Operative Dentistry, Faculty of Dentistry, Ain Shams University, Cairo, Egypt

** Instructor, Operative Dentistry, Faculty of Dentistry, Ain Shams University, Cairo, Egypt

*** Assistant Professor, Operative Dentistry Department, Faculty of Dentistry, Ain Shams University, Cairo, Egypt

INTRODUCTION

Since the introduction of resin-based restorations, their longevity was based on the bond strength and durability of the adhesive joint. Hence, manufacturers kept on developing different adhesive systems that aimed to increase bond strength with enamel and dentin as well as decrease technique sensitivity and clinical steps. In the effort to do so, universal adhesives also known as multimode adhesives were introduced in the field of dentistry. Since then, this category of adhesives has become increasingly trendy due to the ability to be applied in self-etch (SE) as well as etch-and-rinse (ER) mode¹, in addition to their reduced number of steps which can be variable according to mode of application² and compatibility with different substrates. Decreased sensitivity to different degrees of moisture in enamel and dentin was also reported which even made them more popular and user friendly³.

Debates were reported regarding the multi-mode use of universal adhesives, and though universal adhesives' use became common due to their simplicity and flexibility which enables clinicians to decide according to cavity design the most appropriate application protocol, the bond durability of many products were inferior to the performance of two-step self-etch adhesives especially with enamel tissue, regardless of application mode^{4,5}.

Single Bond Universal was the first universal adhesive introduced in the market. The adhesive formula included 10-MDP monomer. This monomer has the advantage of bonding chemically with tooth forming hydroxy appetite crystals, thus improving the bond durability⁶. Universal adhesive formulas kept on improving to widen their use with various material including resin luting cements⁷, in addition to various substrates with no need for additional step of surface treatment^{8,9}

A main advantage of universal adhesive is their compatibility with indirect restorations due

to their silane content. Upon indirect restorations cementation, hindered light penetration was reported due to increased thickness of restorations in some areas, or decreased light penetration due to high opacity of some ceramics. Consequently, low degree of convergence of adhesive in such areas accompanied by lesser bond strength values were stated. In addition, incompatibility of light activated universal adhesive with chemical and dual cured core build up materials was also reported^{10,11}. Hence, the idea of dual and chemical cured universal adhesives was presented. The presence of such adhesives helped to solve the problem of incompatibility between the light activated adhesives and the chemical and dual cured resin materials.

Though chemical and dual cured adhesives helped to solve many problematic clinical situations, their performance regarding bond strength values was not properly studied. Hence, this study was carried to compare Microshear bond strength (mSBS) values of chemical cured and dual cured universal adhesives with the light cured one in both ER and SE modes in the aim to reach a conclusion for the best application strategy for each of the studied adhesives. The following null hypotheses were tested (1) the curing techniques have no influence on the bond strength of resin composite to dentin and (2) the application modes have no effect on bond strength to dentin.

MATERIALS AND METHODS

Materials

A flowable resin composite, and three types of adhesive materials were used in the study. Materials composition is listed in table 1

Methods

A total of 24 anterior bovine teeth were freshly extracted and used in this study. After extraction, teeth were thoroughly washed under tap water to remove soft tissue debris and stored in 0.1% thymol

