Original Article

Evaluation of Resin Penetration Depth in Enamel Surface for Orthodontic Bonding Exposed to Five Types of Enamel Conditioning Methods: A Scanning Electron Microscopic Study

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Abstract

Aims and Objectives: To evaluate the enamel surface depth of resin penetration for orthodontic bonding when exposed to five types of enamel conditioning methods using scanning electron microscope. **Materials and Methods:** The sample comprised of 25 human extracted premolar teeth for orthodontic reasons. The samples are divided into 5 groups with each 5 teeth based on the enamel conditioning methods such as group A with 37% Phosphoric acid, group B with 10% Polyacrylic acid, group C with Self etch primer, group D with Laser etching and group E with air abrasion etching. All the samples in each group are bonded with metal brackets using Transbond XT Adhesive. After bonding, the teeth are sectioned mesiodistally using hard-tissue microtome and observed under scanning electron microscope at 500x and 3000x for the calculation of depth of resin penetration. **Results:** The teeth etched with 37%phosphoric acid exhibited significantly greater depth of resin penetration (15.1μm) than do self-etch and polyacrylic acid. Laser etching has comparable penetration depth with that of acid etching. Air abrasion has got the least penetration of all groups. **Conclusion:** Comparing the enamel treated with these conditioning methods, the penetration of resin material into enamel are greater when it is treated with 37% Phosphoric acid than self-etch or laser etching.

Keywords: *Acid etching, depth of resin penetration, resin tags, laser etching*

Introduction

The enamel conditioning method promotes the adhesion of adhesive resin to the tooth structure by creating microporosity, increasing the surface energy, and thereby permitting the penetration of resin into the enamel as resin tags affording mechanical retention.[1]

Phosphoric acid (35–40%) is used for 30 seconds to clean the surface and dissolve the minerals. This acid etching causes dissolution of interprismatic material in the enamel, producing a roughened and porous layer.[2] Preconditioning with polyacrylic acid is recommended for resin-modified glass ionomer cement.[3] The polyacrylic acid solution that contains residual sulfate ions produces a crystalline deposit that bonds firmly to the enamel surface by forming calcium sulfate dihydrate crystals and resists mechanical removal.[4]

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Newer bonding system like self etching systems combines the etching and primer steps simultaneously. These systems eliminate the need for rinsing and possible damage to gingival tissues and save chairside time.[5] Use of self-etching primer (self etch primer [SEP]; Transbond Plus; 3M Unitek) for bonding provides similar and clinically acceptable bond strength compared to the use of 37% phosphoric acid etching technique.

Zachrisson and Büyükyılmaz[6] in 1996 promoted sandblasting as a mechanical method of retentive

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procedure. Aluminum oxide (Al_2O_3) particles of size 50 μm are sandblasted at high pressure on the tooth surface, leaving a microretentive surface for bonding. In 1997, Reisner *et al.*[7] evaluated sandblasting as a method of enamel preparation prior to bracket bonding.

Laser irradiation of dental enamel causes thermally induced changes within the enamel to a depth of 10–20 μm, depending on the type of laser and energy applied.[8] For bonding of orthodontic brackets, the carbon dioxide (CO_2) laser etching of enamel causes microcracks that are ideal for resin penetration. Water spraying and air drying are not required with laser etching.

Scanning electron microscopy (SEM) is a nondestructive examination of layers up to 100 μm below the surface and produces high-resolution images. Microphotographs were taken through SEM at from 5× to 300000× magnifications. SEM studies have shown that there are wide variations in the appearance of etched enamel surfaces.[3]

Aim

The aim of *in vitro* study was to evaluate the depth of resin penetration through SEM, when exposed to following types of enamel conditioning methods: 37% phosphoric acid, 10% polyacrylic acid, SEP, laser etching, and sandblasting.

Objectives

The objectives of this study were (1) to observe the interface between the enamel and the adhesive using SEM and (2) to calculate and compare the depth of resin penetration among different enamel conditioning methods.

Materials and Methods

Twenty-five human maxillary and mandibular first premolars, extracted for orthodontic purposes, were used in this study as samples. The samples were free of caries and restorations. The teeth were stored in distilled water after extraction. The teeth were allocated into five groups having five teeth in each group. Each tooth was mounted vertically in a self-cure acrylic resin in such a manner that the crowns were exposed.

