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EVALUATION OF THE EFFECT OF 3 BONDING PROTOCOLS ON THE MICRO-TENSILE BOND STRENGTH OF SELF-ADHESIVE COMPOSITE TO ENAMEL AND DENTIN

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Objectives: To investigate the micro tensile bond strength (μ TBS) of self-adhesive bulk-fill composite (Surefil One) to enamel and dentin bonded without bonding agent, with an additional phosphoric acid etch, and with the use of a bonding agent in etch and rinse mode.

Methods: 90 sound extracted wisdom teeth were used in this study. Surefil One (Dentsply Sirona, Konstanz, Germany), Prime&Bond Universal (Dentsply Sirona, Konstanz, Germany) and Detrey Conditioner 36 Etch Gel (Dentsply Sirona, Konstanz, Germany) were tested. Teeth were divided in 6 groups of 15 teeth, according to tooth substrate (enamel and dentin) and bonding protocols (no bonding agent, bonding with 36% phosphoric acid etch, bonding in etch and rinse mode using a universal adhesive). μ TBS was tested using a universal testing machine and micromorphological observation of the interface was investigated using a magnifier. Data were analyzed using STATA version 15.0. Different bonding protocols were compared using appropriate statistical tests including ANOVA, Kruskal Wallis, Mann-Whitney, and Fisher's exact tests (p < 0.05).

Results: Surefil One bonded on enamel using etch and rinse mode with a universal adhesive yielded the highest μ TBS values compared to control and with phosphoric acid etching. Additional phosphoric acid etching significantly increased μ TBS on enamel. On dentin, etch and rinse mode also recorded the highest μ TBS and there was a significant difference between control group and with additional phosphoric acid etching. Control groups showed the lowers μ TBS at all experimental groups.

Conclusions: Bonding Surefil One in etch and rinse mode with a universal adhesive is recommended on both enamel and dentin.

Keywords: Adhesion, Micro-tensile bond strength, Resin Composite, Self-adhesive.

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Conflicts of interest:

The authors declare no conflicts of interest.

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ÉVALUATION DE L'EFFET DE 3 PROTOCOLES DE COLLAGE SUR LA RÉSISTANCE À LA MICRO-TRACTION D'UN COMPOSITE AUTO-ADHÉSIF À L'ÉMAIL ET À LA DENTINE

Objectifs: Étudier la résistance à la microtraction (µTBS) d'un composite auto-adhésif de remplissage (Surefil One) sur l'émail et la dentine, collé sans agent de liaison, avec un mordançage à l'acide phosphorique et avec un agent de liaison en mode mordançage-rinçage.

Méthodes: 90 dents de sagesse saines extraites ont été utilisées dans cette étude. Surefil One (Dentsply Sirona, Constance, Allemagne), Prime&Bond Universal (Dentsply Sirona, Constance, Allemagne) et Detrey Conditioner 36 Etch Gel (Dentsply Sirona, Constance, Allemagne) ont été testés. Les dents ont été divisées en 6 groupes de 15 dents, selon le substrat dentaire (émail et dentine) et les protocoles de liaison (sans agent de liaison, avec un mordançage à l'acide phosphorique à 36 %, avec un agent de liaison en mode mordançage-rinçage avec un adhésif universel). Le μ TBS a été testé à l'aide d'une machine d'essai universelle et l'observation micromorphologique de l'interface a été réalisée à l'aide d'une loupe. Les données ont été analysées avec STATA version 15.0. Différents protocoles de collage ont été comparés à l'aide de tests statistiques appropriés, notamment l'ANOVA, les tests de Kruskal Wallis, de Mann-Whitney et les tests exacts de Fisher (p < 0,05).

Résultats: Surefil One collé sur l'émail en mode mordançage-rinçage avec un adhésif universel a obtenu les valeurs de μ TBS les plus élevées par rapport au groupe témoin et au mordançage à l'acide phosphorique. Un mordançage supplémentaire à l'acide phosphorique a significativement augmenté les valeurs de μ TBS sur l'émail. Sur la dentine, le mode mordançage-rinçage a également enregistré les valeurs de μ TBS les plus élevées, et une différence significative a été observée entre le groupe témoin et le groupe avec mordançage supplémentaire à l'acide phosphorique. Les groupes témoins ont présenté les valeurs de μ TBS les plus faibles dans tous les groupes expérimentaux. **Conclusions**: Le collage de Surefil One en mode mordançage-rinçage avec un adhésif universel est recommandé sur l'émail et la dentine.

