

Comparative Evaluation of the Effect of Different Universal Adhesives and Bonding Techniques on the Marginal Gap of Class I Composite Restoration (A SEM Study)

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ABSTRACT

Background: With the increase in composite material use in posterior teeth, the concerns about the polymerization shrinkage has increased with the concerns about the formation of marginal gaps in the oral cavity environment. New generation of adhesives called universal adhesive have been introduced to the market in order to reduce the technique sensitive bonding procedures to give the advantage of using the bonding system in any etching protocol without compromising the bonding strength. The aim of the study was to study marginal adaptation of two universal adhesives (Single bond™ Universal and Prime and Bond elect) using 3 etching techniques under thermal cycling aging.

Materials and Methods: Forty-eight sound maxillary first premolar teeth were included in the study. Teeth were divided into two groups according to the universal adhesive used then each group was subdivided into 3 subgroups according to the etching protocol used. Standardized class I cavities were prepared in the teeth followed by the restoration of teeth using Filtek™ Bulk Fill Posterior Restorative composite material. After finishing and polishing, teeth were subjected to 500 thermal cycles in 55°-5°C bath with dwell time of 30 seconds. Teeth then were examined using SEM to measure the marginal gap at 12 points. Data obtained were analyzed using one-way ANOVA and LSD test for each group and with student t-test to compare the two adhesives.

Results: The result of this study the showed that etch and rinse technique showed significantly the least marginal gap width for both adhesive types. The selective etch technique showed lower gaps compared to the self-etch technique with no significant difference. The result showed that single bond universal showed significantly the least marginal gap for the all etching techniques compared to Prime and bond elect.

Conclusion: The etch and rinse technique remains the most suitable technique for adhesive restoration. The type of adhesive plays an important role in adhesion.

Key words: Marginal adaptation, universal adhesives, etching technique. (*J Bagh Coll Dentistry* 2016; 28(4):34-42)

INTRODUCTION

The increased demand for the replacement of natural dentition in the posterior region of the mouth with aesthetic restorations has resulted in increased usage of resin-based composite materials worldwide. The increase in usage caused the materials and techniques to continue developing to reduce the time of placement of the restoration as well as creating easier techniques ⁽¹⁾.

Despite many new and innovative developments in the field of resin-based composite materials, a 100% perfect margin is not realistically achievable. Composite materials undergo volumetric polymerization contraction of at least 2%, which may result in gap formation as the composite pulls away from cavity margins during polymerization. Adhesive's ability to seal a cavity preparation can be influenced by its composition, flow, penetration into dentinal tubules, coefficient of thermal expansion, modulus of elasticity and the mechanical stresses caused by cavity shape.

Therefore, a tight marginal seal still has to be the primary goal for the clinician, because once happened; gap formation cannot be counteracted with restorative materials. In addition to stress shrinkage, the occlusal loads and alterations of the temperature of the oral behavior produce stress on the restoration and can also compromise the marginal sealing ^(1,2).

Another disadvantage of resin-based composite restoratives include the increased technique sensitivity and time required to adequately place restorations, which can be up to two and a half times longer when compared with nominally identical dental amalgam restorations. The acid-etch, wash/dry and light irradiation component steps were reported to account for 86% of the increased time required for resin-based composite restoration placement ⁽³⁾.

In an attempt to decrease resin bonded composite placement times, etch and rinse adhesive bonding systems which include a separate etch with acid and rinse step, a priming step followed by the application of the adhesive resin have been simplified by dental adhesive manufacturers. Two-step etch and rinse adhesives were developed and today self-etch adhesives that eliminate the rinsing phase have been advocated

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to significantly reduce resin bonded composite placement time⁽⁴⁾.

Continuous improvements in the self-etch adhesives by better chemical composition resulted in increased adhesion to dentine, however, this improvement remained unsatisfactory in enamel. Therefore, the selective etching procedure for the enamel was recommended specially for mild self-etch adhesives. On contrary, this selective etching was reported to have an adverse effect on the bonding to dentine because it is difficult to perform etching to enamel without accidental etching of dentine⁽⁵⁾.

