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**BENEFITS OF SELF-ETCH ADHESIVES ACTIVE APPLICATION WITH
ROTARY BRUSH TO ENAMEL**

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Keywords: Active application, Microshear bond strength, Enamel, Self-etch, Universal adhesive.

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Abstract

Background/Aim. The aim of this study was to evaluate application of a universal adhesive in a self-etch mode and a self-etch adhesive using a rotary brush active application technique by microshear bond strength (μ SBS) test. **Methods.** The crown parts of 20 bovine teeth were separated from their roots, embedded in acrylic blocks and enamel surfaces were prepared. The prepared crowns were divided into four groups according to the adhesive system tested (1) Nova Compo-B Plus, and (2) Optibond All-in-one, and application technique (1) manual active application and (2) rotary brush active application. Bonded samples were immersed in distilled water for 24-h before bond strength testing by μ SBS test at 1.0 mm / min. Data were analyzed using non-parametric tests ($p = 0.05$). **Results:** When both adhesives were applied by rotary brush active application technique, they showed significantly higher enamel bonding strength compared to the manual active application technique. **Conclusion:** Application of self-etch adhesives to enamel with a rotary brush active application technique can increase initial resin-enamel bond strength compared to the manual active application.

Key words:

adhesives, active application, microshear bond strength, enamel, self-etch.

Apstrakt

Uvod/Cilj. Cilj ove studije je procena primene univerzalnog adheziva u samovezujućem modu i samovezujućeg adheziva primenom tehnike aktivne aplikacije rotirajućom četkicom, korišćenjem testa mikro-smicanja (μ SBS). **Metode.** Delovi krunica 20 goveđih zuba odvojeni su od korenova, ugrađeni su u akrilatne blokove i pripremljene su površine gleđi. Pripremljene krunice su podeljene u četiri grupe prema testiranom adhezivnom sistemu (Nova Compo-B Plus i Optibond All-in-one) i tehnici primene (ručna aktivna aplikacija i aktivna aplikacija rotirajućom četkicom). Vezani uzorci su potopljeni u destilovanu vodu 24 sata pre ispitivanja jačine veze μ SBS testom na 1,0 mm/min. Podaci su analizirani pomoću neparametrijskih testova ($p = 0,05$). **Rezultati:** Kada su oba adheziva primenjena tehnikom aktivne aplikacije rotirajućom četkicom, pokazali su značajno veću čvrstoću vezivanja za gleđ u poređenju sa tehnikom ručne aktivne aplikacije. **Zaključak:** Aplikacija samolepivih adheziva na gleđ tehnikom aktivnog nanošenja rotirajućim četkicama može povećati inicijalnu snagu vezivanja smole za gleđ u poređenju sa tehnikom aktivne ručne aplikacije.

Ključne reči:

aktivna aplikacija, jačina veze mikro-smicanja; gleđ; samovezujući adheziv.

Introduction

Resin adhesive systems available in the dental market are divided into two main categories depending on bonding strategies: etch-and-rinse (ER) and self-etch (SE) adhesive systems. ER adhesive systems have two or three application step versions, and SE adhesive systems have one or two application steps versions^{1,2}. Newly, manufacturers have introduced more versatile adhesive systems, able to be used in both bonding strategies. The manufacturers suggest that clinicians may choose to use bonding strategies according to their preference and type of tooth tissue^{3,4}. Recently introduced to the dentistry market, this novel group of

dental adhesives are called “multi-mode” or “universal” adhesives and constitute the newest generation of adhesives^{5,6}.

However, studies have shown that these new adhesives provide lower bond strength when applied to enamel in self-etch mode^{5,7,8}. This is similar to the low bond strength of the previous single-stage self-etch adhesives with enamel^{9,10}. Compared to phosphoric acid, self-etch adhesives and universal adhesives have lower acidity. Due to these characteristics, the demineralization capacities are not sufficient to produce satisfactory micro-retentive porosity. As a result, the force they provide for enamel bonding is not at the desired level^{2,11}.

This may not be considered as a new problem, the successful methods for solving such problems have already been tested to increase the enamel bonding, such as application of phosphoric acid etching prior to adhesive application^{3,12}. On the other hand, in clinical studies, it was not detected that the universal adhesives used by selective enamel-etching had a positive effect on the survival time of cervical restorations¹³⁻¹⁵. However, it has been found to reduce the marginal staining^{15,16}. In addition, pre-treatment of phosphoric acid increases the number of clinical application steps of the adhesive system.

