



## The effects of various mouth rinses on enamel bond strength of a universal adhesive system

Uticaj različitih sredstava za ispiranje usta na jačinu veze gleđi sa univerzalnim adhezivnim sistemom

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### Abstract

**Background/Aim.** Although essential oil-containing mouth rinses have some advantages, it is not well-known what effects they have on the bond strength of the universal resin adhesives system to the enamel. The aim of the study was to evaluate the effect of essential oil-containing mouth rinses on the enamel bond strength of the universal adhesive system. **Methods.** A total of 96 bovine incisors were used in the study. The teeth were divided into four different groups according to the control group and three different mouth rinses: Group I (Control) – distilled water, Group II – Listerine Cool Mint (essential oil mouth rinse), Group III – Kloroben (0.12% chlorhexidine gluconate mouth rinse), and Group IV – Oxyfresh (0.05% sodium fluoride mouth rinse). Each group was divided into two subgroups according to the application mode of the universal adhesive (etch-and-rinse mode or self-etch mode) ( $n = 12$ ). Mouth rinses were applied daily for 30 sec to the enamel surfaces for a month, and the samples were soaked in distilled water. After the shear bond strength (SBS) tests were performed with the universal test machine at a speed of 1 mm/min, the SBS data were statistically analyzed ( $p = 0.05$ ). **Results.** Two-way ANOVA showed that the enamel bond strength of universal adhesive was not affected by mouth rinse and was significantly affected by the application mode. **Conclusion.** The use of essential oil-containing mouth rinses and other mouth rinses tested in the study is safe in terms of the quality of enamel bonding of the tested adhesive.

**Key words:**  
adhesives; dental enamel; essential oils;  
mouthwashes.

### Apstrakt

**Uvod/Cilj.** Iako sredstva za ispiranje usta koja sadrže eterična ulja imaju određene prednosti, nije dovoljno poznato kakav efekat ona imaju na jačinu veze gleđi sa univerzalnim adhezivnim sistemom. Cilj rada bio je da se proceni uticaj sredstava za ispiranje usta koja sadrže esencijalno ulje na jačinu veze gleđi sa univerzalnim adhezivnim sistemom. **Metode.** U istraživanju je korišćeno 96 goveđih sekutića. Zubi su podeljeni u četiri različite grupe prema kontrolnoj grupi i tri različita sredstva za ispiranje usta: Grupa I (Kontrolna) – destilovana voda, Grupa II – *Listerine Cool Mint* (sredstvo za ispiranje usta sa esencijalnim uljem), Grupa III – Kloroben (0,12% hlorheksidin glukonat u sredstvu za ispiranje usta) i Grupa IV – *Oxyfresh* (0,05% natrijum-fluorid u sredstvu za ispiranje usta). Prema načinu nanošenja univerzalnog lepka adheziva (protokol nagrizanja i ispiranja ili protokol samonagrizanja), svaka grupa je podeljena u dve podgrupe ( $n = 12$ ). Sredstva za ispiranje usta svakodnevno su se nanosila u trajanju od 30 sekundi na površine gleđi tokom mesec dana, a uzorci su zatim potapani u destilovanu vodu. Nakon ispitivanja „čvrstoće smicanja“, primenom univerzalne ispitne test mašine, pri brzini od 1 mm/min, dobijeni podaci su statistički analizirani ( $p = 0,05$ ). **Rezultati.** Dvosmernim ANOVA testom pokazano je da ispiranje usta nije uticalo na jačinu veze gleđi sa univerzalnim adhezivom, ali je zato način nanošenja značajno uticao. **Zaključak.** Korišćenje sredstva za ispiranje usta koje sadrži esencijalno ulje, kao i drugih sredstava za ispiranje usta testiranih u ovom istraživanju, pokazalo se bezbedno u pogledu kvaliteta veze gleđi i ispitivanog adheziva.

**Ključne reči:**  
adhezivi; zub, gleđ; ulja, etarska; usta, sredstva za ispiranje.

## Introduction

One of the most common infectious oral diseases in humans is dental caries, whose primary etiology is dental plaque<sup>1</sup>. The basis of the prevention of dental caries is the mechanical removal of dental plaque. However, using anti-septic mouth rinses to make dental plaque less cariogenic is recommended in order to prevent dental caries disease since it is almost impossible to remove dental plaque, and the reformation of plaque is inevitable<sup>2,3</sup>.