TABLE (1): Material, composition, description , pH and lot number

Material/Manufacturer (Lot #)	Description	Composition
Filtek supreme flowable restorative (3M ESPE St.Paul, MN) Lot #: NC91529	Flowable resin composite	Silane Treated Ceramic, Substituted Dimethacrylate, BISGMA, TEGDMA, N-DIMETHYLBENZOCAINE, Diphenyliodonium Hexafluorophosphate.
Palfique universal bond (PUB) (Tokuyama Dental Copr., Japan) Lot #: 118E00	Self-etch adhesive (pH= 2.2)	Bond A: Phosphoric acid monomer, Bis-GMA, HEMA, TEGDMA MTU-6, Acetone (solvent). Bond B: γ -MPTES, Borate, Peroxide, Isopropyl alcohol, Acetone and Water.
One coat 7 universal (1C) (Colten,Whaledent, AG, Switzerland) Lot #: J33228	Self-etch adhesive (pH=2.8)	10-MDP, methacrylates, photoinitiators, methacrylated polyacrylic acid, water and ethanol
One Coat 7.0 activator (SCA) (Colten,Whaledent, AG, Switzerland) Lot #: J67279	Chemical Activator	Ethanol, water, activator
All-bond universal (ABU) (Bisco,Inc, Schaumburg, Illinois, USA) Lot #: 2000007847	Self-etch adhesive (pH= 3.2)	Bis-GMA, HEMA, MDP, initiators water, ethanol,
Ultra-etch phosphoric acid gel (Ultradent products Inc., South Jordan, Utah, USA)	Acid etch	35% phosphoric acid

for no longer than one month. Teeth were divided into four main groups according to the adhesive used (PUB, 1C, 1C +SCA and ABU) with six teeth for each group. Each group was subdivided into two subgroups (three teeth) according to the application mode used (ER, SE). Teeth were then sectioned longitudinally in a mesio-distal direction to obtain three dentin slices from each tooth. Each slice obtained five polyethylene tubes to have a total number of 15 microshear bond specimens per group (n=15).

The incisal half of each tooth was eliminated, the labial surface of the cervical half was ground under water using #180 SiC paper to expose an area of nearly 10 mm diameter of flat dentin. The roots were cut off at the cervical line and the pulp was extirpated using H-File. Pulp chambers were

cleaned and blocked using sterile cotton pieces to prevent the penetration of acrylic resin material.

A three-quarter inch in diameter Polyvinyl chloride rings (PVC rings) of 1cm height was fixed over a glass slab using double faced adhesive. With the labial dentin facing downwards, the tooth was placed in the center of the tube. A mix of cold cure acrylic resin (Acrostone, Egypt) was poured inside the tube till it became flushed with the upper rim of the tube. To decrease polymerization heat, the whole assembly was placed in tap water. After setting, each specimen was wet ground over #180 SiC paper to remove excess acrylic resin material, if presented. Wet grounding using #600 SiC for 30 seconds was done to create a smear layer.

In each adhesive strategy, bonding procedure was implemented following the manufacturer's

instructions. For the ER groups, the bonding procedure was preceded with dentin etching for 15 seconds using 37% phosphoric acid etchant (Ultradent) followed by rinsing for 15 seconds, followed by blot drying using paper pellet.

For PUB in SE mode, mixing a drop from bond A and a drop from bond B was done, followed by adhesive was application on the dentin surface and gentle dryness with oil-free compressed air till no movement of adhesive was visible. For (1C) in SE mode, the bottle was shaken well, one drop of the adhesives was dispensed, applied, and rubbed to the dentin surface for 20 sec using micro brush. Gentle air dryness for 5 seconds followed. For (1C +SCA) specimens, a drop of One Coat 7.0 activator and a drop One Coat 7 universal bond were mixed into the mixing well before application to the dentine surface followed by the same protocol steps previously mentioned for (1C). For ABU specimens in SE mode, two separate coats of adhesives were scrubbed to the surface using micro brush for 10-15 sec each. The coats were applied successively with no light curing between them. Solvent evaporation followed by drying with oil free compressed air for at least 10 sec and until no visible adhesive movement.

Before adhesive curing, a polyethylene tube of internal diameter 0.9 mm and 1mm height was located over the uncured adhesive then light curing was performed, except for PUB, the adhesive was left to cure with no light curing. Light curing was performed using LED curing unit (Elipar, 3M ESPE, USA) 1200 mW/cm².

Flowable composite was injected inside each tube, covered with a polyester strip and pressed using a glass slide for five seconds so that any excess material was extruded. The glass slide was removed before curing was done. The resin composite was then light cured for ten seconds according to the manufacturer's instruction.