More recently, for the aim of reducing complications of current self-etch adhesives a new family of bonding agents known as universal or multi-mode adhesives have been introduced into the dental market and are essentially one-step self-etch adhesives that can be employed with or without a separate etching step⁽⁶⁾. The key for the success of this new generation is the chemical bonding ability of the functional monomer to hydroxyapatite and not depending on the hybrid layer⁽⁷⁾. However, it was reported that longer resin tags and thicker hybrid layer that results from acid etching may improve the bond strength of universal adhesives, a clear correlation to higher bond strengths could not be established⁽⁶⁾.

The high quality of modern composite materials has made it more difficult to see changes in the quality of restoration margins, which in turn, has increased the need for more sensitive methods to assess the early changes of the marginal adaptation. Scanning electron microscopy (SEM) is a method that can be used for closer examination of the restoration margins because of its ability to magnify and reveal details⁽⁸⁾.

MATERIALS AND METHODS

Teeth selection

Forty-eight teeth were included in the study. The criteria depended on selecting teeth with comparable size. Therefore, the dimensions of all collected teeth were measured from bucco-palatal and mesio-distal distance using a point Vernier caliper. The selected 48 teeth were assigned into 6 groups and distributed in a way that their standard deviation is not exceeding 10% of their means. One-way ANOVA test was performed for each dimension and no statistically significant difference was found among the 6 groups.

All the teeth then were cleaned carefully for any calculus deposits with hand scalar and polished with a rubber cup and slurry of pumice

then rinsed with water to remove the residual debris⁽⁹⁾.

Teeth mounting

A plastic container of 4.6x4.6x3cm dimensions was used to construct the silicon mold of 15x15x20mm dimensions at the center of the container for the construction of 48 acrylic-teeth blocks. A mix of self-cure acrylic resin was prepared in the mold and the tooth is inserted slowly into the center of acrylic and locked in this position for 10 minutes to give time for the acrylic to set in order to separate the rod from the tooth (Fig. 1).



Figure 1: Tooth with 8mm length showing above the acrylic

Cavity preparation

Standardized class I cavities with butt joint cavo-surface margins was prepared according to the cavity design as follows (Fig. 2):

1. Bucco-palatal width 3mm.
2. Mesio-distal width 4mm.
3. Occlusal depth 2mm from the center of the fossa.

A modified dental surveyor with a modified High speed hand piece where used to perform the preparation.

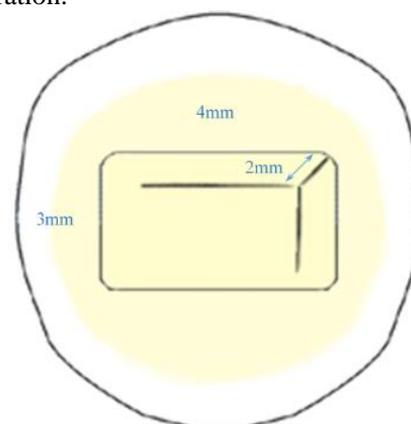


Figure 2: Cavity design

Samples grouping

Teeth were divided into 2 groups according to the type of the adhesive (group A and B) used and each group was subdivided into 3

subgroups of 8 teeth each according to the type of the surface treatment as follows:

In group **A** Single bond™ Universal adhesive (Table 1) where used. In each subgroup different bonding technique were used as follows:

Group A1: teeth in this group were treated with the etch and rinse technique.

Group A2: teeth in this group were treated with the self-etch technique.

Group A3: teeth in this group were treated with the selective etch technique

In group **B** Prime and Bond Elect Universal Adhesive (Table 2) where used. In each subgroup different surface treatment were used as follows:

Group B1: teeth in this group were treated with the etch and rinse technique

Group B2: teeth in this group were treated with the self-etch technique

Group B3: teeth in this group were treated with the selective etch technique

Bonding procedure

Etch and rinse groups (A1, B1)

The teeth were conditioned with a 36% phosphoric acid for 15 second. After that the adhesive of each group is applied according to the manufacture instructions.