Among the many different chemical agents found in mouth rinses, the most recommended ones are those mouth rinses containing fluorides, chlorhexidine, and essential oils<sup>4</sup>. In clinical studies, mouth rinses containing fluoride or chlorhexidine compounds have been found to help control the progression of caries lesions<sup>2,5</sup>. Furthermore, mouth rinses containing essential oils have been shown to reduce dental plaque formation and gingival inflammation in long- and short-term clinical studies<sup>6-8</sup>. The primary mechanism underlying the clinical effect of this mouth rinse is thought to be microbiocidal. It has been shown in *in vitro* studies that this mouth rinse is capable of killing a wide variety of microorganisms in a short time<sup>9</sup>. It can also reduce bacterial load, slow plaque maturation, and reduce the amount and pathogenicity of plaque<sup>10</sup>. Evidence from clinical studies indicates that chlorhexidine and fluoride mouth rinses, along with essential oils, may have contributed to the prevention of tooth decay disease.

Today, significant improvements in the clinical success of resin adhesive systems play an essential role in the more frequent use of tooth-colored aesthetic direct restorations<sup>11</sup>. Universal adhesive systems constitute the latest resin adhesive class introduced to the market. These adhesives have

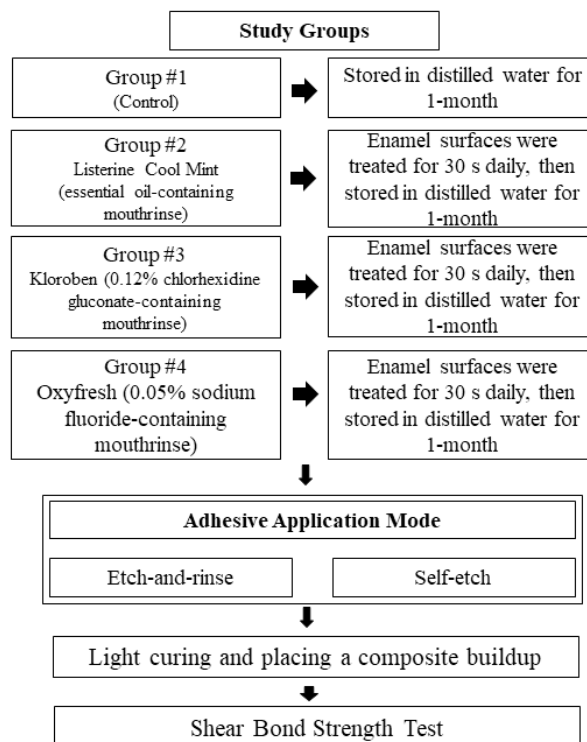
been developed to overcome the shortcomings of one-step self-etch adhesives<sup>11,12</sup>. One of the most important advantages offered by universal adhesives is the fact that the same adhesive is suitable for applying both the etch-and-rinse and the self-etch application modes. This versatility of universal adhesive systems allows clinicians to choose the ideal approach based on the condition of the cavity.

Although essential oil-containing mouth rinses have some advantages, it is not well-known what effects they have on the bond strength of the universal resin adhesives system to the enamel. In addition, there is not enough evidence in the literature about the effect of other mouth rinses containing fluoride or chlorhexidine on resin-enamel bonding. Therefore, the aim of the study was to evaluate the effects of mouth rinses containing fluoride or chlorhexidine or essential oil on the enamel bond strength of the universal resin adhesive system with the shear bond strength (SBS) test.

## Methods

### Study design

The independent variables of the study were as follows: mouth rinse Listerine Cool Mint (essential oil-containing mouth rinse), Johnson & Johnson, New Jersey, USA; Kloroben (0.12% chlorhexidine gluconate-containing mouth rinse), Drogosan Pharmaceuticals, Ankara, Turkey; Oxyfresh (0.05% sodium fluoride-containing mouth rinse), Oxyfresh Inc., Idaho, USA; application mode of the universal adhesive (etch-and-rinse, self-etch). The dependent variable was the enamel bond strength. The schematic presentation of the study design is shown in Figure 1. The details of the materials deployed in the present study are shown in Table 1.