In recent studies, some researchers have shown that when universal adhesives are used in self-etch mode, active application may be an alternative to selective enamel-etching technique in terms of enamel bonding strength². It has already been shown that the active application increases the bond strength of one-step self-etch adhesives with low enamel bonding strength^{17,18}. In a clinical trial, it has been reported that active application of one-step self-etch adhesive provides a higher 2-year retention rate and lower marginal coloration. These clinical findings support the findings of above laboratory researches¹⁹. As another clinical method, self-etch adhesive agitation has been attempted with sonic waves by special device, and it has been reported to increase enamel bonding²⁰.

Theoretically, agitation of self-etch adhesive and solutions can be significantly accelerated by the use of a rotary brush. The bristles of the brush may disrupt the integrity of the smear layer on the enamel surface during rotation. At this time, a circulation in the adhesive solution can be made between the fresh acidic monomers on the surface and the buffered passive monomers after initial superficial demineralization on the enamel surface. As a result, a deeper and more continuous interaction can take place between the enamel and the acidic monomers. All of these facts can improve the enamel bonding of self-etch adhesives and solutions, which do not have good enamel bond strength. For this reason, it is interesting to see how the active application of self-etch adhesives and universal adhesives, applied in self-etch mode with rotating brushes, affects the bond strength of these adhesives, which were evaluated in this study. Null hypothesis of the study was that active application with rotary brush won't have the effect on the bond strength of the adhesives tested.

Methods

Study Design

The adhesive system (brand of adhesives) and the adhesive application mode (application of manufacturer instructions and rotary micro-brush application) are independent variables. The microshear bond strength mean is dependent variable. The contents of the adhesive systems and user instructions are shown in Table 1. In total, there were four groups in the

study. 5 teeth were used for each group. In the microshear test, more than one composite button could be placed on a single tooth. According to the width of the enamel surface, 6-9 bonding test specimens per tooth were prepared.

Sample Preparation

In total, 20 extracted bovine incisors were used. The teeth were kept in a solution of 0.1% sodium azide for a maximum of six months after tooth extraction prior to be used. The root parts of the teeth were cut from the crown parts by cutting with a low-speed diamond disc under water cooling. The crowns, individually were embedded in the plexi molds with autopolymerized acrylic, with the vestibular surface of the tooth upward. The exposed enamel surfaces were first smoothed with 180-grit silicon carbide abrasive paper, then polished with 600-grit silicon carbide abrasive paper for one minute under water-cooling.

Resin-Enamel Microshear Bond Strength (μ SBS)

Procedures of resin-enamel μ SBS test and bonding procedures were shown in Figure 1. Universal adhesives were applied in self-etch mode according to their manufacturer's instructions, except for rotary brush groups (Table 1). A single operator applied all bonding procedures as described below:

(1) Manual active application: Adhesives were actively applied to the enamel according to the manufacturer's instructions.

(2) Rotary brush active application: The 3 mm-diameter prophylaxis brush was attached to the micromotor-mounted contra-angle handpiece operating at 1200 rpm. An adhesive was dispensed to the enamel surface with a regular applicator and then agitated with rotary brush in sweeping motion. The application took 20-30 seconds on average. When there was no visible solvent on the surface, the application was finished. Furthermore, the adhesive was not dried by air-water spray to evaporate the solvent. After the adhesive application procedures were finished, the adhesives were polymerized with the LED light source for 20 seconds.

With the rubber dam puncher, 6 - 8 holes having inside diameter of 1 mm were punched on the double-sided adhesive tape. This adhesive tape was adhered to the prepared enamel surface after adhesive polymerization. The number of perforations was adjusted according to the width of the enamel surface. After adhesive polymerization, Tygon tubes with an inner diameter of 0.8 mm and a height of 0.5 mm were placed corresponding to the holes on the double-sided tape. Because this procedure is sensitive, the operator used a dental loupe with 2.5x magnification during the application.

For the Tygon tubes to remain stable, a flowable composite was placed in the spaces between the tygon tubes. In this way, the possibility of moving the Tygon tubes when placing the composite into them was minimized. Each adhesive was used with the resin composite of its own manufacturer (Table 1). Resin composites were carefully placed into the tygon tubes by the operator using the dental loupe. The composites were polymerized with 20 seconds led light source. After polymerization of the composites, Tygon tubes were cut with scalpel. Bonded samples were immersed in distilled water for 24-h before bond strength testing.

