Original Article

Evaluation and Comparison of Newly Developed Phosphoric Acid Gel (Exir) with Two Different Common Gels in Iran

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Abstract

Introduction: Different surface treatments were preposed to prepare the tooth structure for resin composite bonding. One of the most common preparation is using of phosphoric acid etching. This study conducted to determine the etching effectiveness of a new formulated acid etchant (Exir) and compared it with two commercial products. **Methods:** To determine the physical properties, the viscosity and shelf life were evaluated by a viscometer and a centrifuge over the time, respectively. SEM micrographs were uesd to show the geometry of bovine enamel and dentin surfaces which were treated by three etchants. The µSBS of composite to treated dental tissues was also investigated. Data were analyzed by ANOVA and Tukey test (α=0.05). **Results:** Exir etchant showed the lower viscosity compared with two other gels and two-phase separation did not significantly increase during suggested period. The SEM images of enamel samples treated with Exir etchant showed regular etching patterns and less remnant in comparison with other groups. The SEM mages of dentin samples treated with Exir etchant, unlike others, showed wide open dentin tubules with clear exposed collagen fibers. Enamel and dentin µSBS were 30.08±6.79 and 7.29±1.27 for Exir etchant, 23.31±6.64 and 8.49±3.61 for Morva Etch and 23.22±7.05 and 6.16±1.68 for Ultra-Etch, respectivley. The µSBS values for enamel was significantly higher in Exir treated group (P=0.017), while there was no significant difference in dentin µSBS values among three tested etchants (P=0.07). **Conclusion:** The results of this study showed Exir etchant can provide acceptable results used on dentin and enamel substrates.

Keywords: Phosphoric acid, Acid Etching, Micro-shear bond strength, Etch pattern.

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Introduction

Since teeth and smile are important factors in aesthetics, they more more considered by the patients. Anterior restorations should be mimic shape and color of natural teeth. Currently composites are one of the best materials in terms of esthetic and stability (1). In spite of noticeable achievements in production of composite resins in recent years, polymerization contraction and weak bond are still considered to be the main weaknesses of these tooth-colored restorations (2).

In order to create a strong bond between composite resin and tooth surface, it is necessary to prepare tooth surfaces first (3). Acid etching method was first introduced by Buonocore in 1955 as a standard method of preparing enamel surface for micromechanical bond to composite restorations (4). In this method, mineral acids are used to make enamel surface rough to increase contact and adhesion between tooth and composite resins (5, 6).

Initially, acid etching of dentin surface was doubtful since it was believed that the acid etchant could induce pulp inflammation. However, recent studies has shown this process is safe and an improvement in dentin bond strength was achieved when the smear layer was removed by the etchants (5). Currently, most acid etchants consist of 30-40% phosphoric acid, which are used for 15-30 seconds for dentin and enamel, respectively (7).

The formulation of phosphoric acid as an etchant is not a complicated procedure, while its additives may affect practical properties and final bond strength. Since the ingredients of the common available etchants are different, they showed different etching patterns and adhesion properties in laboratory setting (8). The favorable bond strength of resin materials to tooth structure may be affected by the performance of the acid etchant.Therefore, the present study aimed to introduce and investigate a new phosphoric acid gel (Exir), determine its efficacy, and compare it with two common etching gels (Ultra-Etch and Morva Etch) available in dental market, in Iran. The null hypothesis is that there is no difference in effectiveness of the newly-introduced phosphoric acid gel and two evlauated gels.

Materials and Methods

The evaluation of the new gel (Exir) consisted of three phases as explained below.

1. Assessment of physical properties of the gel:

    A. Viscosity: Flow rate in shear speed range of 1-85 per second was measured using SC4-27 spindle rotational viscometer (model: RV DV III Ultra, Brookfield, USA). Temperature of the sample was also kept constantly at 35˚ C during this procedure using a bath circulator (Julabo, USA) between two cylindrical walls.

    B. Stability: Stability of the gel at ambient temperature was determined using EBA 20 centrifuge (Hettich, Germany) with a rotor diameter of 86 mm over 6 weeks. For this purpose, 5 ml of gel sample was poured into the Falcon tube and centrifuged with an acceleration of 1800 g for 10 min. Finally, stability was measured based on the separated phases.