**Fig. 1 – Schematic presentation of the study design.**

**Table 1**

<b>Materials used in the study</b>	
Material	Chemical composition
Single Bond Universal, 3M, Deutschland GmbH, Neuss, Germany	10-MDP, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, and silane.
Ultra-Etch (Ultradent)	35% phosphoric acid, water, cobalt aluminate blue spinel, glycol, and siloxane.
Dynamic Plus Universal Hybrid Composite, President Dental GmbH, München, Germany	Bis-GMA, TEGDMA, barium aluminosilicate and fumed silica fillers.
Listerine Cool Mint (essential oil-containing mouth rinse), Johnson & Johnson, New Jersey, USA	Thymol, eucalyptol, methyl salicylate, menthol, water, sorbitol solution, alcohol (30%), poloxamer 407, benzoic acid, mint and mint essences, sodium saccharin, sodium benzoate, green dye 3.
Kloroben (0.12% chlorhexidine gluconate-containing mouth rinse), Drogosan Pharmaceuticals, Ankara, Turkey	Chlorhexidine gluconate 0.12%, water, glycerin, ethanol, polysorbate 20, mint flavor aromatic composition, sodium saccharinate, FD & C, Blue 1.
Oxyfresh (0.05% sodium fluoride-containing mouth rinse), Oxyfresh Inc., Idaho, USA	Sodium fluoride, cetylpyridinium chloride, water, glycerin, propylene glycol, sorbitol, poloxamer 407, sodium chloride, potassium sorbate, sodium saccharin, citric acid, green dye, yellow dye.

**10-MDP-10 – methacryloyloxydecyl dihydrogen phosphate; HEMA – 2-hydroxyethyl methacrylate; Bis-GMA – bisphenol A-glycidyl methacrylate; TEGDMA – triethylene glycol dimethacrylate.**

#### *Specimen preparation*

The present *in vitro* study was conducted at Usak University. All operations were performed by the same person. Approval from the Ethics Committee was not obtained as no human material was used in this study. In this study, 96 caries-free bovine incisor teeth were used. After removing all the soft tissues from the teeth with a scalar, the teeth were kept in 0.5% Chloramine-T at room temperature until they were used. For the preparation of flat enamel surfaces, individual teeth were embedded in acrylic resin with a silicone mold. After the acrylic resin was cured, the enamel surfaces were smoothed under water cooling with 400-grit silicon carbide (SiC) abrasive paper. The prepared teeth were then randomly distributed into four main groups of 24 teeth each according to the mouth rinse: Group I (Control – no mouth rinse was applied to the samples in the control group); Group II (essential oil-containing mouth rinse was applied to enamel surfaces of samples for 30 sec every day); Group III (0.12% chlorhexidine gluconate-containing mouth rinse was applied to the enamel surfaces of samples for 30 sec every day); Group IV (0.05% sodium fluoride-containing mouth rinse was applied to enamel surfaces of samples for 30 sec every day). Mouth rinses were applied to the prepared enamel surfaces daily for one month before bonding. The samples were kept in distilled water for a month. Storage solutions were changed weekly.

#### *Shear bond strength testing*

Before the application of the adhesive resin system, the enamel surfaces were polished with 600-grit SiC abrasive paper under water cooling in order to obtain clinically relevant and standardized smear layers on enamel surfaces. Samples in each group were randomly divided into two subgroups ( $n = 12$ ) according to the universal adhesive application mode – the etch-and-rinse mode and the self-etch

mode. For the etch-and-rinse mode, before applying the universal adhesive, the enamel surface was etched with 37% phosphoric acid gel for 15 sec, and the acid was rinsed for 30 sec and dried. Universal adhesive (Single Bond Universal, 3M Deutschland GmbH, Neuss, Germany) was applied to the acid-etched enamel surface according to the application instructions of the manufacturer. The adhesive was applied for 20 sec with active agitation and dried for 5 sec with gentle air pressure. The adhesive was polymerized with a LED light curing device (1,200 mW/cm<sup>2</sup>, Elipar S10; 3M Unitek, Monrovia, CA, USA) for 10 sec. For the self-etch mode, the enamel surfaces were not pre-etched with phosphoric acid, and the universal adhesive was applied to the enamel surfaces, as previously explained.

After the adhesive application steps, the resin composite buildups were made using a silicone mold with a height of 4 mm and an internal diameter of 2 mm. Each layer of resin composite was polymerized for 20 sec with the same LED light-curing device. Bonded samples were kept in distilled water for 24 hrs and then subjected to an SBS test. The SBS tests were performed by the Instron universal testing machine (Instron 3220, Instron Corporation, Canton, MA) with a crosshead speed of 1 mm/min. The SBS was expressed as megapascals (MPa) by dividing the maximum force value (Newton) by the bonding area (mm<sup>2</sup>).

After the SBS test, the debonded surfaces were evaluated under x20 magnification with a stereomicroscope (Meade Bresser Biolux, Meade Bresser, Rhede, Germany), and failure modes were determined. Failure modes were classified as follows: (1) “adhesive failure” – if the debonding occurred in more than 80% of adhesive; (2) “cohesive failure” – if the debonding occurred in one of the substrates (enamel or resin composite) in more than 80% of adhesive; (3) “mixed failure” – with a combination of adhesive and cohesive failure.