In group A, the enamel surfaces were conditioned with 37% phosphoric acid (Scotchbond etchant, 3M ESPE, St. Paul, MN) for 30 seconds, rinsed thoroughly under running water for another 30 seconds, and dried with compressed oil-free air for 5 seconds until a frosted whitish appearance was seen on the enamel surface. In group B, the enamel surfaces were conditioned with 10% polyacrylic acid (GC Corporation, Japan) for 20 seconds, rinsed thoroughly under running water for another 30 seconds, and dried with compressed oil-free air for 5 seconds. In group C, the enamel surfaces were conditioned with SEP (Transbond Plus; 3M Unitek) for 3 seconds and air dried for 5 seconds according to the manufacturer's instructions. In group D, CO_2 laser etching (CO_2 laser unit, ULTRA 25 Plus; Union Medical, Korea) was performed at 3-W power output for 5 seconds. The laser beam was perpendicular to the tooth enamel at a distance of 1 mm. In group E, the teeth were sandblasted with Al_2O_3 powder particles of size 50 μm (Microblaster Standard; Bio-Art, Brazil) for 3 seconds at a distance of 6 mm.

After the enamel surfaces were conditioned in each group, premolar brackets were bonded to the conditioned surface according to the manufacturer's instructions with the following protocol [Figure 1].

In groups A, B, D and E, Transbond XT primer (3M Unitek) was applied as a thin film to etched enamel surface and cured for 20 seconds. Transbond XT paste (3M Unitek) was applied to the bracket (Nu Edge MBT



Figure 1: Mounted samples with bracket bond

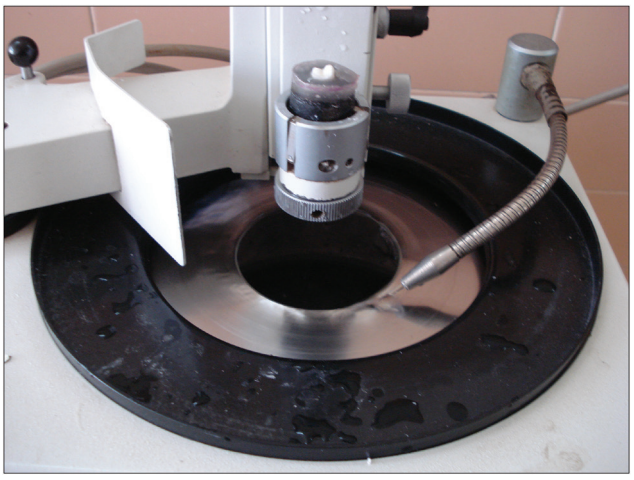


Figure 2: Hard tissue microtome (SP 1600; Leica Biosystems, Germany)

0.022-inch slot; TP Orthodontics) and pressed firmly onto the teeth. The adhesive was cured for 20 seconds by using halogen light curing unit (3M ESPE). In group C, brackets were bonded with Transbond XT paste and cured for 20 seconds.

The teeth with the brackets were sectioned buccolingually along the center of the brackets by using hard tissue microtome (SP 1600; Leica Biosystems, Germany) [Figure 2]. The microtome was set so that each section was 300-μm thick [Figure 3]. After sectioning, the enamel–adhesive interface was observed through SEM (JSM 5610 LV; Hitachi, Japan) by sputter coating the samples with gold palladium, operated at 15 kV with ×500 and ×3000 magnifications [Figure 4].

Statistical analysis

The mean and standard deviation were estimated from the samples for each variable regarding depth of resin penetration in each group. Mean values were compared between groups and assessed by Student's paired *t* test. Mean values were compared within groups by using analysis of variance (ANOVA) and *post hoc* test was conducted. A *P* value of less than 0.05 was considered to be statistically significant.



Figure 3: Sectioned sample

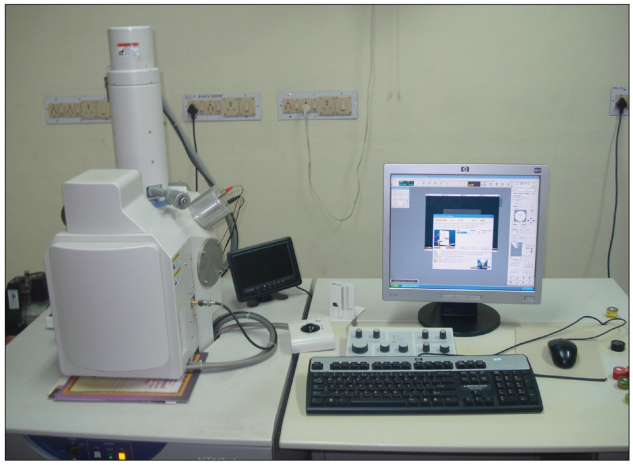


Figure 4: Scanning electron microscope (JSM 5610 LV; Hitachi, Japan)