Mots clés: Adhésion, Force de liaison par microtraction, Résine composite, Auto-adhésif.

Introduction

Composite resins have undergone significant evolution since their introduction in dentistry. Modifications have been made to the matrix and fillers of composites with the aim of reducing polymerization shrinkage and enhancing wear resistance. However, polymerization shrinkage remains a prominent concern, leading to stress development between the tooth and the restoration, resulting in adhesive interface failures and micro-gaps [1].

The clinical implications of such stresses are diverse, including hypersensitivity, secondary caries. pulpitis, and enamel microfractures. These consequences compromise the longevity of restorations. To mitigate shrinkage stress, it is recommended that composite resins be placed within the cavity using an oblique layer technique, with increments no thicker than 2mm. This approach minimizes the number of bonded walls, thus reducing the configuration factor (C-Factor) and overall polymerization contraction [2].

Despite its benefits, the incremental insertion method has some drawbacks. These include the potential for air bubble entrapment, bonding failures, contamination between composite increments, and extended clinical procedure time. In response, bulk-fill composite resins have been introduced, enabling placement in increments of 4 to 5mm thickness [3]. Numerous studies have demonstrated satisfactory outcomes for bulk-fill restorations in posterior teeth, comparable to conventional composite resins [4–6].

Recently, self-adhesive bulk-fill composites have been developed, further simplifying the dental restoration placement technique by eliminating the need to prepare dental tissues and apply an adhesive. Its mode of use offers impressive advantages, simplifying the surgical technique.

Nevertheless, specific criteria must be met for these composites to optimally suit the bulk-fill approach. Beyond extended polymerization depth and effective shrinkage management, the composite's bonding strength is vital to ensure restoration durability and favorable clinical performance. Limited literature exists to assess the chemical properties and clinical performance of this material. This study endeavors to bridge this gap by assessing the adhesive properties of self-adhesive bulk-fill composites through micro-tensile testing on enamel and dentin.

Additionally, we evaluated the effect of an additional selective etching of 30s on enamel and 15s on dentin using 36% orthophosphoric acid on the bonding strength of these self-adhesive composites and the effect of applying a total etching bonding protocol on the bonding of this product.

The null hypothesis was that the micro tensile bond strength of the self-adhesive bulk fill composite (Surefil One) on enamel and dentin was not affected by an additional etching using 36% phosphoric acid, or the application of a bonding agent.

Materials and Methods

The materials used in this study, manufacturer, composition and lot number are shown in table 1.

Ninety recently extracted, intact wisdom teeth (n = 90) were selected for this study after the approval of the Ethical Committee of Saint-Joseph University (Beirut, Lebanon; ref.USJ-2022-89). Teeth with caries. abrasions, erosions, abfractions, or dental restorations were excluded. After confirming crown and root integrity, teeth were cleaned and preserved in 0.1% thymol. Thymol was chosen as a storage material to prevent alterations of dentin and enamel [7, 8]. A graphing software program (SigmaPlot 14.0; Systat Software, Inc., Chicago, IL, USA) was used to calculate the sample size.

The teeth were individually fixed to a sectioning block using acrylic resin. Forty-five randomly selected teeth, destined for measuring the bonding strength on dentin, were cut with a diamond saw (Exact Technologies Inc., Kg of Norderstedt, Germany) mounted on a cutting device with distilled water irrigation, perpendicular to the major axis of the tooth, at mid-distance between the cemento-enamel junction and the highest cusp tip, in order to expose a flattened dentin surface of the crown. Then, the dentin surface was polished with a silicon carbide abrasive paper with water, grain size 600 for 30s in order to create a uniform smear layer.

Table 1. Materials used in the study, manufacturer, composition and lot number.

Material	Manufacturer	Composition	Lot Number
Surefil One	Dentsply Sirona, Konstanz, Germany	Aluminium-phosphor-strontium-sodium-fluoro-silicate glass, water, highly dispersed silicon dioxide, acrylic acid, polycarboxylic acid (MOPOS), ytterbium fluoride, 210500	
Prime&Bond Universal	Dentsply Sirona, Konstanz, Germany	Bi- and multifunctional acrylate, Phosphoric acid modi- fied acrylate resin, Initiator, Stabilizer, Isopropanol, Water.	2110001151

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Figure 1. Photos showing the steps used to flatten the dentin.