The bonded samples were fixed to the shear-test jig and tested on the universal test device. The blade was placed as close as possible to the resin - enamel surface interface. The speed of the crosshead was set to 1 mm / min. The blade was moved downwards until it broke the composite. The μ SBS values were calculated as the MPa unit by dividing the load causing the failure by the surface area. In this way, shear bond strength was determined. Failure modes, only enamel or composite covering cohesive (C), resin-enamel interface that occurs in the adhesive (A) or partially enamel or composite is classified as a mix failure (M). Failure modes were determined by digital microscope with the aid of 20-30x magnification.

Statistical Analysis

The normal distribution of μ SBS means and the homogeneity of the variance between the averages were analyzed by Kolmogorov-Smirnov test and Levene's test, respectively. According to the Levene's test, since the variance was heterogeneous among the means, parametric tests could not be applied to the μ SBS data. Therefore, Kruskal-Wallis and Mann-Whitney U-tests were applied to the data ($p < 0.05$).

Results

Findings of μ SBS test was summarized in Table 2. When all adhesives were applied by rotary brush active application technique, they showed significantly higher enamel bonding strength compared to the manual active application technique ($p < 0.05$; Table 2). Nova Combo B-Plus adhesive gave significantly higher bonding strength than the Optibond All-in-one adhesive in both application techniques ($p < 0.05$; Table 2). Most of the samples showed adhesive or mixed rupture (Table 2).

Discussion

In this study, the technique of applying the adhesives with a rotary brush was tested in order to increase enamel bonding strengths of the universal adhesive applied in self-etch mode and self-etch adhesive as a different method. Null hypothesis of the study was that active application with rotary brush will not have effect on the bond strength of the adhesives tested. Our results suggest that null hypothesis was rejected because the active application technique with rotary brush showed that the two adhesives tested significantly increased their enamel bond strengths.

Since pH values of the self-etch adhesives are usually mild or ultra-mild, these adhesives cannot demineralize the enamel as well as phosphoric acid. Therefore, the enamel bonding strengths of self-etch adhesives are generally lower than those of the etch-and-rinse adhesives^{9,10}. However, this problem also applies to the universal adhesives applied in the self-etch mode⁶. In order to solve this problem, methods such as pre-treatment of enamel surfaces with phosphoric acid, and active application of adhesive with sonic devices have been proposed^{12,20}.

The problem of low enamel bonding strength, which is an issue of self-etch solutions, can be solved by active application of the rotary brush. In previous studies, it has been shown that the self-etch protocol does not produce retentive patterns on the enamel surface²¹. Probably, the superficial interaction of the self-etch solutions with enamel prevents the acidic monomers from demineralizing to give the enamel sufficiently strong bond strength.

When applying the self-etch solution to the enamel surface with a rotary brush active application, it is undoubted that the brush's bristles will impair the integrity of the smear layer as a result of the rotation of the brush. As a result, the acidic monomers can pass through the smear layer to the underlying intact enamel. In addition, the rotating effect of the brush can act in the self-etch solution itself, allowing the movement of the fresh monomers towards the enamel and demineralization waste being transported outwardly. This may have led to a deeper chemical interaction between fresh acidic monomers and enamel. Finally, self-etch solution applied by rotary brush may have increased capacity to produce retentive area in the enamel for further mechanical interlocking. In previous studies, it has been reported that active application with sonic device enables self-etch solutions to make better demineralization in enamel²⁰. The similar mechanism of action may also apply to the rotary brush active application.

The problem of low enamel bonding strength of self-etch solutions can be solved by phosphoric acid pre-treatment. But this solution makes the adhesive application less user-friendly. Furthermore, poor taste of phosphoric acid gel is not comfortable for the patients. Application of a self-etch solution with a rotary brush has no such disadvantages. Moreover, this new technique evaporates the solvent in the adhesive solution during accelerated active application. It does not require the solvent to be dried by air spray after adhesive application. When the adhesive is applied for a sufficient time with a rotating brush, the enamel surface covered by uncured adhesive solution has a clinical appearance as if it were air-dried. In this respect, it can be argued that this technique makes the adhesive application procedures more user-friendly. However, the issue that the rotary brush active application may eliminate the solvent evaporation step would be an important improvement that will significantly simplify the application of self-etch solutions. For this suggestion, further studies are needed.