Each acrylic block with the specimen fixed to

it, was mounted to the Universal Testing Machine (Lloyd instruments LR5, Leicester, UK). Testing for mSBS was done by using 0.2 mm orthodontic ligature wire applied around each specimen as close as possible to resin/dentin interface. Testing was run with crosshead speed of 0.5mm/min. until failure. Shear bond strength in Mega Pascal was calculated. $SBS = \text{load (Newton)} / \text{bonded area (mm}^2\text{)}$.

Data was represented as mean and standard deviation (SD) values. Shapiro-Wilk's test was used to test for normality, followed by Levene's test to test for Homogeneity of variances. Due to parametric data distribution and variance homogeneity, two-way ANOVA followed by Tukey's post hoc test were performed. Comparison of simple main effects was done utilizing the error term of the two-way model with p-values adjustment using Bonferroni correction. The level of significance was set at $p < 0.05$. Statistical analysis was performed with R statistical analysis software version 4.1.3 for Windows.

RESULTS

Results of two-way ANOVA presented in table (2), showed no significant effect for the adhesive strategy on microshear bond strength with p -value 0.253. On the other hand, the curing mode and the interaction between adhesive strategy and curing mode had significant effect on mSBS with p -value of < 0.001 and 0.002 respectively.

Comparison of simple main effects presented in table (3) showed that for samples treated with ER protocol, there was significant differences between different adhesives with (1C), (1C+SCA) and ABU having significantly higher values than PUB ($p < 0.05$). For samples subjected to SE protocol, there was also a significant difference with (1C) having significantly higher value than (1C +SCA) and PUB ($p < 0.05$). The ABU Universal adhesive showed nonsignificant results with all groups in SE mode.

For PUB, samples treated with SE protocol had significantly higher values than ER samples ($p < 0.05$), while for (1C +SCA) samples treated with ER had significantly higher values than SE samples ($p < 0.05$).

For other adhesives, no significant difference between different strategies was present ($p > 0.05$). Mean and standard deviation values for μ SBS in different groups were presented in figures (1) and (2).

TABLE (2): Two-way ANOVA for the effect of different adhesion strategy, curing mode and their interactions on μ SBS

Parameter	Sum of squares	df	Mean square	f-value	p-value
Adhesive strategy	27.12	1	27.12	1.35	0.253
Curing mode	1505.39	3	501.8	24.9	<0.001*
Adhesive strategy * Curing mode	358.39	3	119.46	5.93	0.002*
Error	805.97	40	20.15		

* statistically significant difference with significance level of ($p < 0.05$).

TABLE (3): Means \pm Standard Deviations regarding the effect of different adhesion strategies for adhesives under study on the μ SBS (MPa)

Adhesive strategy	Micro-shear bond strength (MPa) (Mean \pm SD)				p-value
	Palfique (PUB)	One coat 7 (1C)	One coat 7.0/DC activator (1C + SCA)	All-bond (ABU)	
ER	5.61 \pm 0.73 ^B	23.87 \pm 2.40 ^A	22.33 \pm 7.60 ^A	22.36 \pm 5.23 ^A	<0.001*
SE	11.99 \pm 1.46 ^B	24.28 \pm 5.53 ^A	14.24 \pm 4.88 ^B	17.64 \pm 3.64 ^{AB}	<0.001*
p-value	0.018*	0.874	0.003*	0.076	

Different capital letters within the row indicate statistically significant difference ($p < 0.05$). * Below each column represents significance within the same column ($p < 0.05$)