The remaining forty-five teeth were designated for measuring the bonding strength on enamel. The buccal surface of the crown of the teeth was just polished with a silicon carbide abrasive paper, grain size 600 for 30s in order to create a uniform surface on all specimens.

The teeth were divided into 6 groups (15 teeth per group) according to the bonding substrate (enamel, dentin) and the bonding protocol (No bonding agent, acid etching, etch and rinse) and each tooth was restored according to the corresponding bonding procedure, as shown in table 2.

The restored teeth were placed in distilled water at 37 degrees for 24 hours, and subjected to 10,000 thermal cycles between 5°C and 55°C using a thermocycler (THE-1100 thermocycler, SD Mechatronik, Feldkirchen-Westerham, Deutschland), with 30 seconds of exposure in each bath and 10 seconds of transfer time between baths.

evaluation of bonding The strength in this study utilized the micro-tensile test, a widely accepted method since its introduction by Sano in 1994. The micro-tensile test has become a standard technique for assessing adhesive systems due to its ability to provide precise discrimination. This method, though strength-based, offers valuable insights through morphological and spectroscopic analysis, significantly contributing to the advancement of adhesive technology [9].

Each tooth was mounted on a hard tissue microtome and serially cut at 1mm intervals in the occlusal-gingival direction for dentin bonding groups and vestibulo-lingual for enamel bonding groups. This action produces 1mm thick blocks. Each tooth was then turned 90° and the serial was repeated. Finally, sticks shapes are obtained from each tooth with composite resin in the upper part and dentin or enamel in the lower part. Three sticks with 4mm of healthy dentin or enamel were selected from each tooth.

Each stick was then placed in a Universal testing machine YL-01 (YLE GmbH Waldstraße 1/1a, 64732 Bad König, Germany) using a Geraldeli's jig to measure the micro tensile strength at a speed of 1mm/ min. The fracture force was noted by software. The microtensile bonding strength was calculated by the formula μ TBS = F / S with μ TBS = microtensile bonding strength (MPa), F = fracture force (N), and S = bonding surface (mm²).

Fractured beams were mounted on aluminum stubs and examined under a stereomicroscope (40× magnification, Stereo-zoom S8, Leica, Heidelberg, Germany). The fractures were classified according to the fractured structure: adhesive fracture between resin and dentin, cohesive fracture at the resin level, cohesive fracture at the dentin level.

Statistical Study

The micro tensile bond strength of the 90 teeth were analyzed using STATA version 15.0. Mean of the scores and standard deviations were calculated across the three groups of bonding protocol within each of the enamel and dentin categories and between the two. Normal distribution was first assessed among the different categories using Shapiro Wilk and Kolmogorov Smirnov tests. In the case of normal distribution, One-way ANOVA test was used to compare the means of the micro tensile bond strength followed by multiple comparisons using the Bonferroni correction tests. In case of absence of normal distribution, Kruskal Wallis test followed by Mann-Whitney one was used to evaluate the corresponding values. Lastly, Fisher's exact test was used to assess the association between the type of fracture and the bonding protocol across dentin ad enamel categories.

Table 2. Specimens grouping, bonding substrate & protocol, and bonding procedure.

Group	Bonding Substrate and Protocol	Bonding Procedure		
Group 1	Dentin (Control)	Surefil One without bonding agent		
Group 2	Dentin with acid etching	15s acid etching with 36% phosphoric acid + Surefil One		
Group 3	Dentin in etch and rinse mode	15s acid etching with 36% phosphoric acid + universal adhesive + Surefil One		
Group 4	Enamel (Control)	Surefil One without bonding agent		
Group 5	Enamel with acid etching	30s acid etching with 36% phosphoric acid + Surefil One		
Group 6	Enamel in etch and rinse mode	30s acid etching with 36% phosphoric acid + universal adhesive + Surefil One		

Results

Results of Micro Tensile Bond Strength in enamel

According to the Kolmogorov-Smirnov test, the micro-tensile bond strength data on enamel were not normally distributed (P < 0.05).