When the literature is reviewed, it is seen that sonic application of self-etch solutions also improves the enamel bonding performances of these adhesives²⁰. In this method, sonic waves are transmitted to the adhesive solution by a special device. It has been suggested that the sound waves make a circulation in the adhesive solution, facilitating solvent evaporation. An advantage of the rotary brush active application from sonic application is that this technique does not require any special equipment. An active application of self-etch solutions can be made with a small diameter brush attached to any micromotor with adjustable rotation speed. In this technique, the smear layer is physically deteriorated, the circulation of the monomers within the adhesive solution is maintained, and the solvents in the adhesive are evaporated.

One of limitations of the rotary-brush active application technique is that it is yet a novel technique. The rotational speed of the brush, the diameter of the brush, the application pressure of the brush, the geometric structure of the cavity, the adhesive materials that being used etc. would affect the effect of the rotating brush active application on the resin-mine bond strength. Therefore, it is seen that further studies are needed on this respect in the future.

Conclusion

Application of self-etch adhesives to enamel with a rotary brush active application technique can increase the resin-enamel bond strength compared to the manual active application. Therefore, the active application technique with rotary brush can be an alternative method to increase the enamel bonding strengths of the self-etch adhesives or universal adhesives applied in self-etch mode.

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Disclosure statement

The author does not have any potential conflict of interest.

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Table 1. Description of the tested adhesive systems.

Adhesive	Chemical Composition	ph	Instructions for use
Nova Compo-B Plus (Imicryl, Konya, Turkey)	Bis-GMA, HEMA, ethanol, 10-MDP, 4-META, silanated nano silica, initiators, water	2.5-2.7	Apply with agitation for 20 s. Gently air-dry for 5 s. Light cure for 10 s.
Optibond All-in-one (Kerr, Orange, CA, USA)	GPDM, HEMA, GDMA, Bis-GMA, water, acetone, ethanol, CQ, silica filler	2.5-3.0	Apply with agitation for 20s. Start with gentle air blowing, followed by stronger air blow.

10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA: bisphenol glycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; 4-META; 4-methacryloxyethyl trimellitateanhydride;

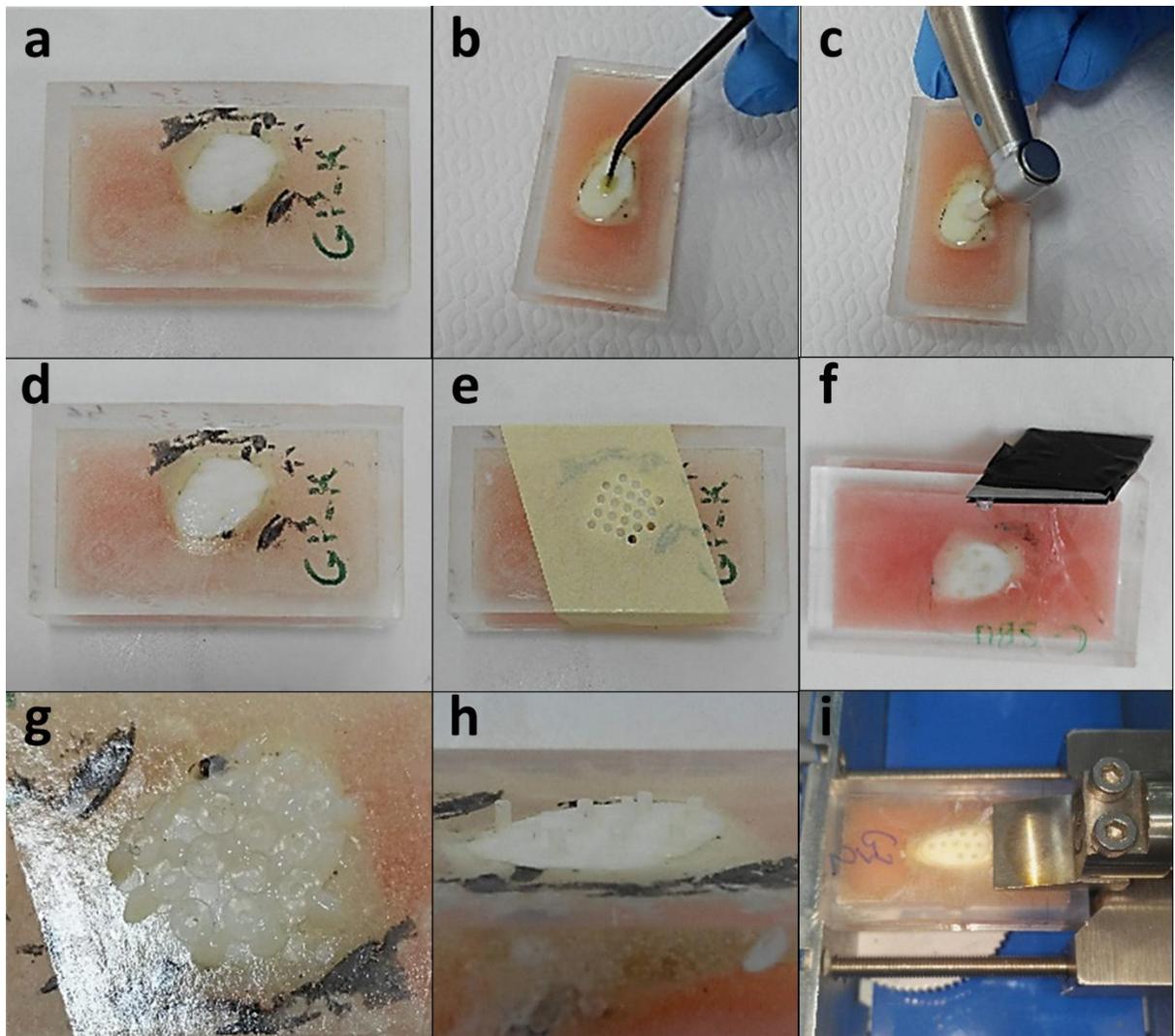
Table 2. Means, standard deviations of the Microshear bond strength (μ SBS; MPa) and failure modes (%).