Results

In group A, after etching with 37% phosphoric acid and bonding, the SEM images showed an interface between enamel and adhesive with many long, thick resin tags that had penetrated into enamel [Figure 5]. The hydroxyapatite dissolved by phosphoric acid produced tags and rough surfaces that provided the mechanical lock for the resin. Table 1 depicts mean depth of resin penetration for 37% phosphoric acid within each variable observed under ×500 magnification through SEM is 15.10 μm. In group B, the interface between enamel and adhesive showed less visible tags. In group C, consecutively a regular resin tag distribution was observed, which showed less magnitude of 7.9 μm when compared with phosphoric acid etching. In group D, the irradiated enamel surfaces had microcracks, which aided in the penetration of resin at 11.6 μm. In group E, the enamel surfaces of specimen exposed to Al_2O_3 powder particles showed some cavitations and presence of powder particles.

Table 1 depicts descriptive statistics including mean, standard deviations, standard errors, and maximum and minimum values of depth of penetration for all five groups. Group A (acid etch) has the greatest mean depth of resin penetration at 15.10 μm after bonding followed by group D (laser etching) at 11.6 μm, group B (polyacrylic acid) at 8.73 μm, and group C (self-etch) at 7.9 μm. Group E (air abrasion) has the lowest mean depth of penetration at 6.7 μm.

Table 2 represents ANOVA analysis conducted to determine significance. This analysis showed that there is a statistically significant difference between the groups at a *P* value of 0.001.

Table 3 shows that when a statistically significant difference was noted, the *t* test was conducted to identify which group was different at $P \leq 0.05$. *P* value between group A and B is 0.0135, group A and C is 0.0067, and group A and E is 0.0022.

On comparing the mean differences in the values of penetration depth between the groups, the difference in the mean value of group A is statistically significant compared with other groups [Table 4].