Regarding the effect of different bonding protocols on enamel micro-tensile bond strength, as shown in table 3, the highest micro-tensile bond strength values was recorded when in etch and rinse mode followed by selective etch mode, while control group showed the lowest bond strength value with a statistically significant difference (P = 0.03) according to Kruskal Wallis Test.

Thus, Mann Whitney test was applied for pairwise comparisons and showed significant associations between the micro tensile bond strength and bonding protocols across the three groups (after adjustment of p-value, 0.01*3 = 0.03).

Results of Micro Tensile Bond Strength in dentin

According to the Kolmogorov-Smirnov test, the micro-tensile bond strength data on dentin were normally distributed (P > 0.05).

As shown in table 4, μ TBS to dentin recorded the highest micro-tensile bond strength values when bonded in etch and rinse mode followed by selective etch mode, while control group showed the lowest bond strength value with a statistically significant difference (P = 0.001) according to one-way ANOVA.

As shown in table 5, multiple comparisons using the Bonferroni correction tests conducted between

the different bonding protocols revealed the following findings:

- A statistical mean difference of the μ TBS between control and selective etch; Selective etch protocol at fractured a strength of 4.24 MPa higher than the control group (p-value < 0.001).
- A statistical mean difference of the μ TBS between control group and etch & Rinse; Etch & Rinse protocol fractured at a strength of 10.47 MPa than the control group (p-value<0.001).
- A statistical mean difference of the μ TBS between etch and etch bond; Etch Bond protocol needed higher μ TBS of 6.22 MPa than the etch group.

Table 3. Medians of micro tensile bond strength across bonding protocols groups among enamel.

Ponding Protocol	Enamel		P-value
Bonding Protocol	Median (MPa)	Interquartile Range	
Control Group	7.33	(7.33; 8)	
Selective Etch	14.33	(13.33; 17.33)	0.03*
Etch & Rinse	19	(18; 20.33)	

*Significant at p< 0.05.

Table 4. Mean micro tensile bond strength values of different bonding protocols, across dentin category.

Dending Drate col	Dentin	P-value	
Bonding Protocol	Median (MPa)	CI (95%)	
Control Group	7.17	(6.49; 7.86)	
Selective Etch	11.42	(10.76; 12.07)	<0.001*
Etch & Rinse	17.64	(16.44; 18.85)	

*Significant at p<0.05.

Table 5. Mean micro tensile bond strength difference between bonding protocols, across dentin category.

Ponding Protocol	Dentin	P-value	
Bonding Protocol	Mean Difference (MPa)	CI	
Control Group with Etched Group	-4.24	(-5.70; -2.79)	
Control Group with Etch and rinse Group	-10.47	(-11.92; -9)	<0.001*
Etched Group with Etch and Rinse Group	-6.22	(-7.68; -4.76)	

*Significant at p<0.05.

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Micro-tensile bond strength of Dentin vs Enamel

As shown in table 6, significant differences of the μ TBS medians were detected between dentin and enamel in the groups of etch and rinse and selective etch according to Mann Whitney test. Etch and rinse, and selective etch on enamel had higher μ TBS than on dentin. There was no significant difference between the control groups of enamel and dentin.

Mode of Failures in Enamel

As shown in table 7, no significant association was found between the type of fracture and bonding protocol at the level of the enamel according to Fisher Exact test (p-value>0.05).

Mode of Failures in Dentin

According to Fisher Exact test, a significant association between the type of fracture and the bonding protocol was found (p-value = 0.017) (table 8). The proportion of cohesive type of fracture is higher among the etch and rinse group compared to the other two bonding protocols. The proportion of cohesive fracture type among the acid etched group is higher than the control group, and the proportion of adhesive type of fracture is the highest across the control group. Etch and rinse protocol has the lowest proportion of adhesive type of fracture.

Mann Whitney test showed a significant association between the type of fracture and bonding protocol between the control group and the etch and risne group (p-value adjusted =0.036). The Control group has a higher percentage of adhesive fractures, while the Etch & Rinse group has a higher percentage of cohesive fractures.

Discussion

The null hypothesis, that the micro tensile bond strength of the self-adhesive bulk fill composite (Surefil One) on enamel and dentin was not Table 6. Mean micro tensile bond strength difference between bonding protocols, across dentin category.