2. Geometric assessment of etching through scanning electron microscope (SEM) micrographs

To study the etching pattern of enamel and dentin, six healthy and non-cracked bovine incisors were selected and kept in a 0.2% thymol solution until the study initiation. The crowns were separated from roots by a diamond disk (D&G, Germany) and divided equally into two groups of three teeth to assess enamel and dentin surfaces. For enamel surface evaluations, the labial surface of all teeth were thoroughly cleaned with a slurry of pumice and brushed. Then, they were grounded with a medium diamond disk (D&G, Germany) and 600-800 grit sandpapers (Matador, Germany). Afterward, the etchants containing Ultra-Etch (Ultradent, South Jordan, UT, USA) and Morva Etch (Morvabon, Iran) as well as the new etchant (Exir, Iran) were applied to the enamel surfaces for 30 seconds.

To obtain dentin surfaces, labial surfaces of other three bovine incisors were trimmed to remove the enamel layer. Then, they were sanded with 600-800 grit sandpaper (Matador, Germany). The surface of each tooth was then covered with one of the gels for 15 seconds.

After the etching treatment, the sample surfaces were rinsed with copious amount of water for 20 seconds and then they were dried for 15 seconds with a air spray.

For microscopic evaluation samples were submerged. They were then covered with gold for 60 seconds at 18 mAh by a sputter coater. Afterward, they were prepared for being tested by SEM (model: LEO 1450VP, Zeiss, Ramsey, New Jersey, USA) under 10 kV voltage using a secondary electron detector to study morphology of the surface.

3. Bond strength investigation

In total, 72 bovine incisors were collected to determine the micro-shear bond strength (µSBS) of composite resin to the surfaces treated with different etchants. The samples for this study section were removed from 0.2% thymol solution before starting the study The roots were separated from the crown and then the cowns were mounted in self-cure acrylic resin (Acropars acrylic powder, Marlic Medical Ind., Iran) and kept in distilled water and ambient temperature until bonding procedure.

Teeth were randomly divided into two groups of dentin and enamel surfaces. Buccal surfaces of teeth in enamel group were ground with silicon carbide discs to create smooth surfaces and remove the fluoride-riched layer, without exposing the dentin. Later, grounded surfaces were polished using 600-800 grit paper discs (Matador, Germany).

Buccal surfaces of the teeth in dentin group were trimmed to expose dentin. Trimmed surfaces were then polished using 600-800 grit paper discs (Matador, Germany). After preparation, samples of each group were divided into three subgroups based on type of etchant, and following steps were performed for each of the samples, respectively. Manufacture and application mode of acid etchants, adhesive system and composite resin used in the study are mentioned in Table I.

Tables I. Manufacture, application mode of acid etchants, adhesive system and composite resin used in the study.

Material	Manufacture	Application Mode
Morva etch (37% Phosphoric acid)	Morvabon, Iran	For all three etchants: Etch 30 seconds on enamel and 15 seconds on dentin. Rinse for 15 seconds and then dry gently.
Ultra etch (35% Phosphoric acid)	Ultradent, South Jordan, UT, USA	
New etchant (Exir)	Exir, Iran	
Amelogen Plus resin composite	Ultradent, South Jordan, UT, USA	Place in layers with a maximum thickness of 2 mm and light cure for at least 20 seconds.
Adper Single Bond 2	3M ESPE, St. Paul, MN, USA	Apply two consecutive coats. Dry gently for 5 seconds. Light cure for 20 seconds.

1. Each subgroup was etched using one of the Ultra-Etch (Ultradent, South Jordan, UT, USA), Morva Etch (Morvabon, Iran), and the new etchant (Exir, Iran) gels. Enamel and dentin samples were etched for 30 and 15 sec, respectively.

2. Surfaces were rinsed and air dried.

3. The dentin bonding agent (Adper Single Bond 2, 3M ESPE, St. Paul, MN, USA) was applied in two consecutive layers on surface of the samples, according to the manufacturer instructions. Then bonding was cured using LED curing light (Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein) for 20 seconds.

4. Transparent plastic tubes (with inner diameter of 1.15 mm and length of 3 mm) were placed on tooth surfaces and the composite resin shade A1 (Amelogen Plus, Ultradent, South Jordan, UT, USA) was packed into the plastic mold using a small hand instrument and was light cured from different angels for 40 seconds.