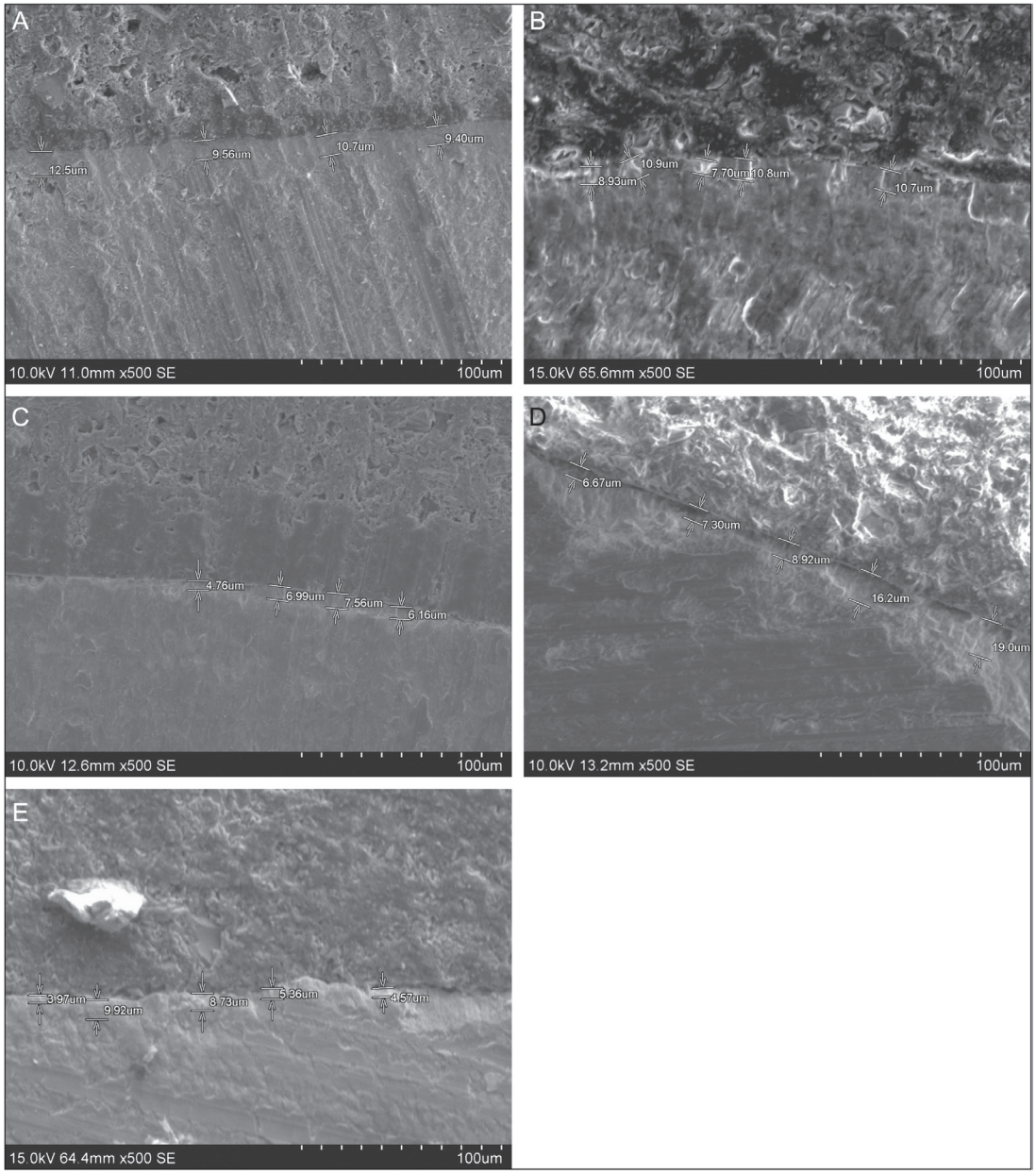


Figure 5: (A) 37% Phosphoric acid SEM image ×500, (B) 10% polyacrylic acid SEM image ×500, (C) SEP SEM image ×500, (D) laser etching SEM images ×500, (E) sandblasting SEM images ×500

Table 1: Mean and standard deviations of the resin tags for the five groups

Group	*n*	Mean (µm)	SD	SE	Minimum (µm)	Maximum (µm)
A	5	15.10	4.2	1.8	6.8	26.2
B	5	8.73	1.6	0.7	5.2	14.1
C	5	7.9	1.3	0.7	4.4	14.6
D	5	11.6	4.4	1.9	6.6	24.1
E	5	6.7	0.5	0.2	3.9	9.92

Discussion

Bonding of orthodontic brackets to the enamel surface requires the creation of microretentions in the enamel. The quality of the retention can be determined by the degree of surface irregularities on the resin enamel interface. For an orthodontist, 37% phosphoric acid concentration with 30-second application time is the gold standard for enamel etching.[9] Today, 37%

Table 2: Analysis of variance

	Sum of squares	Degrees of freedom (df)	Mean squares	*F* value
Between	227.973	4	56.993	6.704
Within	170.040	20	8.502	
Total	398.013	24		

There is a statistically significant difference between the groups (*P* value = 0.001)

Table 3: Comparisons between the groups with the *t* test

Groups	*n*	*P*
A	5	0.0135*
B	5	
A	5	0.0067*
C	5	
A	5	0.2387
D	5	
A	5	0.0022*
E	5	
B	5	0.390
C	5	
B	5	0.212
D	5	
B	5	0.0286*
E	5	
C	5	0.112
D	5	
C	5	0.112
E	5	
D	5	0.04*
E	5	

*This difference is considered to be statistically very significant at *P* value of 0.05

phosphoric acid is effectively used to change enamel surface characteristics and provide micromechanical retention areas.

Gwinnett and Buonocore were the first persons to describe resin tags inside the enamel as filamentous resin projections, similar to enamel prism and approximately 10–20 μm in length. Resin tags are factors for adhesive retention. According to Hosein *et al.*,[10] a depth of 3–15 μm or more is necessary to provide optimum shear bond strength and penetration.

The depth of resin penetration was first studied by Sheykholeslam and Buonocore[11] in which permanent teeth had depth in dissolution of the prism cores that ranged from 12 to 24 μm. According to Fjeld and Øgaard,[12] bonding brackets to 35% phosphoric acid–etched enamel resulted in thick resin tags relatively deep into the enamel of length 10–20 μm. Similar type of results are obtained in this study where 37% phosphoric acid etching for 30 seconds showed more depth in resin penetration in the range of 6.8–26.2 μm with a mean of 15.10 μm. This value is similar to values obtained by dos Santos *et al.*[13] (16–24 μm) and Wickwire and Rentz[14] (25 μm).