	Enamel Vs [P-value	
Bonding Protocol	Mean Difference (MPa)	IQR	
Control Group	7.33 vs 7.17	(7.33;8) vs (6.49;7.86)	0.37
Etched Group	14.33 vs 11.42	(13.33;17.33) vs (10.76;12.07)	<0.001*
Etch and Rinse Group	19 vs 17.64	(18;20.33) vs (16.44;18.85)	0.034*

*Significant at p<0.05.

Table 7. Proportions of type of fracture across bonding protocols groups among enamel category.

	Bonding Protocol				
Type of Fracture	Control	Etched	Etch and rinse	P-value	
Cohesive (in composite)	8.9%	11.1%	9.1%	0.928	
Adhesive	91.1%	88.9%	90.9%	0.928	

*Significant at p<0.05.

Table 8. Proportions of type of fracture across bonding protocols groups among enamel category.

	Bo				
Type of Fracture	Control	Etched	Etch and rinse	P-value	
Cohesive (in composite)	11.1%	29.5%	35.5%	0.017*	
Adhesive	88.9%	70.4%	64.5%	0.017*	

*Significant at p<0.05.

affected by an additional etching using 36% phosphoric acid or the application of a bonding agent was rejected. The results showed that the μ TBS was significantly higher in the etch and rinse group in enamel (19 MPa) and dentin (17.64 MPa), and an additional etching of enamel and dentin significantly increased bond strength compared to the control groups without adhesive system, which indicates that acid etching using 36% phosphoric acid and using an adhesive before the application of the self-adhesive bulk fill composite would help improve the bond strength.

Concerning fracture modes, there was a significant difference in fracture mode between the three differ-

ent groups, both in enamel and dentin. The control groups had a higher percentage of adhesive fractures, indicating that the bonding interface was weakest, whereas the etch and rinse groups had a higher percentage of cohesive fractures, indicating a strong bonding interface. All of the cohesive fractures were in the composite, with no cohesive fractures in the dentin or enamel, which can be explained by the composites' poor mechanical properties.

Acid etching is a popular technique for roughening the enamel surface and enhancing the adhesive materials' bond strength to enamel. The development of porosities in the enamel surface and the penetration of resin into the porosities are likely the causes of the total etch and the selective etch groups' noticeably higher bond strength compared to the control group which didn't undergo acid etching. According to the literature, when enamel is acid-etched, the enamel rods are selectively dissolved, resulting in macro- and micro-porosities that can be easily penetrated by capillary attraction and even common hydrophobic bonding agents [10, 11]. After polymerization, the acid-etched enamel surface's micromechanical interlocking of tiny resin tags still offers the strongest bond possible to the dental substrate [12].

However, there are some issues related to the use of self-etch adhesives on enamel [13]. The bond strength and durability may be adversely affected by the enamel's superficial etching pattern and a decrease in micromechanical retention [14]. Furthermore, it is unknown if self-etch adhesives with moderate pH levels applied to enamel will be able to offer the same level of mechanical and chemical resistance as etch and rinse adhesives in an oral environment [15].

According to van Landuyt et al.'s research, which is consistent with our own, acid etching of the enamel's surface significantly boosts the bond strength [16]. Therefore, it should be realized that, while eliminating the etch and rinse steps from the adhesive application process simplifies the process and improves the bonding process, it does not always translate into a higher clinical success rate.

Because of the composition of dentin, adhesion to dentin is more difficult than adhesion to enamel, making the etch-and-rinse technique a very sensitive one [17, 18]. Acid-etching encourages dentin demineralization over a depth of 3-5 μ m, exposing a collagen fibril scaffold that is almost entirely depleted of hydroxyapatite [19]. In the bonding step, a solvent-free adhesive resin is applied to the prepared surface, allowing hydrophobic monomers to enter both the dentin tubules and the interfibrillar spaces of the collagen network. When these monomers are infiltrated, they are polymerized there, creating a hybrid layer that, along with the presence of resin tags inside the dentin tubules, gives the composite restoration micromechanical retention [20, 21].