Self-etch groups (A2, B2)

The adhesive of each group is applied according to the manufacture instructions directly without any surface treatment.

Selective etch groups (A3, B3)

The enamel margins of the cavity were conditioned with a 36% phosphoric acid for 15 second making sure no acid etch gel reach or touch the dentine. After that the adhesive of each group is applied according to the manufacture instructions.

Restoration procedure

After completion of the adhesive procedure, each tooth was restored with Filtek™ Bulk Fill

Posterior Restorative composite material using a single increment Finishing and polishing was performed followed by checking of teeth with a stereomicroscope (Altay, Italy) at 20X magnification to ensure that no overhangs of the restoration material remain along the margins of the restorations.

Thermocycling procedure

All samples were subjected to 500 thermal cycles in 5 °C and 55°C water baths with dwell time of 30 seconds according to the ISO TR 11405 (10) in order to simulate the oral cavity environment.

SEM examination

All the samples were examined by Inspect S50 SEM at 2000X magnification under low vacuum to detect marginal gaps along the composite/enamel interfaces at the occlusal regions (Fig. 4). The measurement of marginal gap width (the distance between the enamel wall and the restoration) in each sample were taken at twelve points at the occlusal region (3 points in buccal side, 3 points in palatal side, 3 points at the mesial side and 3 points in distal side) (Fig. 3) (1).

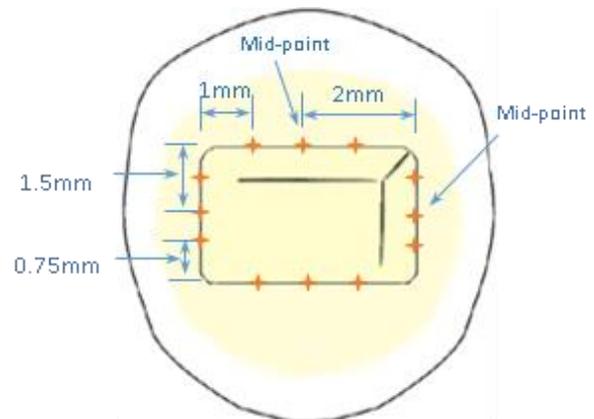
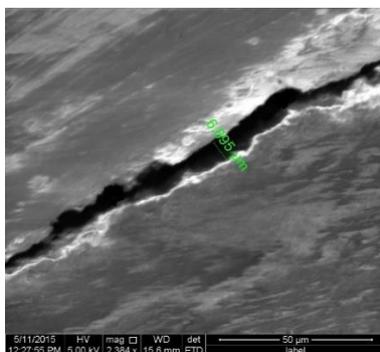
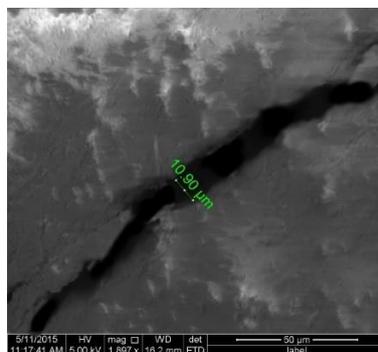


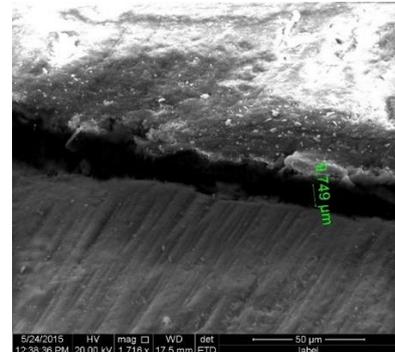
Figure 3: Points location used for the SEM examination