But in a few studies such as Cal-Neto and Miguel[15] (64.09 μm), Diedrich[16] (80 μm), Arakawa *et al.*[17] (50 μm), and Ramesh Kumar *et al.*[18](53.04 μm), the depth of resin penetration was comparatively less than that of this study. The reason behind the difference on tag length by phosphoric acid etching might be attributed to the difference in concentration and duration of phosphoric acid and prism orientation.

In 10% polyacrylic acid, the interface between the enamel and the adhesive showed less resin tag length than that of phosphoric acid. The mean depth of resin penetration was found to be 8.73 μm. This is in accordance with the observation by Fjeld and Øgaard[12] in whose study polyacrylic acid showed less or no visible tags.

According to Fjeld and Øgaard,[12] the surface treated with SEP showed very few, thin, and shorter resin tags (5–10 μm). In this study, most tags were in the range of 4.4–14.6 μm with mean of 7.9 μm in length. Cal-Neto and Migue[15] and dos Santos *et al.*[14] found similar results.

The studies by Bishara *et al.*[19] and dos Santos *et al.*[14] showed that tag lengths with SEP were shorter than with phosphoric acid. In this study also it is confirmed that depth of penetration of SEP is less than that of phosphoric acid.

Though depth of resin penetration is short, the statistically consistent tag length might have increased the surface area of mechanical undercuts that probably made the SEP system clinically efficient.[19] Although SEP is conservative on enamel with short resin tags, it produced bond strength that was clinically acceptable, with lower rates of bond failure.[15]

Laser irradiation has microcracks, which aids in the penetration of resin. Laser irradiation causes surface roughening and irregularity similar to that of acid etching to a depth of 10–20 μm.[9] Similar results are obtained in this study, where the mean depth of resin penetration in laser irradiation group is 11.6 μm. Furthermore, the depth of penetration of laser irradiation was less than that for the acid-etched group, but more than SEP. In this study, attempt is made to use lasers as an alternative method for etching enamel, which may prove a more convenient and time-saving procedure.

Table 4: *Post hoc* bonferroni test

Multiple comparisons						
Dependent variable: Etchants						
(I) Groups	(J) Groups	Mean difference (I – J)	Standard error	Significance	95% Confidence interval	
					Lower bound	Upper bound
1	2	6.36600*	1.84669	0.025	0.5426	12.1894
	3	7.19720*	1.84669	0.009	1.3738	13.0206
	4	3.50680	1.84669	0.721	−2.3166	9.3302
	5	8.40120*	1.84669	0.002	2.5778	14.2246
2	1	−6.36600*	1.84669	0.025	−12.1894	−0.5426
	3	0.83120	1.84669	1.000	−4.9922	6.6546
	4	−2.85920	1.84669	1.000	−8.6826	2.9642
	5	2.03520	1.84669	1.000	−3.7882	7.8586
3	1	−7.19720*	1.84669	0.009	−13.0206	−1.3738
	2	−.83120	1.84669	1.000	−6.6546	4.9922
	4	−3.69040	1.84669	0.595	−9.5138	2.1330
	5	1.20400	1.84669	1.000	−4.6194	7.0274
4	1	−3.50680	1.84669	0.721	−9.3302	2.3166
	2	2.85920	1.84669	1.000	−2.9642	8.6826
	3	3.69040	1.84669	0.595	−2.1330	9.5138
	5	4.89440	1.84669	0.154	−.9290	10.7178
5	1	−8.40120*	1.84669	0.002	−14.2246	−2.5778
	2	−2.03520	1.84669	1.000	−7.8586	3.7882
	3	−1.20400	1.84669	1.000	−7.0274	4.6194
	4	−4.89440	1.84669	0.154	−10.7178	0.9290

*The mean difference is significant at the 0.05 level

Air abrasion uses a high speed of Al_2O_3 particles propelled by air pressure. Sandblasting with the least mean depth of penetration was the most inconsistent of all groups. The depth of resin penetration is in the range of 3.9–9.9 μm. The literature reviews were also attributed to the reduced depth of resin penetration and bond strength than acid etching.[18] Sandblasting enamel is not recommended as a means of enamel preparation for orthodontic bonding.[7] Similar result is obtained in this study.

In this study, SEM was used to evaluate and compare depth of resin penetration between five different enamel conditioning methods used for orthodontic bonding. The depth of resin penetration of etched enamel surface varied significantly between the groups and also within the group due to morphological variation in the crystal orientation of the enamel surface.