5. The bonded samples were kept in incubator for 24 h at 37˚ C to complete polymerization of composite resin.

6. The Plastic molds were removed from the periphery of the resin composite cylinders using a sharp surgical blade. Samples were then aged for 3000 cycles in a thermocycling machine (Nemo, Iran) to simulate the thermal stress. For this purpose, two separate warm (54 to 58 °C) and cold (5 to 10 °C) water baths were used and the samples were kept in each water bath for 30 seconds.

7. At the end of the thermal cycles, the bonded samples were subjected to shear forces using a Universal Testing Machine (SANTAM, Tehran, Iran). A wedge-shape blade was used, which exerted a force on tooth interface and composite resin at a crosshead speed of 1 mm per min. The force (N) at failure point was recorded and shear bond strength values (MPa) were calculated.

Statistical analysis

Data description was performed using appropriate statistical tables and graphs. Moreover, the collected quantitative data were analyzed using one-way ANOVA and Tukey post hoc test was used for multiple comparisons of mean values. For statistical analysis, P-value of 5% was considered statistically significant.

Results

1. Physical properties of the gel

A) Viscosity: All evaluated acid etchants had a shear thinning behavior and their viscosity gradually decreased with increasing shear velocity (Figure 1). The viscosity of tested gels was recorded at 35˚ C with a shear rate of 21 per second and presented in Figure 2.

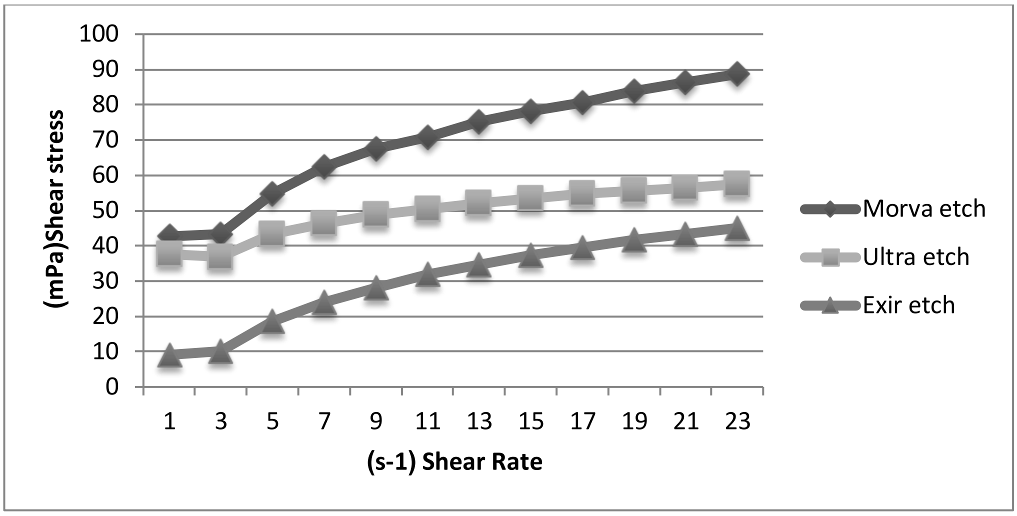

Figure 1: Shear stresses of the evaluated acids at different shear rates.

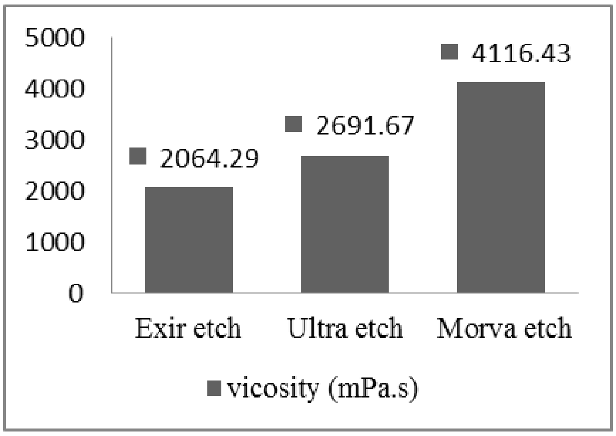

Figure 2: Viscosity of the evaluated acid etchants at a shear rate of 21 per second.