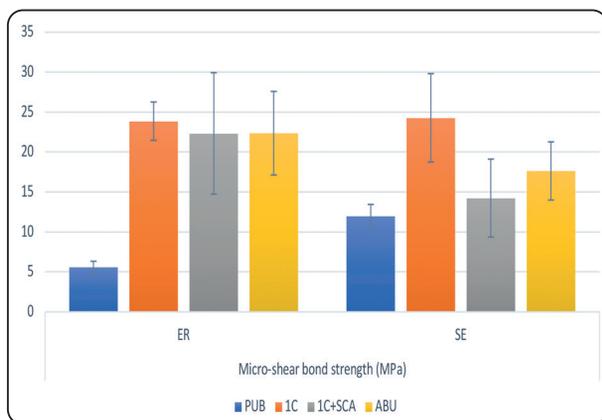


Fig. (1) Bar chart showing mean and standard deviation values of μ SBS (MPa) values for different adhesive strategies

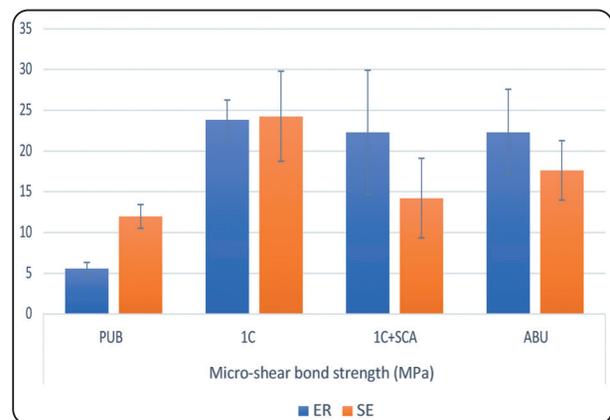


Fig. (2) Bar chart showing mean and standard deviation values of μ SBS (MPa) values for different adhesives

DISCUSSION

Upon the launch of universal adhesives (multi-mode adhesives), their use expanded due to manufacturers' claim that these adhesives can be used in SE as well as ER mode in addition to their ability to be used in variety of clinical situations including direct as well as indirect restorative approaches¹². The application mode depends on the substrate to be bonded where etch and rinse mode is mostly preferred upon bonding to enamel due to smear layer removal¹³. For dentin, ER mode was debatable due to the exposure of collagen network and increased permeability to flow of fluid, exposing the collagen network to hydrolytic and enzymatic degradation which could have detrimental effect on bond durability¹⁴.

Upon considering the two strategies for using multimode adhesives, bonding to dentin can be performed either by ER or SE mode, many studies were performed to evaluate the bonding efficacy of multimode adhesive in both modes, the results were quiet contradictory. Bond strength produced using universal adhesives upon application to dentin in ER mode was reported to be higher than SE mode which was consistent with the results of the present study for all adhesive types and groups used in the present study except for PUB^{15,16}. Authors attributed such results to the possibility of proper dentin hybridization due to elimination of smear layer, presence of patent tubules and establishment of thick and properly formed hybrid layers and long resin tags, both of which were responsible for enhanced micromechanical interlocking reflected as higher bond strength values^{17,18,19}. Using universal adhesive in SE protocol resulted in the establishment of thin hybrid layer in addition to the partial demineralization of collagen matrix, which was reflected by thinner hybrid layer, shorter and less dense resin tags and over all less monomer penetration when viewed by SEM^{20,21}. On the other hand, other studies had contradictory

results and suggested that the use of ER protocol showed no improvement in bond strength of multi-mode adhesive, since both hybrid layer thickness and resin tags length have no direct correlation with bond strength^{22,23}. When the performance of universal adhesives on dentin substrate in SE mode was compared to two step SE adhesives, the later was reported to have higher bond durability²⁴. This contradictory information concerning the performance of multi-mode adhesives while using different bonding protocols particularly with newer brands and materials categories was reported to be mainly adhesive dependent and no agreement is present on specific application protocol²⁵.