A



B



C

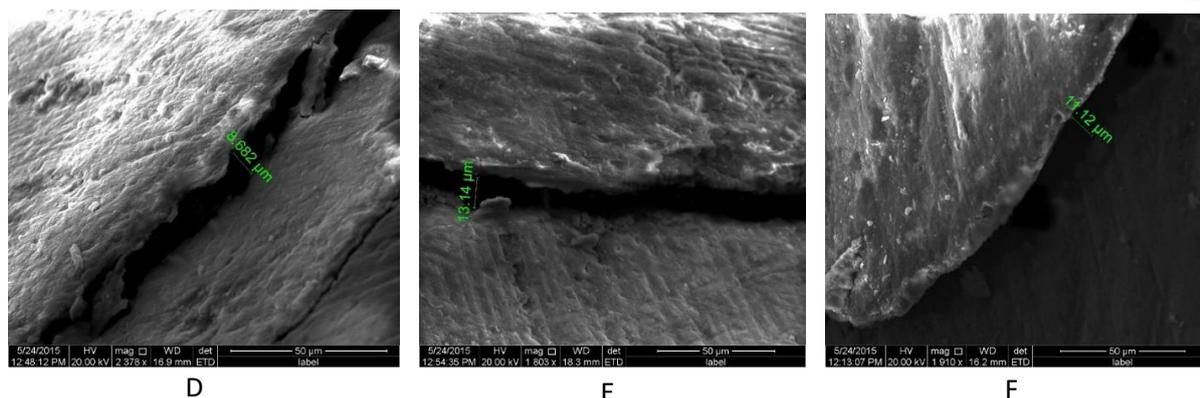


Figure 4: (A) Represent gaps of group A1 (B) Represent gaps of group A2 (C) Represent gaps of group A3 (D) Represent gaps of group B1 (E) Represent gaps of group B2 (F) Represent gaps of group B3

Table 1: Composition of the two universal adhesives bonding used

Materials	Composition
Single Universal Adhesive (SbU)	10-MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond™ Copolymer, Filler, Ethanol, Water, Initiators, Silane.
Prime and Bond Elect Universal Adhesive (PBE)	Mono-, di- and trimethacrylate resins; PENTA (dipentaerythritol penta acrylate monophosphate); Diketone; Organic phosphine oxide; Stabilizers; Cetylamine hydrofluoride; Acetone; Water.

Statistical analyses

The data were collected and analyzed using SPSS (Statistical Package for the Social Science, version 22) for statistical analysis.

RESULTS

A total of 576 enamel/composite gap measurement were examined by SEM and recorded. The largest measurements in each surface were used in the statistics.

Descriptive statistics

The means, standard deviations, minimum and maximum values for each group are summarized in (Table 2). The lowest mean values for the marginal gaps width (μm) was for the group A1, while the highest mean values for the marginal gaps width (μm) was for group B2.

Inferential statistics

Effect of the adhesive technique

One-way ANOVA test showed that there were statistically highly significant differences among the different subgroups for both types of adhesives (Table 3).

LSD test result showed that subgroup A1 produced the least gap which was statistically very highly significant difference compared with subgroups A2 and A3. However, there was no statistically significant difference between subgroup A2 and A3 (Table 4).

The result also showed that subgroup B1 produced the least gap which was statistically very highly significant difference compared with subgroups B2 and statistically significant difference with subgroup B3. LSD test also showed that there was no significant difference in the marginal gaps between subgroups B2 and B3 (Table 4).

Effect of the type of adhesive type

Student t-test was used to examine if there is any significant difference between each subgroup of group A with its corresponding subgroup in group B. The result showed that there was a significant difference in the gap width between Group A1 and B1 and group A3 and B3 and also a highly significant difference in the gap width between Group A2 and B2 (Table 5).