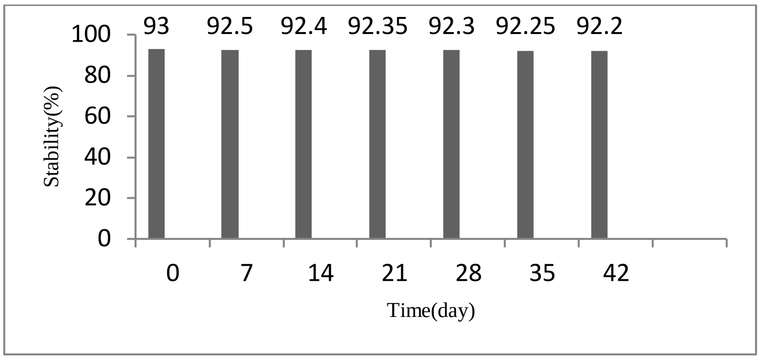

Figure 3. Stability of the Exir acid etchant.

B) Stability: The obtained results of the stability test in a period of 42 days are shown in Figure 3. Data was obtained after the test was repeated for two times. Over time, two-phase separation did not increase which means the gel has a good stability.

2. Geometric assessment of etching through SEM microscopy

Enamel etching pattern

The samples prepared with Exir acid etchant: The SEM micrographs showed third types of enamel etching patterns (Figure 4. A). At 5,000 magnification, in spite of regular etching patterns, the deposites of salts covered the surface which may be resulted from the reaction of the etchant with mineral components of enamel (Figure 4.B). They were more obvious in 10000 magnification (Figure 4.C). Totally, this etchant could produce the regular structure and clear etching patterns of enamel samples.

The samples prepared with Morva Etch: For this etchant, type two of the etching pattern was predominant in which most of the minerals were removed from periphery of the enamel prisms. Similar to the study group that was prepared by Exir etchant, the residues of etchant ingredients were observed on the surface. The regular pattern which was found after using of the Exir etchant was not observed in this group (Figure 4.D, E, F).

The samples prepared with Ultra-Etch: In this group, a clear pattern of three type was seen. Also, a large amounts of dissolved salt were visible on the surfaces (Figure 4.G, H, I).