The self-adhesive composite's ability to bond to enamel and dentin was greatly enhanced by pre-etching. This was anticipated when bonding to enamel because phosphoric acid significantly increases enamel's surface energy and thereby offers significantly greater micro-retention. A self-adhesive luting composite was previously found to bond to enamel with a similar improved bonding effectiveness after phosphoric-acid etching [22]. However, it was not anticipated that pre-etching dentin with phosphoric acid would improve Surefil One's bonding efficiency because earlier bond-strength tests had shown the opposite, and thus detrimental effect [22]. In De Munck's study, TEM revealed that phosphoric acid clearly exposed collagen up to a few micrometers' depth. The relatively viscous self-adhesive composite was unable to adequately hybridize the collagen mesh, resulting in a significant reduction in bonding effectiveness [22]. In our study, we found that pre-etching dentin increased bond strength.

Similar to our study, Poitevin et al. compared the effectiveness of self-adhesive flowable composites to enamel and dentin using various bonding protocols. They discovered that the μ TBS of self-adhesive composite (SAC) alone to dentin was significantly lower than that of the combination of a self-etch adhesive with SAC, and that the highest value was obtained using a combination of etch and rinse adhesive with SAC [23]. These results are consistent with our findings.

Fu et al. compared the μ TBS values after applying to the dentin a SAC without adhesive and two traditional resin composites with two different SE adhesive systems. SE

adhesives and conventional resin composites had higher μ TBS values than SACs without an adhesive, and the difference was statistically significant [24]. Similar to the findings of this study, our study found that using SACs with adhesive systems resulted in higher μ TBS values than non-adhesive uses.

A study conducted by Cengiz et al. evaluated the μ TBS of two different self-adhesive flowable composite resins using different universal adhesives and bonding protocols. It was found that the use of self-adhesive composites with universal adhesive systems increased the μ TBS values [25].

In contrast with our study, Pouyanfar et al. did not find any statistical difference in the μ TBS when using a universal adhesive with or without acid pre-etching on enamel. They also found that the μ TBS of universal adhesive is as high as that of 2-steps etch and rinse and 2 steps self-etch bonding agents [15].

Sadeghyar et al. compared the bond strength of 4 different self-adhesive restorative materials with and without pre-treatment of dentin. One of the materials was Surefil One. They found that pretreatment of dentin significantly increased bond strength [26].

Marigano et al. analysed the cytocompatibility of Surefil One with respect to the release of monomers from the material. They found that the toxic effect induced by the material was insignificant and that the material have good cytocompatibility consistent with the non-determinability of the monomers released after polymerization [27].

In a recent study, the flexural strength of Surefil One was compared between a light-curing protocol and a self-curing protocol. It was found that using a light-curing protocol for Surefil one yielded the highest flexural strength. It was also found that shear bond strength of Surefil One significantly increased when a universal adhesive was used before applying the self-adhesive composite [28].

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Alzahrani et al. compared the biaxial flexural strength (BFS) of Surefil One and 3 other materials before and after storage in water and ethanol. They found that Surefil One showed the lowest BFS values and that it wasn't recommended to use in stress bearing areas [29].

The reliability of the research findings is negatively impacted in in-vitro studies using composite resins and adhesive systems when intra-oral conditions cannot be accurately reflected in the studies [30]. As a result, a variety of aging techniques, including thermal cycling application, are used in numerous studies [31, 32]. According to reports, 1 years' worth of a natural cycle was represented in the studies by applying thermal cycling 10.000 times between 5 and 55C [33]. In our study, we used thermal cycling to treat all of the samples at a rate of 10.000, with dipping times of 30s and transfer times of 10s between containers, at temperatures ranging from 5 to 55C.

The μ TBS test was used in the current study to assess the bond's strength. When compared to traditional tensile or shear loads, the application of micro tensile load leads to better stress distribution at the adhesive interface and produces more accurate results with

less diversity. The smaller interface area, which was 1 mm in our study, allows for better stress distribution during this test.

However, because many factors, including masticatory loads, pH changes, and thermal changes are present in the oral environment and affect the bond strength of adhesives to tooth structure [34], the bond strength tests are only suitable for ranking adhesives. Therefore, the performance of adhesives in a clinical setting cannot be accurately predicted by the results of in vitro tests for bond strength. Another limitation of this study is that only three specimens were taken from each tooth. Knowing that bonding strength is hugely affected by the different types and location of dentin, this may influence the values of the μ TBS obtained during testing.

Conclusion

Self-adhesive composites are clinically appealing because they simplify the way teeth can be restored adhesively and thus minimally invasively. This in vitro bond-strength study revealed that an additional etching before the application of a self-adhesive composite would improve the bond strength, and that optimal bond strength is obtained when the self-adhesive composite is used in combination with 2 steps etch and rinse adhesive system. As a result, routine clinical application of SACs should be approached with caution, especially if no macro-retention is provided.