Table 2: Table showing mean, SD, SE, minimum and maximum of the data collected

Material group	Surface treatment group	Descriptive statistics				
		Mean	S.D.	S.E.	Min	Max
Group A (3M ESPE Single Bond Universal Adhesive)	A 1 Etch and rinse group	6.495	1.189	.42	4.69	8.17
	A 2 Self-etch group	10.406	1.867	.66	7.712	13.151
	A3 Selective etch group	9.89	1.72	.608	7.045	11.427
Group B (DENTSPLY Prime and Bond Elect Universal Adhesive)	B 1 Etch and rinse group	8.792	1.208	.427	7.102	10.6
	B 2 Self-etch group	13.995	2.618	.925	9.637	16.751
	B 3 Selective etch group	11.901	2.492	1.101	8.634	14.832

Table 3: ANOVA test for both group A and group B

Group	ANOVA	Sum of Squares	df	Mean Square	F-test	Sig.
A	Between Groups	72.227	2	36.114	13.788	.000 (VHS)
	Within Groups	55.004	21	2.619		
	Total	127.231	23			
B	Between Groups	109.686	2	54.843	11.329	.000 (VHS)
	Within Groups	101.658	21	4.841		
	Total	211.344	23			

Table 4: LSD test between the subgroup of both group A and group B

Groups	Subgroups	Mean Difference	Sig.
Group A	A1	A2	-3.911 .000 (VHS)
		A3	-3.395 .000 (VHS)
	A2	A3	.5157 .531 (NS)
Group B	B1	B2	-5.204 .000 (VHS)
		B3	-3.109 .010 (HS)
	B2	B3	-2.094 .071 (NS)

Table 5: Student t-test to study the effect of the type of adhesive on the marginal gap

Subgroup	t-test	df	Sig.
A1 – B1	-2.836	7	.025 (S)
A2 – B2	-4.981	7	.002 (HS)
A3 – B3	-2.367	7	.05 (S)

DISCUSSION

Type of adhesive technique

In this study, the marginal quality of two types of universal adhesives was investigated under different etching modes with the effect of aging with thermal cycling. The results of this study revealed that etch and rinse technique produced the lowest gap which is significantly lower than self and selective etch techniques for both adhesives.

The superiority of etch and rinse technique could be due to many reasons:

1. In enamel, etching creates micro-retentive porosities that facilitate the micro mechanical interlocking of adhesive and this may be considered the mechanism of etch and rinse bonding technique^(11,12). A more reliable clinical result was obtained with the use of phosphoric acid etching which increase the surface area for micromechanical retention⁽¹¹⁻¹³⁾. In a study done by Alessandro et al⁽¹⁴⁾, they showed that with phosphoric acid pre-etching there is an increase in the surface area in intact and ground enamel

after the application of self-etch adhesives which is lower than that achieved with self-etch adhesive alone. Consequently, the performance of self-etch adhesives is significantly improved with preliminary phosphoric acid etching which is in an agreement with the result of this study where EandR technique performed better with the adhesives used.

Moreover, it was reported from earlier studies that etch and rinse technique creates a micro morphological interaction that extend deeper into enamel⁽¹⁵⁾. When compared with universal and self-etch adhesives, the reduced acidity renders the adhesive to create less and shallower micro-retentive porosities which is due to the fact that they are unable to fully demineralize the mineral phase of enamel and this may compromise the adhesion to enamel⁽¹⁶⁾. To explain this more, the self-etch technique renders the monomer to a shallower inter-crystallite infiltration and also lack the presence of inter-prismatic resin tags resulting in lower micromechanical interlocking with enamel⁽¹⁷⁾. This was supported by many

studies that reported an increase in the micro shear and micro tensile bond strength to enamel of universal adhesives when etching step was employed⁽¹⁸⁾.

In one study the presence of air void along the enamel-composite interface was reported to be a stress raiser which may provoke crack propagation along the interface. In the same study they found that the absence of resin tags in self-etch systems may be responsible for the decreased marginal quality. On the other hand, the presence of resin tags with the etch and rinse technique may provide 3 dimensional grasp along the etched surface. This may act as a crack deflector that may consume the fracture energy and improve the fracture toughness of the interface producing a lower gap⁽¹³⁾.