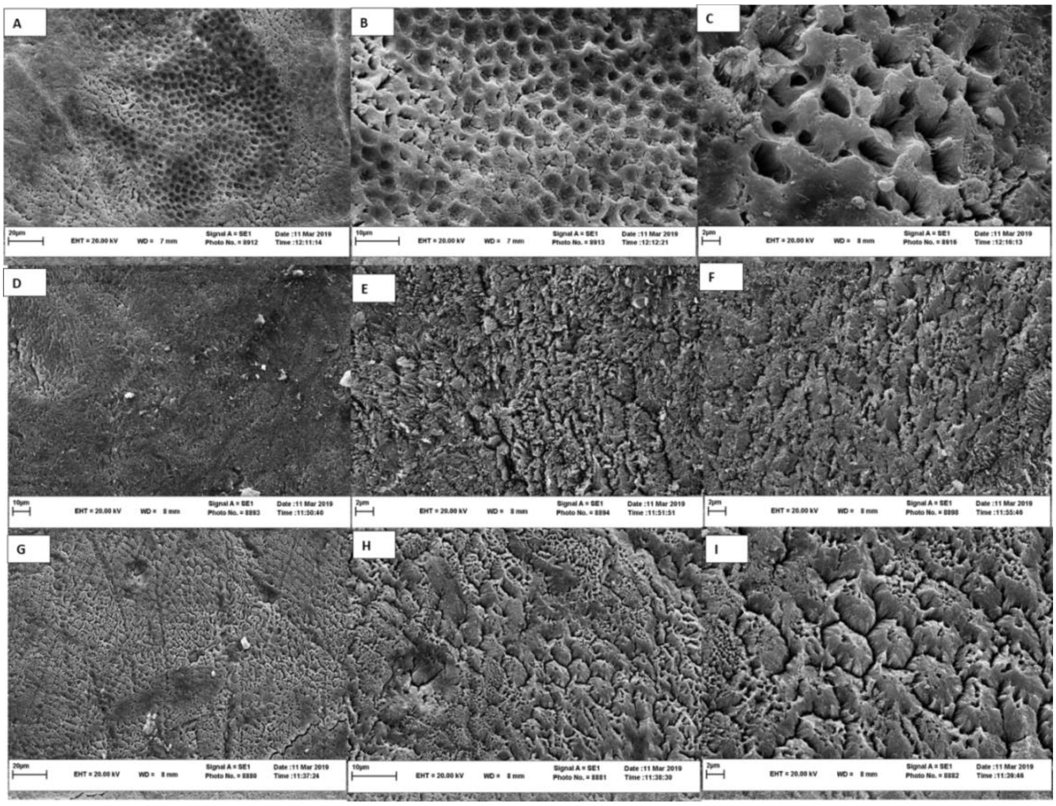

Figure 4. Enamel etching pattern resulted from Exir etchant treatment (A: at 2,000 magnification, B: at 5,000 magnification, C: at 10,000 magnification), enamel etching pattern resulted from Morva Etch treatment (D: at 2,000 magnification, E: at 5,000 magnification, F: at 10,000 magnification), enamel etching pattern resulted from the application of Ultra-Etch (G: at 2,000 magnification, H: at 5,000 magnification, I: at 10,000 magnification)

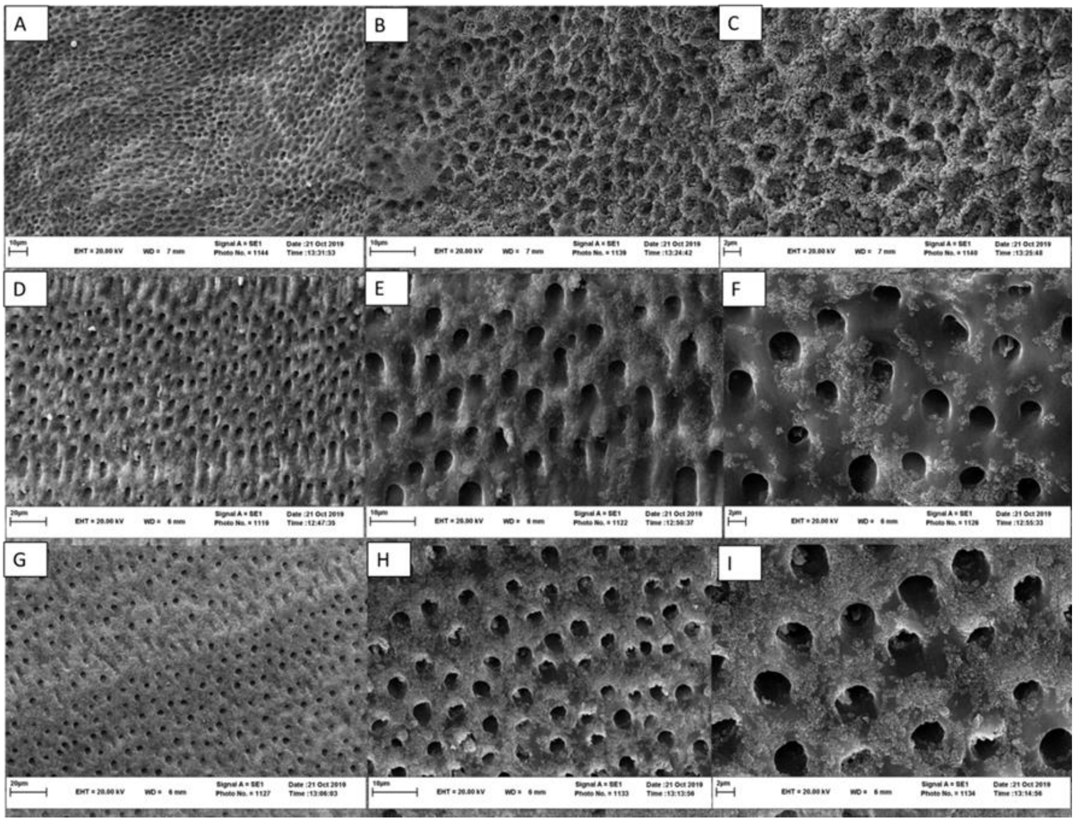

Figure 5. Dentin Etching Pattern resulted from Exir etchant treatment (A: at 2,000 magnification, B: at 5,000 magnification, C: at 10,000 magnification), dentin etching pattern resulted from Morva etch treatment (D: at 2,000 magnification, E: at 5,000 magnification, F: at 10,000 magnification), dentin etching pattern resulted from the application of Ultra Etch (G: at 2,000 magnification, H: at 5,000 magnification, I: at 10,000 magnification)