Statistical analysis

A two-way Analysis of Variance (ANOVA) and Tukey’s HSD test ( $p = 0.05$ ) were used in analyzing SBS data. Factors were mouth rinse (Listerine Cool Mint/Chlorhexidine mouth rinse/fluoride mouth rinse) and adhesive application mode (etch-and-rinse/self-etch). A two-way ANOVA, along with Tukey’s HSD test, was applied together with each application mode ( $p = 0.05$ ). All statistics were made with the SPSS version 12 software (SPSS, Chicago, IL, USA).

Results

The SBS mean values and standard deviations, failure mode distributions for adhesive application modes, and mouth rinses were summarized in Table 2. Likewise, a bar graph shows the SBS of the mouth rinses groups concerning adhe-

sive application modes in Figure 2. Two-way ANOVA revealed that there was no statistically significant interaction between the effects of mouth rinse and adhesive application mode on SBS ( $p = 0.971$ ). However, it showed that only adhesive application mode significantly affected SBS ( $p < 0.05$ ) and not mouth rinse ( $p = 0.434$ ). Application of universal adhesive in etch-and-rinse mode showed significantly higher SBS than those in self-etch mode regardless of mouth rinse. Predominance failure modes were cohesive and mix failures for all etch-and-rinse groups, while predominance failure modes were adhesive and mix failures for all self-etch groups.

Discussion

Antibacterial mouth rinses are mostly used by patients with high caries risk in order to reduce the cariogenicity of dental plaque since it is difficult to completely clean the dental plaque, which is the primary factor of dental caries, and

Table 2

Shear bond strength (SBS) and distribution of failure types for all groups (n = 12)

Groups	Adhesive application mode			
	etch-and-rinse		self-etch	
	SBS	failure mode	SBS	failure mode
I	33.79 ± 5.9 <sup>aA</sup>	C > M > A	23.85 ± 7.4 <sup>aA</sup>	A > M > C
II	32.53 ± 4.3 <sup>aA</sup>	C > M > A	21.87 ± 5.9 <sup>aB</sup>	A > M > C
III	37.51 ± 5.8 <sup>aA</sup>	C > M = A	27.27 ± 6.1 <sup>aB</sup>	A = M > C
IV	35.52 ± 5.2 <sup>aA</sup>	C > M > A	21.38 ± 5.4 <sup>aB</sup>	A > M > C

A – adhesive failure; M – mixed failure; C – cohesive failure. Group I – distilled water; Group II – Listerine Cool Mint (essential oil mouth rinse); Group III – Kloroben (0.12 chlorhexidine gluconate mouth rinse); Group IV – Oxyfresh (0.05% sodium fluoride mouth rinse). Results of SBS test are given as mean ± standard deviation. Different lowercase superscripts represent the significant difference in the same column ( $p < 0.05$ ). Different uppercase superscripts represent the significant difference in the same row ( $p < 0.05$ ).

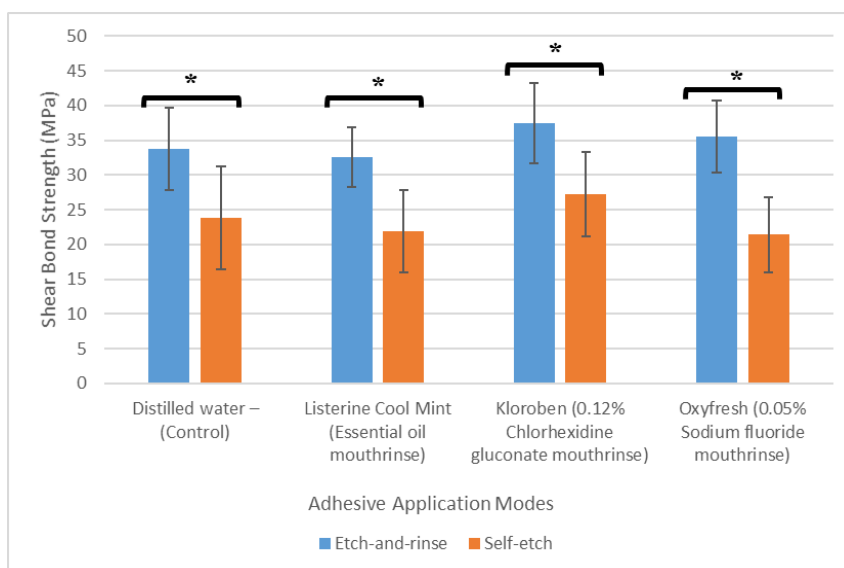


Fig. 2 – Bar graph depicting shear bond strength of the tested groups concerning adhesive application modes.