The result of this study came in agreement with a study that indicates that performing the etching step is still the most reliable technique to get a better bond strength and to have an enamel bond with the highest fatigue resistance. This is reflected on the marginal quality of the enamel/composite interface and since universal adhesive is essentially one step self-etch its ability to etch enamel is limited^(14,19,20). Additional agreement was found in a study done by Nihan et al⁽²¹⁾, were they found that samples treated with etch and rise technique and thermocycled between 1000 and 5000 cycle had the lowest microleakage scores which indicate longer bonding resistance in enamel.

2. In dentin, the etching step before the application of the self-etch adhesive aid in removing the smear layer to make it easier for the adhesive to penetrate and infiltrate the surface morphology and this creates longer resin tags and thicker hybrid layer. this increase in the penetration has been reported for universal adhesive as well where an increase in the infiltration and improvement in the resin tag length and morphology was also reported compared with the self-etch mode alone⁽⁶⁾.

This result was in agreement with one study where its result indicates that prior etching of dentine produce a better impregnated hybrid layer which resulted in increased μ tensile bond strength when compared with only self-etching technique⁽²²⁾.

In one study, when the universal adhesives applied in self-etch mode did no modification of smear layer or penetration into the dentinal tubules and the hybrid layer was very thin or inexistent and was in agreement with others studies conducted with self-etch adhesives. For this reason, decrease in the overall bond strength

may occur and this may reflect on the marginal quality of the restoration^(23,24).

From the result of this study, the mean of gap was lower for the selective etching giving it the advantage when compared with the self-etch for both adhesive systems used, however the difference was not significant.

The superiority of selective etching over the self-etch groups could be related to the additional step of enamel acid etching which might provide a better micro mechanically prepared enamel surface for bonding. Although the result showed that there is no significant difference between the self and selective modes. This could be due to the number of cycles used in this study which is 500 thermal cycle which may be not enough to show a significant difference.

This result came in agreement with one study that showed a non-significant difference in the marginal quality and restoration retention with and without prior etching of the enamel⁽²⁵⁾. Moreover, Peumans et al⁽²⁶⁾ mentioned that there was no significant difference between restoration retention in cervical region with and without selective enamel etching.

It was suggested that enamel pre-etching with phosphoric acid may provide greater bond strength and better sealing ability of the margins⁽²⁵⁾. However, in one clinical trial study, a universal adhesive was evaluated with and without the selective etching step. The result showed there was no different in the retention of the restoration and only a reduced in the marginal quality of the self-etch group after 18 months⁽²⁷⁾.

Clinical studies pointed out that enamel pre-etching resulted in a more durable marginal integrity of restorations bonded with self-etch adhesives. Nevertheless, it is a challenge in clinical situations to use phosphoric acid only on enamel margins, as accidental dentin etching might occur; especially if a low-viscosity etchant is used⁽¹¹⁾.

Type of adhesive material

The result of this study showed that Single Bond Universal produced lower composite-enamel gap compared to Prime and Bond elect Universal adhesive regardless to method used. There is no previous study in literature that measure the two adhesives used in this study (SBU, PBE) regarding marginal gap.

The result of this study came in agreement with a study performed by **Luque-Martinez et al**⁽²⁸⁾ who found an increase in the micro tensile bond strength (μ TBS) of SbU compared to PBE. This increase in the μ TBS of the SbU may suggest a better marginal adaptation with lower

gap. Moreover, the result was also in agreement with **Issis et al** ⁽²⁹⁾ who found also that PBE has the lowest μ TBS compared to SbU in different evaporation times.

This superiority may be explained by the difference in the composition of the two adhesives in term of different monomer, solvent and presence or absence of filler ⁽¹⁴⁾.

The effect of each component can be explained in the following point.