Dentin etching pattern

The samples prepared with the Exir etchant: In this samples, most of the minerals were removed from the dentin tubules and tubular wall and left them completely opened. At 10000 magnification, the network of exposed collagen fibers without minerals can be seen, which shows optimal performance of Exir acid etchant (Figure 5).

The samples prepared with Morva Etch: In SEM images of this group, only entrance of dentin tubules is visible. The smear plugs appear to be removed as a result of etching of this etchant, while this was unable to remove the minerals from the dentinal walls. At 10000 magnification, the remnants of the etchan ingredients was observed at inter-tubular distances (Figure 5).

The samples prepared with Ultra-Etch: The eching pattern of Ultra-Etch was similar to Exir gel. The SEM images showed the open entrance of dentin tubules with residues of dissolved salts and constituent materials of the etchant gel. At higher magnifications (5K and 10K), the inability of etchant gel to remove smear plugs and residues from dentin tubules can be observed (Figure 5).

3. Bond strength

The µSBS of resin composite to enamel which were prepared using three acid etchants are presented in Table

II. Based on Kolmogorov–Smirnov test, data showed normal distribution. The mean values of μSBS of composite to enamel treated with the Exir etchant, Morva Etch, and Ultra-Etch were 30.08±6.79, 23.31±6.64, and 23.22±7.05 MPa, respectively. Therefore, significant differences were observed between three groups in terms of enamel μSBS (P=0.017). According to Tukey's post hoc test, there was a significant difference between mean values of Exir and Morva Etch (P=0.035) groups as well as that of Exir and Ultra-Etch groups (P=0.031). However, difference between mean values of Ultra-Etch and Morva Etch groups was not significant (P=0.998).

Tables II. Comparison of micro-shear bond strength of enamel samples treated with three acid etchants

Etchant type	Number	Mean (SD)	Min	Max	ANOVA results	
					F	P-value
Exir etch	12	30.08(6.79)[a]	22.98	47.24	4.63	0.017
Morva etch	12	23.31(6.64)[b]	15.88	37.26		
Ultra etch	12	23.22(7.05)[b]	17.02	39.43		

Different letters indicate a significant difference between the etchants.

Descriptive information on μSBS of composite to dentin etched with three etchants is shown in Table III. Based on Kolmogorov–Smirnov test, the normal distribution of data confirmed. The mean values of μSBS of composite to dentin for Exir Etch, Morva Etch and Ultra-Etch were 7.29±1.27, 8.49±3.61, and 6.16±1.68 MPa, respectively. The results regarding μSBS of composite to the dentin showed that there was no significant difference between three tested acid etchants (P=0.07).

Table III. Comparison of micro-shear bond strength of dentin samples treated with three acid etchants

Etchant type	Number	Mean (SD)	Min	Max	ANOVA results	
					F	P-value
Exir etch	12	7.29(1.27)	5.05	9.70	2.77	0.077
Morva etch	12	8.49(3.61)	4.90	14.45		
Ultra etch	12	6.16(1.68)	4.22	9.63		

Discussion

The results of viscosity assessment revealed that Exir etchant had a lower viscosity, compared to other studied samples and it was comparable to Ultra-Etch gel. In general, it can be said that dilute liquids and gels provide a more regular and uniform etching pattern compared to concentrated or thick gels. However, viscosity of the gel should be high enough to resist sagging. In terms of stability, the two-phase separation did not increase over time which confirmed the optimal stability of the etching gel.