Values are expressed in megapascals (MPa).

\*Statistically significant difference between groups analyzed using two-way ANOVA ( $p < 0.05$ ).

prevent its recurrence. However, there is not enough information in the literature about how mouth rinses affect the enamel bonding of universal adhesives. Therefore, the present study evaluated the effects of three different mouth rinses, including an essential oil-containing mouth rinse, on the enamel bond strength of a universal adhesive applied in different modes.

Bovine teeth were used instead of human teeth in the SBS test in the present study. It has been shown that bovine teeth can replace human teeth in both enamel and dentin bond strength tests<sup>13</sup>. It has been reported that bovine teeth have a mineral distribution similar to human enamel<sup>14</sup>. One advantage of using bovine teeth is obtaining large numbers of teeth from animals of similar age groups and with similar diets in a short time, which enables the age and chemical structures of the teeth to be standardized. Because of these advantages, bovine teeth were preferred in the present study as an alternative to human teeth.

In the present study, it was noticed that enamel bonding was significantly affected by the application mode of the adhesive regardless of mouth rinses. Since the universal adhesive system used in this study is an adhesive system – Single Bond Universal (3M ESPE, USA), which has been used in many studies in the literature – it was deliberately chosen in order to understand whether the findings of the present study correlated with the literature. In previous studies, it has been reported that the application of Single Bond Universal adhesive to enamel in the etch-and-rinse mode provides significantly higher bonding strengths compared to the self-etch mode<sup>15–17</sup>. In this respect, the findings of the present study are compatible with the literature.

Etching the enamel surface with a separate acid agent before the application of the universal adhesive, in other words, applying the universal adhesive in the etch-and-rinse mode, has been shown to increase the enamel bonding due to the increase in the porosity on the enamel surface and the infiltration of the resin into these porosities<sup>15</sup>. During the acid etching process, there is a loss of material at a depth of 10 µm from the enamel surface, and a porous layer of 5–50 µm is formed<sup>15, 18</sup>. Polymerization of resin after infiltrating into this porous structure allows the composite to adhere micro-mechanically to the enamel surface.

The two-way ANOVA revealed no significant interaction between factors types of mouth rinse and adhesive application modes. That may explain the findings of the study. It was observed that the application of mouth rinses tested on enamel surfaces for 30 sec daily for a month did not affect the enamel bonding even when applying the universal adhe-

sive used in the study with two different application modes. In previous studies, it has been reported that mouth rinses containing fluoride cause the formation of fluorapatite crystals on the enamel surface and increase the number of minerals on the enamel surface<sup>19</sup>. Similar to the findings in our study, it was reported in the study of Elzuhery et al.<sup>19</sup> that the fluoride-containing mouth rinse did not affect the enamel bonding of the adhesive systems. Although the mouth rinse containing fluoride increases the number of minerals on the enamel surface, this layer is removed by grinding during the smear layer formation process before the application of the adhesive. Therefore, the presence of this mineral-dense layer may not have affected the enamel bonding of the adhesive in both application modes.

In the failure type analysis, cohesive failure type was predominant in etch-and-rinse groups regardless of mouth rinse. However, adhesive and mix failure types were predominant in self-etch groups regardless of mouth rinse. It can be anticipated that cohesive would correspond to higher SBS values in etch-and-rinse groups or *vice versa* in self-etch groups<sup>20</sup>.

Similarly, the reason why the bonding of the tested adhesive was not affected by chlorhexidine and Listerine-containing mouth rinses might be because the layers on the enamel surface affected by these mouth rinses were removed during the grinding prior to adhesive bonding. A study supporting this finding was conducted by Demir et al.<sup>21</sup>, where it was reported that the mouth rinses containing chlorhexidine gluconate applied daily for 30 sec for two weeks did not affect the enamel bonding of the orthodontic composite.

## Conclusion

Pre-etching of the enamel surface with phosphoric acid before the application of universal adhesive to enamel surfaces on which mouth rinses containing essential oil, chlorhexidine, or fluoride have been applied can significantly increase the bonding strength of the adhesive. On the other hand, it was determined that the mouth rinses tested in the study did not affect the adhesion of the universal adhesive to the enamel negatively in the self-etch mode. For this reason, no waiting period may be required to ensure the bonding quality of the resin-enamel interface in patients using mouth rinses tested in this study prior to the treatment of dental caries using resin adhesive systems.

## Conflict of interest

The author declares no conflict of interest.

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