Conclusion

Twenty-five human extracted premolar teeth were divided into five groups of five each and were subjected to five different types of enamel conditioning methods, namely, 37% phosphoric acid, 10% polyacrylic acid, SEP, laser etching, and sandblasting. The teeth were sectioned buccolingually for the evaluation of the depth of resin penetration after bonding of brackets. This study concluded that 37% phosphoric acid etching resulted in the greater depth of resin penetration than did other methods. Acid etching is preferred for the orthodontic bonding over other methods. 10% Polyacrylic acid produced low depth of resin penetration. SEP produced lower resin penetration depth than acid etching. Laser etching resulted in depth of resin penetration comparable to acid etching. Sandblasting had the least resin penetration depth among all groups.

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Nil.

Conflicts of interest

There are no conflicts of interest.

References

1. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. J Dent Res 1955;34:849-53.
2. Gardner A, Hobson R. Variations in acid-etch patterns with different acids and etch times. Am J Orthod Dentofacial Orthop 2001;120:64-7.
3. Movahhed HZ, Ogaard B, Syverud M. An in vitro comparison of the shear bond strength of a resin-reinforced glass ionomer cement and a composite adhesive for bonding orthodontic brackets. Eur J Orthod 2005;27:477-83.

4. Artun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. Am J Orthod 1984;85:333-40.
5. Buyunkilmaz T, Usumez S, Karaman AI. Effect of self-etching primers on bond strength—Are they reliable? Angle Orthod 2003;73:64-70.
6. Zachrisson BU, Büyükyılmaz T. Bonding in orthodontics. In: Graber TM, Vanarsdall RL, Vig KW, editors. Orthodontics: current principles and techniques. St Louis (MO): Elsevier Health Sciences; 2005. p. 579-659.
7. Reisner KR, Levitt HL, Mante F. Enamel preparation for orthodontic bonding: A comparison between the use of a sandblaster and current techniques. Am J Orthod Dentofacial Orthop 1997;111:366-73.
8. von Fraunhofer JA, Allen DJ, Orbell GM. Laser etching of enamel for direct bonding. Angle Orthod 1993;63: 73-6.
9. Gardner A, Hobson R. Variations in acid-etch patterns with different acids and etch times. Am J Orthod Dentofacial Orthop 2001;120:64-7.
10. Hosein I, Sherriff M, Ireland AJ. Enamel loss during bonding, debonding and cleanup with use of self-etching primer. Am J Orthod Dentofacial Orthop 2004;126:717-24.
11. Sheykholeslam Z, Buonocore MG. Bonding of resins to phosphoric acid-etched enamel surfaces of permanent and deciduous teeth. J Dent Res 1972;51:1572-6.
12. Fjeld M, Øgaard B. Scanning electron microscopic evaluation of enamel surfaces exposed to 3 orthodontic bonding systems. Am J Orthod Dentofacial Orthop 2006;130:575-81.
13. dos Santos KT, Sundfeld RH, Garbin CA, de Alexandre RS, Sundefeld ML, Ceolim BN. Length of resin tags in pit-and-fissure sealants: All-in-one self-etching adhesive vs phosphoric acid etching. Compend Contin Educ Dent 2008;29:186-92.
14. Wickwire NA, Rentz D. Enamel pretreatment: A critical variable in direct bonding systems. Am J Orthod 1973;64:499-512.
15. Cal-Neto JP, Miguel JA. Scanning electron microscopy evaluation of the bonding mechanism of self-etching primer on enamel. Angle Orthod 2006;76:132-6.
16. Diedrich P. Enamel alterations from bracket bonding and debonding: A study with the scanning electron microscope. Am J Orthod 1981;7:500-22.
17. Arakawa Y, Takahashi Y, Sebata M. The effect of acid etching on the cervical region of the buccal surface of the human premolar, with special reference to direct bonding techniques. Am J Orthod 1979;76:201-8.
18. Ramesh Kumar KR, Shanta Sundari KK, Venkatesan A, Chandrasekar S. Depth of resin penetration into enamel with 3 types of enamel conditioning methods: A confocal microscopic study. Am J Orthod Dentofacial Orthop 2011;140:479-85.
19. Bishara SE, Gordan VV, VonWald L, Olson ME. Effect of an acidic primer on shear bond strength of orthodontic brackets. Am J Orthod Dentofacial Orthop 1998;114:243-7.