Comparison of SEM images of enamel samples revealed a regular structure and well-defined etching pattern in samples prepared with Exir etchant, which was different from the group prepared with Morva Etch acid etchant. The samples prepared with Ultra-Etch showed a very uniform context of third type of etching pattern. However, there were more residues of dissolved salts on surface of samples which were etched with Ultra-Etch, compared to those prepared with Exir etchant. Since these sediments have not been removed with usual washing time, they can be considered as di-calcium phosphate dihydrate, which is less soluble in clinical conditions and is caused by acid corrosion. These sediments may be prevented the resin penetration (9).

the SEM images of dentin samples ttreated with Exir gel revealed opening of dentin tubules, as well as removal of minerals from tubular wall which exposed collagen fiber network without minerals. This indicates optimal performance of Exir etchant. In contrast, in samples prepared with the other two etchants, smear layer and minerals remained on the wall and between dentin tubules, and residues of etchant material or sediments of etching were observed at inter-tubular distances and even inside the dentinal tubules.

A significant issue that was observed in SEM images was a certain regularity in patterns created by Exir etch and Ultra etch which was less in the group prepared with Morva etch. In a study by Guba and Cochran (10) it was shown that diluted solutions and gels create a more uniform etching pattern than concentrated gels. Also, they showed that diluted gels had the most distinctive etching pattern among three types of conditioners (solutions, diluted gels, and concentrated gels). In the present study, another contributing factor to difference between etching patterns can be slightly different concentration and viscosity of used gels. Therefore, since Exir etchant and Ultra-Etch had lower viscosity, they presented obvious and clear etching pattern compared with Morva Etch.

Another difference found in SEM images between groups was of the lack of residual formation in Exir treated samples compared to the other tested etchans. It seems that the greater viscosity of other two etchants prevents acid from being completely washed and makes deposites as it was observed in similar previous studies (11). Another reason for formation of deposites may be presence of thickener and impurities in chemical structure of the study etchants.

SEM images of samples etched with Ultra-Etch and Morva Etch also revealed that they were unable to remove smear plugs from inside of dentin tubules. However, Exir etchant easily absorbed minerals from inside of dentin tubules.

Similar to the present study, bovine teeth is usually used for bond strength tests (12-14). It is because of similar chemical composition to human teeth (15), almost equal calcium and phosphorus content (16), comparable acid resistance (17), and similar dentin hardness and enamel thickness (18). Furthermore, bovine teeth can be easily obtained in large amount, with the possibility to standardize size and age of the teeth. So bovine teeth can be a reliable substitute for human ones on bond strength studies of adhesive systems to both enamel and dentin substrates (19).

Bond strength of composite resin to enamel was observed in several studies to be 20-25 MPa (20, 21). Moreover, bond strength of resin to dentin was observed in several studies to be 5-15 MPa (22-24). Despite the fact that significant differences were observed between three groups of etchants in terms of enamel micro-shear bond strength (P=0.017), no significant difference was found regarding adhesion to dentin (P=0.07). The Tukey's post hoc test revealed that mean value of enamel bond strength in Exir group was significantly higher than the samples prepared with Morva Etch and Ultra-Etch. Moreover, no difference was found between Ultra-Etch and Morva Etch groups. Bonding materials with low viscosity can easily penetrate etched enamel and dentin irregularities and create short and long resin tags (1). It can be said that due to higher viscosity of Morva Etch and uncertainty of its complete removal after washing during same time with other groups, gel residues in dentin tubules or inside enamel pores may interfere with bonding agent infiltration (11). Tags causes mechanical locking in etched enamel or dentin (20). One of the possible reasons for high values of µSBS in samples prepared with Exir etchant, compared to Morva Etch could be regular patterns that were result of enamel etching. Such patterns were not observed in groups prepared with two common acids. Furthermore, less sediment was formed in dentin and enamel samples which were etched with Exir etchant, compared to those etched with other two acid phosphoric gels.

Conclusion

Examination of physical properties of Exir etchant showed acceptable results in terms of viscosity, which was comparable or even better than two other gels. Moreover, it must be noticed that Exir etchant has desirable stability during the time. In addition, short-term results of composite bonding to enamel and dentin etched with the Exir etchant revealed comparable or even better bond strength values compared with the other tested etchants. Therfore, this gel can be considered as an acceptable alternative to existing etchants in the market.

Conflicts of interest

The authors declare that they have no conflict of interest regarding the publication of the current article.

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