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Author Contributions

Conceptualization, M.Y. and C.K.; methodology, L.H.; software, G.N.; validation, C.K and L.H.; formal analysis, M.Y.; investigation, M.Y.; resources, M.Y.; data curation, G.N.; writing—original draft preparation, M.Y.; writing—review and editing, M.Y.; visualization, C.K.; supervision, L.H.; project administration, C.K.; funding acquisition, M.Y. All authors have read and agreed to the published version of the manuscript.

References

- 1. Aqil NSA. Polymerization problems in flowable bulkfill dental composite in comparison to conventional composite: A systematic review. 2020;9(3):5.
- He Z, Shimada Y, Tagami J. The effects of cavity size and incremental technique on micro-tensile bond strength of resin composite in Class I cavities. Dent Mater. 2007 May;23(5):533–8.
- Annelies VE. Bulk-Fill Composites: A Review of the Current Literature. J Adhes Dent. 2017 May 12;19(2):95–109.
- 4. Veloso SRM, Lemos CAA, de Moraes SLD, do Egito Vasconcelos BC, Pellizzer EP, de Melo Monteiro GQ. Clinical performance of bulk-fill and conventional resin composite restorations in posterior teeth: a systematic review and meta-analysis. Clin Oral Investig. 2019 Jan 1;23(1):221–33.
- Schmidt M, Dige I, Kirkevang LL, Vaeth M, Hørsted-Bindslev P. Five-year evaluation of a low-shrinkage Silorane resin composite material: a randomized clinical trial. Clin Oral Investig. 2015 Mar;19(2):245–51.
- Balkaya H, Arslan S, Pala K. A randomized, prospective clinical study evaluating effectiveness of a bulkfill composite resin, a conventional composite resin and a reinforced glass ionomer in Class II cavities: one-year results. J Appl Oral Sci. 2019 Oct 7;27.
- 7. Jarahi N, Borouziniat A, Jarahi L, Nejat AH. Effect of Different Storage Solutions and Autoclaving on Shear Bond Strength of Composite to Dentin. J Res Med Dent Sci. 2018;6(6).
- Aydın B, Pamir T, Baltaci A, Orman MN, Turk T. Effect of storage solutions on microhardness of crown enamel and dentin. Eur J Dent. 2015;9(2):262–6.
- Sano H, Chowdhury AFMA, Saikaew P, Matsumoto M, Hoshika S, Yamauti M. The microtensile bond strength test: Its historical background and application to bond testing. Jpn Dent Sci Rev. 2020 Nov;56(1):24–31.
- Rivera G, Paula L, An T, Garcia F, Yamaguti P, Marsiglio A, et al. A Comparative Study of a New Microtensile Testing Device for Dental Research. Br J Appl Sci Technol. 2016 Jan 10;13(4):1–9.
- 11. Gwinnett AJ, Matsui A. A study of enamel adhesives. The physical relationship between enamel and adhesive. Arch Oral Biol. 1967 Dec;12(12):1615–20.
- Van Meerbeek B, De Munck J, Mattar D, Van Landuyt K, Lambrechts P. Microtensile bond strengths of an etch&rinse and self-etch adhesive to enamel and dentin as a function of surface treatment. Oper Dent. 2003;28(5):647–60.