Most current adhesives polymerization is based on photoinitiation and production of free radicals²⁶. Though photoinitiation provides the clinician with complete control over the initiation step, it proved to be problematic in areas where light delivery was compromised such as in endodontic preparations and deep cavity preparations²⁷. Hence, dual cured adhesive systems were developed by adding chemical initiations as organic peroxide and aryl sulfinate salt activators to be mixed with the adhesive just before application which allowed the adhesive system to cure in the absence of light²⁸. Still, the dual cured adhesive polymerization starts basically by light activation and should continue chemically by the self-cured activators. In the areas where light proved to be deficient, radical generation was spontaneous and non-controllable which was reflected by lower bond strength values²⁹⁻³⁰. To counteract this major drawback, self-cured adhesives were developed which relayed totally on chemical polymerization through the presence of benzoyl peroxide as chemical initiator and tertiary amine as co-initiator³¹. Furthermore, many studies reported the incompatibility between dual and self-cured resin composites with simplified adhesives^{32,33}. Both of which resulted in reduced bond strength values, post-operative sensitivity and microleakage. It was reported that the the pH of the

adhesive plays a major role in the incompatibility issues with dual cure resin composite, where no incompatibility issues were reported with ultra-mild adhesives (pH >3) ³⁴.

The first null hypothesis was rejected as the curing mode showed significant effect ($p < 0.001$) on the bond strength to dentin. According to manufacturer, the chemical composition of the activator of (1C) adhesive contains ethanol and water in addition to the activator³⁵, it was stated that upon mixing the activator with the adhesive, a change in solvent/water ratio is to be expected presented as monomer dilution and disruption in components proportion of the adhesive. High solvent/water content could have a detrimental effect on solvent evaporation and adhesive polymerization, which is by default reflected on adhesive performance and bond strength results³⁵. This could explain the significance difference in bond strength with or without activator when the (1C) was applied in SE mode ³¹. Conversely, the use of (1C) without the addition of activator produced the highest bond strength values in all groups. This could be attributed to the fact that upon using the (1C) in ER mode, removal of the smear layer and proper hybridization within dentin occurred presented by high bond strength value as mentioned earlier ¹⁷⁻¹⁹. On the other hand, using (1C) universal in SE mode presented similar bond strength values due to the presence of MDP monomer as stated by manufacturer, which can chemically bond to hydroxy apatite crystals present within dentin and produces a stable and strong bond reflected by the significantly highest bond strength values within self-etch mode of all groups within this study ⁶. Though ABU contains MDP monomer, the difference between the performance of the adhesives in SE mode could be attributed to the difference in their pH. The ABU is an ultra-mild adhesive with a pH= 3.2, which could result in lesser degree of demineralization depth, hence being insufficient for

effective etching for the dentin surface hence, the monomers infiltration will be hampered³⁶.

The second null hypothesis was partially rejected as the mode of application had a significant effect on the bond strength of both PUB and 1C+SCA ($p=0.018$ and 0.003 respectively). Regarding the PUB, results in SE mode which were comparable to other adhesives except for (1C), this could be attributed to scarcer resin tags formed in the hybrid layer, as reported by Campos et al. in their study, where they found that the PUB produced lower bond strength than the other tested SE adhesives used (Clearfil SE, Optibond All-in-One), though the resin tags of the PUB had deeper penetration than the other tested groups³⁷. Regarding this, it could be assumed that deeper resin tags penetration associated with a pre-etching step in the ER mode as well as having a lower pH (pH=2.2) than the other groups, could have produced very deep etching, that in turn would have affected the easiness of resin infiltration, and solvent evaporation. Hence, affecting in the quality of the final hybrid layer, reflecting even lower bond strength than that reflected in the self etch mode^{38,39}.

CONCLUSION

Under the limitation of this study, the following conclusions could be suggested:

1. Regarding the curing modes of universal adhesives, light cured adhesives performed better than other modes when bonding to light cured resin composite.
2. Considering the mode of application, universal adhesives showed better performance with etch and rinse mode except for self cured adhesives.

ACKNOWLEDGMENT



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