1. The presence of 10-methacryloyloxydecyl dihydrogenphosphate (10-MDP) in the SbU may be one reason why it performs better than the PBE. This functional monomer can form a low soluble calcium salt on the hydroxyapatite surface which creates a durable and more effective bond to dentine ⁽³⁰⁾. 10-MDP not only bonds to the HAp but also self-assemble into nanolayers that have a high hydrophobic feature that helps in protecting the hybrid layer from hydrolytic degradation ⁽⁵⁾. In one study, the author found that there is an increase in the μ TBS bond strength in two universal adhesives that contain 10-MDP monomer which was statistically significant than that observed with adhesive that does not include this monomer ⁽⁶⁾. On the other hand, the PENTA monomer has no data in literature to prove its efficiency and effect on bonding.

The presence of HEMA in SbU may be the reason for the better mechanical properties of this adhesive which resulted in higher μ TBS which reflected on the decreased marginal gap. Since SbU is a HEMA containing adhesive, the solvents can be easier to remove and since the HEMA also functions as a solvent this result in lower solvents (Water, Ethanol) concentration ⁽³¹⁾. This feature also prevents phase separation where the HEMA replace the evaporated solvent and keep the components together ⁽³²⁾. In addition, HEMA is a very hydrophilic monomer that's makes it very effective in wetting the dentine and that is why it is one of the best adhesion improving monomers ⁽³³⁾.

Since PBE is a HEMA free adhesive, the mixture of the hydrophobic and hydrophilic components makes the adhesive susceptible to phase separation which may be the reason for its lower mechanical properties ⁽³¹⁾.

2. The presence of filler in SbU may be one of the reasons that it performed better since the PBE is unfilled adhesive. Two reasons may be responsible for this effect. Adhesive is considered the weak link between composite and tooth structure and since it was traditionally unfilled, the addition of filler was proposed in several studies to fortify and to

enhance the physical properties of adhesives which will lead to increase performance and may lead to a lower marginal gap ⁽³⁴⁾. Moreover, some manufactures add fillers to adhesive to change the viscosity and to achieve a thicker hybrid layer to overcome the problem of insufficient polymerization, due to the air inhibited layer, of overly thinned adhesive after air thinning specially on enamel margins which may lead to marginal discrepancy ⁽³⁵⁾.

3. The difference in the solvent composition between SbU (ethanol/ water) and the PBE (acetone/water) may be the reason for the superiority of SbU.

It was found that ethanol can re-expand the collapsed collagen fibers because of the H-bonding property of the solvent and this may be an enhancing feature for the SbU ⁽³⁵⁾. On the other hand, acetone lack the ability of re-expanding collapsed collagen fibers due to the absence of H-bonding capability which in turn affect the PandB ability to expand the collapsed collagen fibers ⁽³⁶⁾.

Solvents help the infiltration of monomers into the demineralized spaces of dentin and enamel and lower the viscosity of the adhesive ⁽²⁹⁾. Hence acetone has a higher vapor pressure, it can evaporate so quickly after it's been dispensed and this leads to a fast increase in the viscosity of the liquid that may hinder its ability to infiltrate the dentin leading to a lower bonding strength ⁽³⁶⁾.

It was reported if solvents are not removed entirely from the adhesive, it can inhibit the polymerization of the resin monomers leading to a lower bonding strength ⁽²⁹⁾. It was reported that even with the increase of the vapor pressure of acetone where it should evaporate easily ⁽³⁷⁾, the result was in disagreement in one study where the PBE required a longer evaporation time than the recommended by the manufacture and this may be due to the high concentration of the acetone 50 wt%. This led to the presence of residual solvent in the adhesive resin decreasing the bonding strength ⁽³⁸⁾. On the other hand, SbU have a low concentration of ethanol, 10-15 wt%, which in turn evaporated easily according to the manufacture instruction ⁽²⁹⁾.

Under the experimental conditions of this in vitro study, the following conclusions can be drawn:

1. None of the universal adhesives produced a zero gap margins regardless of the etching mode employed.
2. Etch and rinse technique produced the lowest marginal gap compared with other bonding techniques for both types of adhesives.

3. Self-etch technique produced the largest gap for both types of adhesives, although it was not significant compared with selective etch technique.
4. Single bond universal adhesive produced the lowest marginal gap compared with the Prime and Bond elect universal adhesive regardless of the etching technique used.

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