- Tay FR, Pashley DH, King NM, Carvalho RM, Tsai J, Lai SCN, et al. Aggressiveness of self-etch adhesives on unground enamel. Oper Dent. 2004;29(3):309– 16.
- Miyazaki M, Sato M, Onose H. Durability of enamel bond strength of simplified bonding systems. Oper Dent. 2000;25(2):7580.
- 15. Pouyanfar H, Tabaii ES, Aghazadeh S, Nobari SPTN, Imani MM. Microtensile Bond Strength of Composite to Enamel Using Universal Adhesive with/without Acid Etching Compared To Etch and Rinse and Self-Etch Bonding Agents. Open Access Maced J Med Sci. 2018 Nov 25;6(11):2186–92.
- Van Landuyt KL, Kanumilli P, De Munck J, Peumans M, Lambrechts P, Van Meerbeek B. Bond strength of a mild self-etch adhesive with and without prior acid-etching. J Dent. 2006 Jan;34(1):77–85.
- Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, Van Meerbeek B. Clinical effectiveness of contemporary adhesives: a systematic review of current clinical trials. Dent Mater Off Publ Acad Dent Mater. 2005 Sep;21(9):864–81.
- Hardan L, Bourgi R, Cuevas-Suárez CE, Zarow M, Kharouf N, Mancino D, et al. The Bond Strength and Antibacterial Activity of the Universal Dentin Bonding System: A Systematic Review and Meta-Analysis. Microorganisms. 2021 Jun 6;9(6):1230.
- J P, P L, B VM, M B, E Y, T Y, et al. The interaction of adhesive systems with human dentin. Am J Dent. 1996 Aug;9(4).
- Van Meerbeek B, Dhem A, Goret-Nicaise M, Braem M, Lambrechts P, VanHerle G. Comparative SEM and TEM examination of the ultrastructure of the resin-dentin interdiffusion zone. J Dent Res. 1993 Feb;72(2):495–501.
- Hardan L, Bourgi R, Kharouf N, Mancino D, Zarow M, Jakubowicz N, et al. Bond Strength of Universal Adhesives to Dentin: A Systematic Review and Meta-Analysis. Polymers. 2021 Mar 7;13(5):814.
- De Munck J, Vargas M, Van Landuyt K, Hikita K, Lambrechts P, Van Meerbeek B. Bonding of an auto-adhesive luting material to enamel and dentin. Dent Mater Off Publ Acad Dent Mater. 2004 Dec;20(10):963–71.
- Poitevin A, De Munck J, Van Ende A, Suyama Y, Mine A, Peumans M, et al. Bonding effectiveness of self-adhesive composites to dentin and enamel. Dent Mater. 2013 Feb;29(2):221–30.

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- Fu J, Kakuda S, Pan F, Hoshika S, Ting S, Fukuoka A, et al. Bonding performance of a newly developed step-less all-in-one system on dentin. Dent Mater J. 2013;32(2):203–11.
- 25. Cengiz T, Ünal M. Comparison of microtensile bond strength and resin–dentin interfaces of two self-adhesive flowable composite resins by using different universal adhesives: Scanning electron microscope study. Microsc Res Tech. 2019 Jul;82(7):1032–40.
- Sadeghyar A, Lettner S, Watts DC, Schedle A. Alternatives to amalgam: Is pretreatment necessary for effective bonding to dentin? Dent Mater. 2022 Nov;38(11):1703–9.
- Marigo L, Triestino A, Castagnola R, Vincenzoni F, Cordaro M, Di Stasio E, et al. Cytotoxic Evaluation of the New Composite Resin through an Artificial Pulp Chamber. Guven Y, editor. BioMed Res Int. 2022 Nov 16;2022:1–10.
- Francois P, Fouquet V, Attal JP, Dursun E. Commercially Available Fluoride-Releasing Restorative Materials: A Review and a Proposal for Classification. Materials. 2020 May 18;13(10):2313.
- 29. Alzahrani B, Alshabib A, Awliya W. Surface hardness and flexural strength of dual-cured bulk-fill restorative materials after solvent storage. BMC Oral Health. 2023 May 19;23(1):306.

- Munksgaard EC, Itoh K, Jörgensen KD. Dentin-polymer bond in resin fillings tested in vitro by thermoand load-cycling. J Dent Res. 1985 Feb;64(2):144–6.
- 31. Guler S, Unal M. The Evaluation of Color and Surface Roughness Changes in Resin based Restorative Materials with Different Contents After Waiting in Various Liquids: An SEM and AFM study. Microsc Res Tech. 2018 Dec;81(12):1422–33.
- Baracco B, Fuentes MV, Garrido MA, González-López S, Ceballos L. Effect of thermal aging on the tensile bond strength at reduced areas of seven current adhesives. Odontology. 2013 Jul;101(2):177–85.
- Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. J Dent. 1999 Feb;27(2):89–99.
- Iliev G, Hardan L, Kassis C, Bourgi R, Cuevas-Suárez CE, Lukomska-Szymanska M, et al. Shelf Life and Storage Conditions of Universal Adhesives: A Literature Review. Polymers. 2021 Aug 13;13(16):2708.