Application technique	Adhesive	μ SBS (MPa)	<i>n</i>	Failure modes (%)		
				A	C	M
Manual active application	Nova Combo B-Plus	14.88 \pm 6.7 ^a	32	58.2	12.7	21.8
Manual active application	Optibond All-in-one	10.82 _b \pm 5.2	40	72.7	7.3	16.4
Rotary brush active application	Nova Combo B-Plus	19.82 \pm 7.8 ^c	28	67.3	9.6	19.3
Rotary brush active application	Optibond All-in-one	14.62 \pm 6.0 ^a	31	61.6	10	26.7

Different superscripts indicate significant differences within the same column ($p < 0.05$). A: adhesive failure mode; C: cohesive failure mode; M: mix failure mode.

FIGURE LEGENDS

Figure 1. Procedures of specimen preparation for microshear bond strength test and rotary-brush active application technique. **(a)** the crowns of the bovine incisors were embedded in acrylic as seen in the figure, and the enamel surface was finished with 600-gran SiC abrasive paper under water cooling; **(b)** in manual active application groups, adhesive was applied to enamel surfaces with an applicator; **(c)** in rotary brush active application groups, adhesive was applied to enamel surface with micro-brush with 3-mm diameter. The brush was applied to the entire enamel surface with light pressure and sweeping movements until the solvent of the adhesive was visibly evaporated. This time was 30-40 seconds at 1200 rpm; **(d)** after the adhesive is applied to the enamel surface by a rotary brush the adhesive was seen before it is polymerized; **(e)** in order to keep the Tygon tubes fixed on bonding surface during packing resin composite, a double-sided tape with holes with diameter of 1.5 mm was placed on the surface. The area where the holes were placed was limited to the enamel surface; **(f)** 0.5 mm high Tygon tubes are prepared with a simple utility knife assembly; **(g)** Tygon tubes were placed onto holes on the adhesive tape by using a 2.5x magnification loop. Flowable composite filled spaces between tubes to ensure better fixation of the Tygon tubes; **(h)** the finished, composite buttons are seen in the figure. Adhesive and composite placement took an estimated 40-60 minutes for a micro-shear sample of a single tooth; **(i)** the prepared micro-shear test sample was fixed to the test device as shown in the figure. Shear force was applied to each sample by knife at a speed of 1 mm / min from the closest possible location of the resin-mine until the sample failed.



PAPER

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